T.C.

FIRAT UNIVERSITY

THE INSTITUTE OF NATURAL AND APPLIED SCIENCES

INVESTIGATION OF ELECTRICAL AND THERMAL PROPERTIES OF AMINATED POLY (VINYL CHLORIDE)-OXIDIZED MULTIWALLED CARBON NANOTUBE COMPOSITES

MASTER THESIS
Hassan Haruna

Department of Chemistry

Thesis Supervisor
Prof. Dr. Mehmet COŞKUN

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ABSTRACT

Carbon Nanotubes (CNTs) are of great demands because of their noble physical properties and many application possibilities that covered a lot of area in nanotechnology. This study is concerned with the preparation and characterization of composites of oxidized MWCNT with aminated PVC. Ranges of 2 %, 5 %, 8 %, and 11 % in weight percentages of oxidized MWCNTs were added to THF solution of aminated PVC containing 10.77 % of amine groups. Sonicated for homogeneity followed by precipitation in ethanol made the final mixing of each concentration. Composites containing oxidized MWCNT in different weight percentages were doped with nanographite in different percentages (1 %, 3 %, 5 %, 8 % and 12% by weight). FT-IR, DSC, TGA, and SEM are used to characterized these composites. The electric properties at different temperatures and frequencies were measured for all composites. It was found that the dielectric constant values of all composites decrease with increasing of the applied field frequency and increase with increasing oxidized MWCNT content. This is due to the decrease in polarization and the dipoles can no longer follow the rapid changes in field direction. However, A.C. conductivities for the all composites increase rapidly with increasing frequency and decrease as the temperature increases. Also its thermal properties is directly proportional to the oxidized MWCNTs contents.

Key Words: PVC, MWCNT, , CNTs, Composite, Electrical Properties, Thermal Properties

ÖZET

Aminlenmiş Poli (Vinil Klorür)- Oksitlenmiş Çok Duvarli Karbon Nanotüp Kompozitlerinin Elektrik ve Termal ve Mekanik Özelliklerinin İncelenmesi

Karbon nanotüpler, fiziksel özelliklerinden ve nanoteknolojide çok fazla alanı kapsayan birçok uygulama olanağı sunduğundan dolayı rağbet görmektedirler. Bu çalışmada poli (vinil klorür)-çok duvarlı karbon nanotüp kompozitlerinin termal ve elektriksel özellikleri incelenmiştir. Ağırlıkça % 2, % 5, % 8 ve % 11 oranlarında karboksilli MWCNT, %10.77 amin grubu içeren aminlenmiş PVC' nin THF içerisinde hazırlanmış çözeltisine eklendi. Bütün kompozitler homojenize olması için sonike edilerek ve ardından etanolde çöktürülürek hazırlandı. Aminlenmiş PVC / % 4, % 8 ve % 12 oranlarında karboksilli MWCNT kompozitleri, sırasıyla %1, %3, %5, %8 ve %11 oranlarında nanografitle doplama yapıldı. Sentezlenen tüm kompozitler FT-IR, DSC, TGA ve SEM ile karekterize edildi. Hem aminlenmiş PVC / karboksilli MWCNT kompozitleri hem de nanografit ile doplanmış kompozitlerin farklı sıcaklık ve frekanslarda dielektrik ölçümleri yapıldı. Aminlenmiş PVC / karboksilli MWCNT kompozitlerinin elektriksel özelliklerinin artan karboksilli MWCNT içeriğiyle arttığı bulundu. Ancak artan freakansla azaldığı görülmüştür. Sonuçlar, artan freakansla dielektrik kaybının azaldığını ve A.C iletkenliğinin karboksilli MWCNT' lerin konsantrasyonu ile doğru orantılı olduğunu göstermiştir. Farklı sıcaklık ve frekanslarda doplanmış nanografitlerin elektriksel ölçümleri yapılmış ve nanografitle doplanmış kompozitlerin elektriksel özelliklerinin, nanografit içeriğinin artmasıyla ve frekansla ters orantılı olarak arttığı bulunmuştur. Ayrıca termal özelliklerinin de karboksilli MWCNT ve nanografit oranlarıyla doğru orantılı olduğu tespit edilmiştir.

Anahtar Kelimeler: PVC, MWCNT, CNTs, Kompozit, Elektriksel Özellikler, Termal Özellikler

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ABBREVIATIONS

AFM : Atomic force microscopy
CNTs : Carbon nanotubes
DSC : Differential scanning colarimeter
h : Hour
H_2SO_4 : sulphurı acıd
HCl : Hydrochloride
HNO_3 : NITRIC ACID
IR : Infrared
L : Litre
M : Molar
MHz : Mega Hert
mL : Millitre
mmol : Millimoles
MWCNTs : Multi walled carbon nanotubes
NaOH : Sodium Hydroxide
PE : Polyethylene
PP : Polypropylene
PS : Polystyrene
PVC : Polyvinyl chloride
SEM EDX : Scanning electron microscopy elementary analyzer
SWCNTs : Single walled carbon nanotubes
TEM : Transition electron microscopy
Tg : Glass transition temperature
TGA : Thermal grametri analyzer
THF : Tetrahydrofuran

1. INTRODUCTION

The primarily targets of the polymer science are advancement of brand new polymer substance, new improvement in existing polymer substances and modern applications. Polymer science also plays an important role in manufacture and methodological development. Industrial-scale manufacture polymers are polystyrene (PS), polyethylene (PE), polyvinyl chloride (PVC) and polypropylene (PP). Nanocomposite substances of polymer have noble thermal and mechanical stability (superior-tensile durability, flexible modulus and toughness than ceramic and granular compounds) [1].

Carbon nanotubes (CNTs) are of great demands because of their noble physical properties and many application possibilities that covered a lot of area in nanotechnology. Furthermore, recently research on the surface area adaptation of CNTs attracted significant interest [2], although, its lack of solubility in organic solvents restricted them to be used as drug delivery agents in living systems. There are distinct methods of adaptation of CNTs such as chemical, physical, or combination to have been used for their homogeneous scattering in common solvents to enhance solubility [3].

Covalent functionalization nanotubes were attained by different methodologies revealed in literature. Covalent adaptation of carbon substrate substance is helped by electrochemically, which has become a modern day to the area of chemically recast electrodes that comprised the development of the conjugate system among the qualifier molecule and substrate [3]. The formation of oxygen comprises of functionalities and carboxylic groups, which are usually known as surface oxide. These surface oxides designed by the vigorous oxidation treatment using strong acids. CNTs can also be attached onto the surface of polymer via grafting method, which processed that the reactive groups unite to the surface of nanotube by covalent bond.

Because of its cheap, superior mechanical strength, flame retardant and noble resilience to chemical which makes PVC the most-used polymer [1]. The undesirable feature of PVC is its poor thermal stability. The Conjugated double bonds can form within the structure due to the removal of HCl. The property of the polymer is impaired went undergoes rapidly oxidized. Nanocomposite can be used to minimize this disadvantage [4].

1.1. Composite

According to Talreja Et al. (2009) composite is defined as joining of substances varying in composition, where the particular components keep their distinct characters. The required stiffness or mechanical stability to the composite part is achieved by joining these different components together. The substance that comprises of two or more different stages (i.e. scattered stage and matrix stage), and composing of bulked properties considerably distinct from those of any of the components is known as composite substance.

The primary phase containing a continuous identity is called matrix stage. The less hard phase and more ductile is usually found in the matrix. It also influences the scattered stage and partakes in a load with it. The discontinuous form of matrix embedded the dispersed phase (reinforcing). This phase is known as dispersed phase which can be called secondary phase. Furthermore, this dispersed phase is stronger than the matrix phase.

The structural applications have the successive features in composites as follows:

i. Composite materials have exceptional mechanical properties and exceptionally in a few matters, distinct from the properties of their components.
ii. Composite substance mostly comprises of two or more materially different and mechanically distinguishable substances.
iii. Composite substances are manufactured by blending the distinct substances to attain monitored and homogeneous scattering of the components [5].

Examples of materials composite are cellulose fibres, cement concrete, straw, mud and Portland asphalt. The mainly developed example of composite substance is aircraft in interesting surroundings. Developed composites have great function fibre reinforcement in a substance of polymer matrix which is epoxy. Instances are epoxy/kevlar, epoxy/boron as well as epoxy/graphite composites. Functions of modern composites are conventionally in the aircraft factories, although they can also be used in industrial scale.

1.2. Composite classification

Composites classification is based on stage of matrix, which can be grouped into composites of matrix polymer, composites of matrix metal and composites of matrix ceramic (Fig1.2) [5]. The types of reinforcement composed in composites which are composites of particles (consisted of particles), composites of fibrous (consisted of fibres) and composites of laminate (consisted of laminates) offer composites classifications.

Composites of fibrous are further classified based on synthetic or natural/bio-fibre. Composites of bio-fibre encompassing are known as bio-fibre composites. They can be segmented once more upon the fundamental of the matrix. These are biodegradable matrix and nonbiodegradable matrix [5]. Composites of bio-based created from biodegradable and natural/biofibre polymers, which are also known as green composites. These can also be categorised into textile composites and hybrid composites. Composites of hybrid composed of a blend of two or more kinds of fibres.

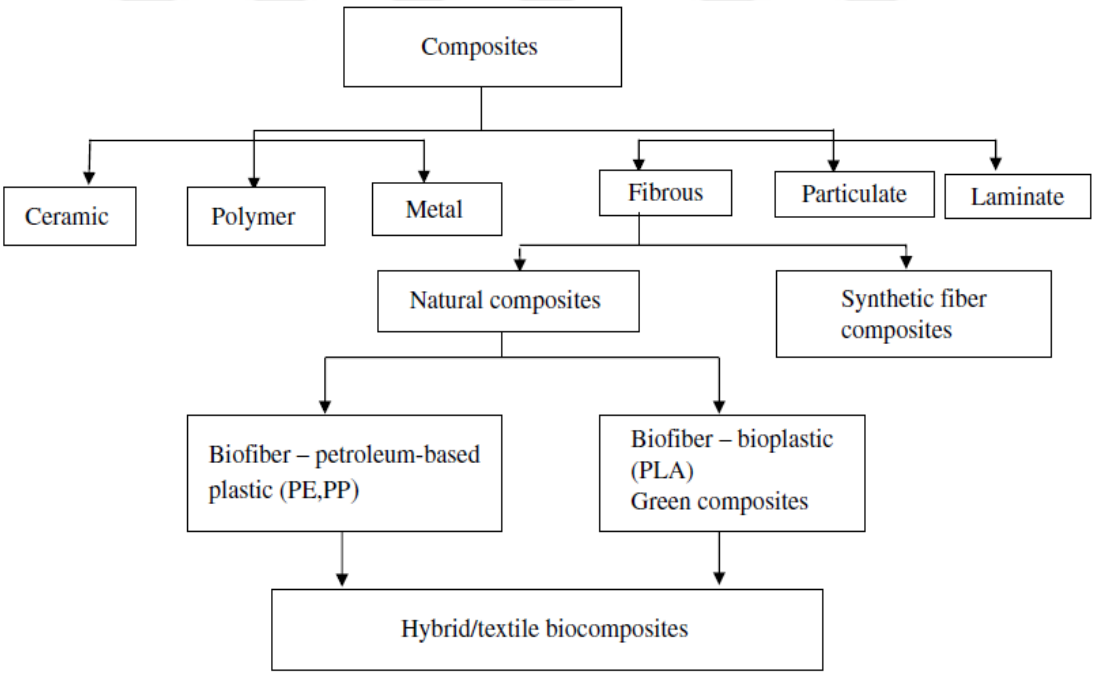


Fig 1.1. Classification of composite [5]

1.2.1. Polymer matrix composite

Many of the commercially manufactured composites use polymer matrix substance generally known as resin solution. The distinct classes of polymers available relying on the initiating raw constituents. There are many wide classes, individually with a lot of diversities. Examples of commercially manufactured composites are vinyl ester, polypropylene, phenolic, polyamide, polyester, etc. Fibres are generally used as reinforcement substances, although, ground can also be used as minerals [5] . The resin content of the final product can reduce by various methods such as vacuum infusion, hand lay-up and rule of thumb. The stability in the product is extremely relying on the fraction of resin content.

Because of its cheap and easy manufacture processes, PMCs are highly common. Polymers as structure substances are restricted by use of nonreinforce which result in poor level of their mechanical properties, which include: stability, collision resilience and modulus. Polymer reinforcement allows manufacture of PMCs by strong fibrous network.

1.2.1.1. Advantages of polymer matrix composite

i. It is Inexpensive
ii. It has superior weariness resilience
iii. It has superior particular durability
iv. It has superior particular tension
v. It has superior erosion resilience
vi. It has superior break resilience
vii. It has superior contact resilience
viii. It has superior corrosion resilience

1.2.1.2. Disadvantages of polymer matrix composite

i. Thermic durability
ii. Superior coefficient of thermic increase

1.2.1.3. Properties affecting factors of polymer matrix composite

1.2.1.3.1. Properties of matrix

The use of distinct polymers properties will be decided by it suitability. The used of polymer as matrix is due to it cheap, easily manufacture processes, excellent chemical resilience and despicable specific gravity. It disadvantages include: lack of durability, despicable acting temperature and lack of modulus restriction their exploit [5]. Different types of polymer matrix are elastomers, thermosetting polymer, thermoplastic polymers and their compounds.

Thermosetting polymers: Is a polymer comprises of the network framework or cross-link by the conjugated system with every atom. It does not disintegrate under flame. They cannot be recasted when it hardened by a cross-linking method, Examples of thermosetting polymer are polyesters, polyimides, epoxies, silicone, polyesters, melamine, phenolics and urea [5].

Elastomer: Is a polymer that is characterized by viscoelasticity, their high-yield strain and Young's modulus are mostly found to be low in comparisons with other constituents. Its name originated from elastic polymer, which is generally served to replace the name rubber. However, vulcanizates is the most recently recommended name. Monomers that joint together to design the polymer is mostly manufactured from the followings: oxygen, hydrogen, and carbon [5]. They are amorphous polymers having to be over their glass transition temperature. Hence noticeable segmented movement is attainable. At room temperatures, elastomers are comparatively distorted and soft. The main exploitations of elastomers which are included: sealant pastes and shaped flexible, rubber, polybutadiene, fluoroelastomers, parts natural rubber, epichlorohydrin, thermoplastic elastomers, synthetic polyisoprene, rubber of chloroprene, rubber of butyl, rubber, polysulfide, rubber of ethylene propylene, rubber of silicone, etc.

Thermoplastic polymers: These are polymers that composed of the branched chain or linear atoms held together by intramolecular bonds, though, they have weak intermolecular bonds between the atoms. The polymers can be recasted by use with high temperature and compression. They can either be amorphous or semi-crystalline in shape. Their Examples

are polyether polyphenylene, polyethylene, nylons, polypropylene, polystyrene, polysulfone, polyamide-imides, polyamide-imides, etc [5].

1.2.1.3.2. Interfacial. Adhesion

The composite substance behaviour is described by the integrated performance of the matrix fibre/interface, polymer matrix and reinforcing element (Figure 1.2.1.3.2). In order to achieve great properties of mechanical the interfacial adhesion must be tough. Particles within the matrix can be attached to the surface fibre by adsorption or reaction of chemical that decides the degree of interfacial adhesion. The introduction of atomic force microscopy (AFM) as well as nano indentation devices has aided the investigation of the interface which sometime called mesophase [5].

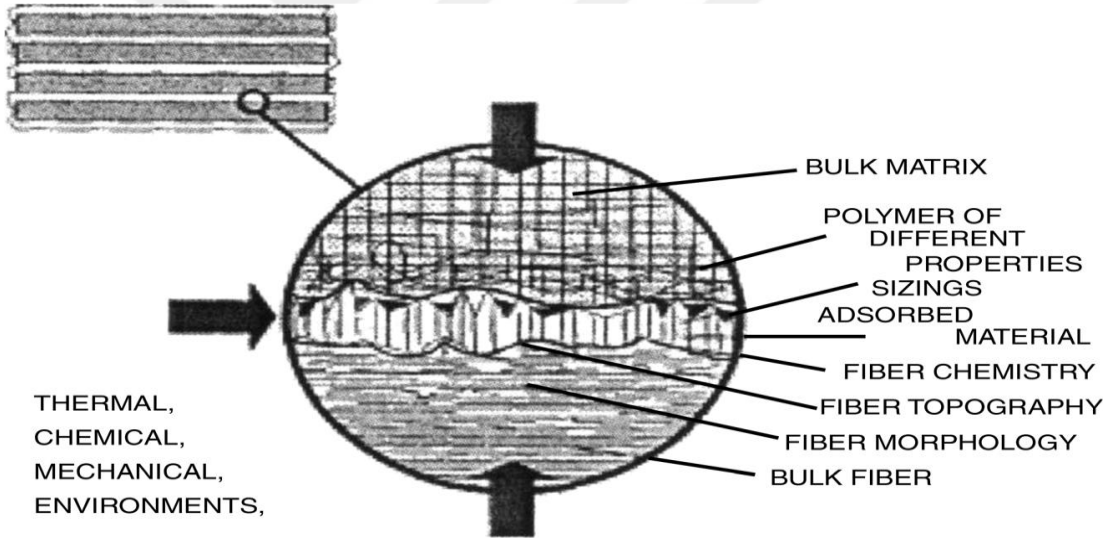


Fig 1.2. Schematic. model of interphase [5]

1.2.1.3.3. Dispersed phase inclusions' Orientation and Shape. (Laminates, fibres, particles, and flakes)

Bednarcyk *Et al.* (2003) disclosed that an atomic particle has no specific dimensions, which is mostly served to lower the cost of isotropic substances and or enhance its properties. Reinforced atomic particles can either be irregular geometry, or regular geometry, cubic, platelet and spherical in shapes. Particulate reinforcements possess directions, which are nearly same in all dimensions. Particle-reinforced composites

composed of dispersion-strengthened as well as large particle. Composite laminar is comprised of panels or two-dimensional layers which favoured high-strength dimension example found in wood [5].

1.2.1.4. Composites Fabrication.

Manufacture as well as casting of composites to final products as reported by Chrysanthopoulos Et al. (2009) is generally integrated the composition of the substance itself through the manufacture method [5]. These fabrications processed are injection molding, hand lay-up, pultrusion, filament winding, resin transfer molding, sheet molding, etc.

1.2.1.4.1. Process of Spray-up

The process of spray – up involved spraying of chopped reinforce as well as liquid resin matrix fibres onto the surface of mold. The fibres are chopped into fibres of 1–200 (25–50mm) length and later spraying by an air jet at the same time with a resin spray at a fixed fraction among the matrix stage and reinforcing. The fast production of uniform composite coating is achieved by spray-up method. Nevertheless, the technique is incapable of use continuous reinforcing fibres. Therefore, the mechanical properties from the substance are moderate.

1.2.1.4.2. Hand lay-up

It is the easiest, earliest, as well as the most popular method used during the fabrication of reinforcing yields. Its method comprises of a smooth surface, positive-shaped mold and/or a cavity created from plastic, metal, wood or a combination of these substances.

1.2.1.4.3. Filament Winding

It is used during the making of surfaces of reformation, which included: spheres, pipes, cylinders and tubes, and they are also commonly used during pipe work construction for chemical industries and large of tanks. The basis of the filament winding method is reinforcement of lay down of continuous precise at high-speed.

1.2.1.4.4. Pultrusion

Pultrusion is a mechanized method for composite substance fabricated to constant cross-section profiles, and continuous profiles. Product is removed from perish then pressurizing forcibly out in puitrusion method. These profiles are of different shapes. Also using suitable dies, different structural shapes can be form.

1.2.1.4.5. Process of molding Bag

Most of the adaptable used methods in parts of composite fabrication are molding bag method. The setup of lamina takes place in mold and resin is dispersed, enclosed along a bag or elastic diaphragm which can be treated under pressure and heat in bag molding method. The substances turn out to be uniting part of molded shaped to the needed conformation after the desired curing cycle [5]. The molding processes comprised of three essential, which included: autoclave, pressure bag and vacuum bag.

1.2.1.4.6. Molding Preformed Compounds

This method comprises a huge amount of thermosetting reinforced resin products which are manufactured by methods of matched die molding that included: injection molding, transfer molding and hot press compression molding. The use of preformed molding compound or premix is more suitable where all vital constituents are comfortably added since preformed compound method is a wet process [5]. This allows the achievement of quicker formation rates. Molding performed compounds are further classified into: prepregs, molding sheet and molding dough.

1.2.1.4.7. Injection Molding

It is used during the manufacturing the substances of thermosetting plastic as well as thermoplastic. The composite passed into a barrel heated, blended then delivered into a cavity mold where it cool down and solidifies into the shape of the cavity mold. It is also used to make several things, which included: pocket combs, packaging, wire spools, automotive dashboards, bottle caps, etc. They are used for manufacturing same object of high volumes [5]. Injection mold has some advantages, which are included: rapid rates formation, high repeatable tolerances, as well as its capability for using a broad variety of substances cheap labour, few desires to final parts after molding and minimum part losses. On the other hands, the method is very costly to carry out and also their desire to form mouldable parts.

1.2.1.4.8. Transfer of resin Molding

Due to transfer of resin potentiality of agreeable inexpensive, it has an advantage of manufacturing enormous, consolidated, and higher yield performance. A reinforced dry substances use during this process has been cleavage and moulded into a design portion, known as perform, is positioned in an arranged cavity mold. Usually, at the bottom point, the resin is inserted, which then filled the mold up to decrease the entrapped air. The loss of resin is minimizing by clamping the tube if the resin is found leaking within the resin trap. The flow of resin is then blocked and the cure of component of the mold occurs while the flow of resin excess starts from the vent regions of the mold. Development of composite sufficient green strength called for its removal from the tool and postcured (Fig 1.3).

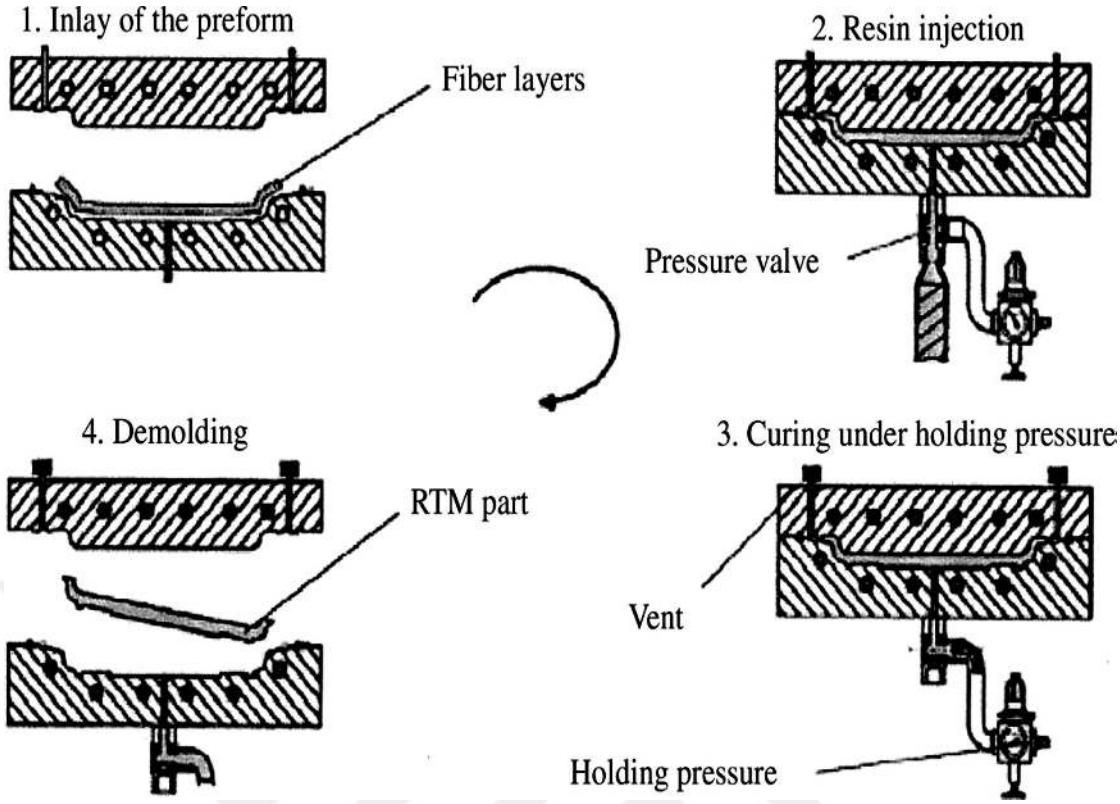


Fig 1.3. Schematic representation of RTM technique [5]

1.2.1.4.9. The reaction of Reinforced injection molding

Reaction of reinforced injection mold involved addition of reinforced agents to the blend. Such technique is called reaction of reinforced injection moulding. These reinforced additives are mica, fibres and glass. They are used to manufacture automotive panels of stiff scum. Reaction of reinforced injection moulding is a subset of reaction of structural injection moulding that served as reinforced additive for mesh fibre. The arrangement of mesh fibre is in the mold, and consolidated polymer is added to it.

1.2.1.5. Applications of polymer composites matrix

The application of polymer composite matrix includes: aerospace frameworks, marine stuffs, sport commodities, chemical storage, biomedical, bridges and bullet roofs [5].

1.2.2. Ceramic matrix composite

The word ceramic originated from Greek word keramikos, which means pottery. The term ceramics are defined as hard substances that display extremely strong electrovalent bonding and a time shows covalent bonding. They are non-metallic solids, inorganic and also crystalline in nature [6].

The advantages of using reinforcement materials in composites of metal matrix as well as composites of polymer matrix are to enhance stability of the mixtures while, in composites of ceramic matrix is to improve composites hardiness. Definition of composites of ceramic matrix are substances that contained one or more different ceramic stages internationally in addition to the other, which improve some properties that are not found in monolithic ceramic materials [6].

Ceramic matrix composite comprises of ceramics glass, non-oxides, oxides glass, and carbon. The important of these constituents is to offer reinforced stage in the designate place as well as prevent reinforcement from been exposed to the environment. The addition of fillers sometimes takes place to the composite of ceramic matrix for enhancement of its properties which included: electrical conductivity, thermal conductivity, thermal conductivity, and thermal expansion and hardness properties. These materials can exists as a molecule that have diverse forms which include faceted, spherical, and irregular are generally used during the treating of composites of ceramic matrix [6].

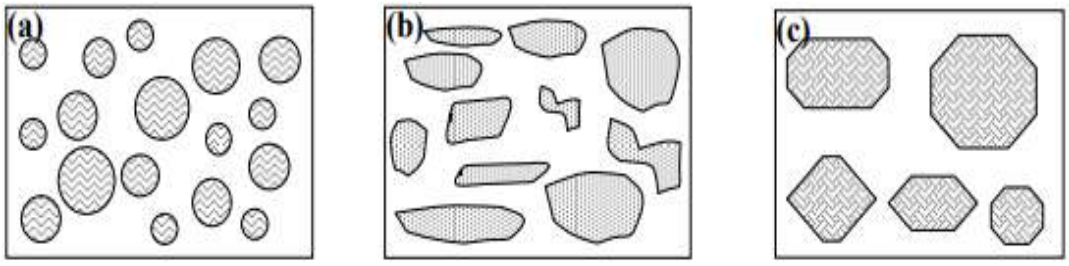


Fig 1.4. Morphology of particulate (a) spherical (b) irregular (c) faceted

They can be treated either by adapted method or by other more specific and conventional powder treating approaches. The temperature for treating of ceramic matrix composites is very high compared to metal or polymer matrix composites that result to an extremely hard and the treating is costly [6].

1.2.2.1. Types of ceramic matrix composites

i. Oxides/oxide matrix composite
ii. Continuous fibre/ glass matrix composite
iii. Continuous fibre/ glass matrix composite
iv. SiC whisker/ Si_3N_4 matrix composites
v. SiC whisker/ Al_2O_3 matrix composites
vi. SiC particulate/ Si_3N_4 matrix composites
vii. Al_2O_3-ZrO_2 matrix composites

1.2.2.2. Advantage of ceramic matrix composites

i. It has superior toughness
ii. It has actual lightweight
iii. It has crispiness
iv. It has stronger durability to load proportion
v. It has stronger compound durability

1.2.2.3. Disadvantage of ceramic matrix composites

i. The treating of ceramic matrix composites carries out at high temperatures.
ii. The main disadvantage of ceramic matrix composite is brittleness, which is formed to enhance of monolithic ceramics hardness.
iii. Temperature of treating of ceramic matrix composite is very high due to their complication in production and therefore, processing is costly.
iv. Distinction exists among reinforcement, the coefficients of thermal expansion as well as a matrix which on cooling causes thermal stresses by treating temperatures [6].

1.2.2.4. Composites of ceramic matrix applications

Composites of ceramic matrix can be served in the followings: hot gas ducts, cutting tools, rocket engines , shield systems for heat, flame holders, gas turbines components,

burners components, brake disks as well as airplanes brake system components or cars that courses thermal shock extremely, components of bearing necessitating high wear resistance and corrosion [6].

1.2.3. Metal Composite

These are substances composing of a metallic matrix joined with a metallic (molybdenum, tungsten, lead) or ceramic (carbides, oxides) scattered stage. The reinforcement is normally in the range of 10-70% by volume fraction. It can also provide a variety of property improvement over monolithic alloys [7].

Metal matrix composite reinforcement can have several objectives as follows;

i. At room-temperature, yield durability and ductile durability are increased whereas the minimum ductility and/or toughness are maintained,
ii. At higher temperatures creep resistance is increase in comparisons to conventional alloys,
iii. At higher temperatures fatigue strength is increased,
iv. Thermal shock resistance is enhanced,
v. Corrosion resistance is enhanced,
vi. Young's modulus is increased,
vii. Elongation lower thermal reduction.

1.2.3.1. Advantages of metal composite

i. It has actual lightweight
ii. It has high durability
iii. It operation is at higher temperatures
iv. It has low density and also had superior wear resistance

1.2.3.2. Classification of metal matrix composites

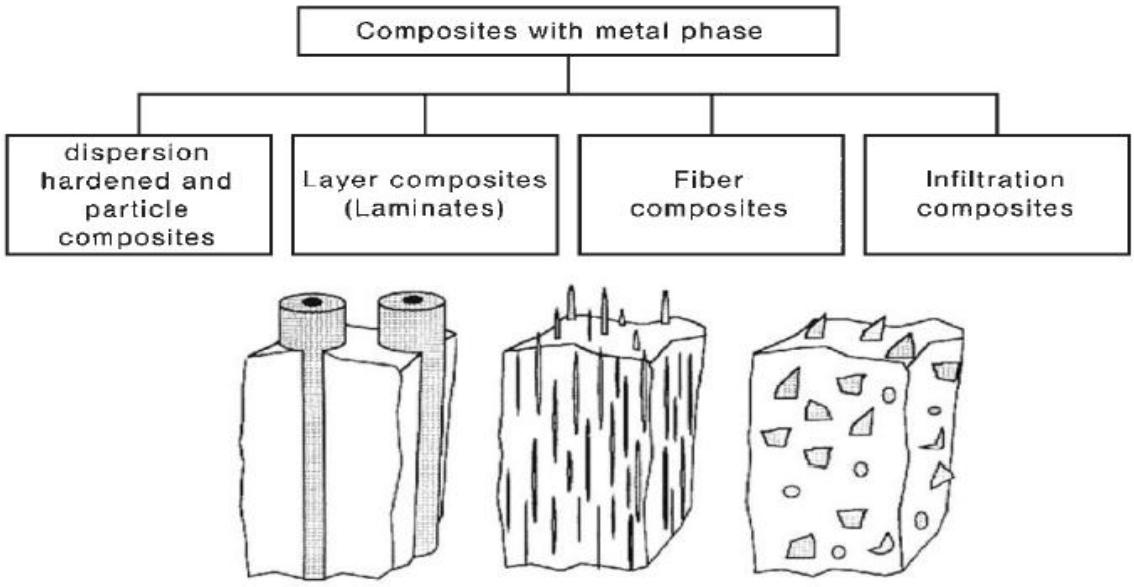


Fig 1.5. Classification. of metal matrix. composites [7]

1.2.3.3. Metal matrix Composites Processing

The substances of composite of metal matrix can be manufactured and processed via several methods as follows; liquid-state techniques, casting or liquid infiltrations, liquid-state methods, casting or liquid infiltration, liquid-state systems, squeeze casting or pressure infiltration, solid-state processes, diffusion bonding, solid-state method, distortion preparing, solid-state processes powder preparing. Solid-state methods sinter-forging, solid-state processes deposition techniques, in situ processes and spray-forming of particulate matter of metal matrix composites [7].

1.3. Carbon nanotubes (CNTs)

According to Lillehei *et al.* (2002) CNTs is defined as graphene layers, which rolled into tubes and achieved great interest because they show a broad diversity of novel chemical, electrical, mechanical, and optical properties [8]]. Baughman *et al.* (1999) also described CNTs as either metallic or semi conducting properties, depending on their diameter and chirality which make them strengthen filler in composite substances.

With the advent of science and technology on CNTs, the attention of many experts internationally has been drawn. Due to their stability, small sizes, and outstanding physical properties of these structures make a novel substance with many favourable diversity uses.

The carbon nanostructure are well understood after disclosed of fullerenes in a way that architectures build from sp^2 carbons united based on simple geometrical principles. Their applications are included: electronic and electrochemical, as mechanical reinforcement performance composites in nanotubes base field emitters, biological and chemical investigation, etc [9].

1.3.1. History of carbon nanotube

A clear image of 50 nano-meter diameter tube created from carbon was disclosed by V.M Radushkevich *Et al*. (1952) at Journal of physical chemistry, Soviet. The publication of the paper was in Russian language. Thus, Western experts were unavailable to release the paper to the Soviet press because of cold war. As a result of these, the paper was unpublished to the world. There is possibility that CNTs were fabricated prior that time. However, the discovery of CNTs using an advance transmission electron microscope permits visualization of these structures directly [9].

The manufacturing and monitoring of CNTs at various conditions were before 1991. Koyama *Et al.* (1976) revealed that CNTs displayed distinctly hollow carbon fibres with scale of nano-meter diameter using a method of vapour-growth. Moreover, the writers display a transmission electron microscope image of a CNT comprising of a single walled graphene. This image discovered was associating with SWCNT [9].

Proof of CNTs existed was revealed in 1979 by John Abraham in Pennsylvania state university at the 14th biennial conference of carbon. Article conferences explained CNT as fibres of carbon manufactured on carbon anodes through method of arc discharge. The fibres were characterized by hypothesis under low pressures in a nitrogen atmosphere [34].

Team of USSR experts disclosed the result of chemical characterization as well as structural of nanoparticles of carbon manufactured by a disproportion of thermo-catalytically carbon in 1981. The writers using XRD and TEM image forms to propose that the multi-sheet carbon tubular crystals were designed by coiling sheets of graphene. The

group consider rolling graphene sheets to comprise of a cylinder and distinct arrangements of graphene layers. They proposed two prospects of such arrangements: a spiral, helical arrangement (chiral tube) and armchair tube [9].

1.3.2. Discovery of carbon nanotubes

Sumio Iijima NEC laboratory in 1991 disclosed CNTs, by using advance TEM to monitoring of CNTs as well as MWCNTs in the arc-burned graphite rods of insoluble material. Moreover, Mintmira and White (1991) stated that CNTs could exhibit remarkable conducting properties with an initial scramble through a single-walled carbon nanotube [9].

IBM and NEC (1991) facilitated CNTs investigation with tremendous development of SWCNTs and with catalyst addition of transition metal to the carbon by method of an arc discharge. The method of arc discharge has been discovered to form the Buckminster famed fullerence on a preparatory scale, and these seemed to develop the run of fortuitous disclosures associating to fullerences [9].

1.3.3. Classification of carbon nanotubes

The classifications of CNTs are included; SWCNTs as well as MWCNTs [9].

1.3.3.1. SWCNTs

It's comprised of particular sheet of atomic thickness of graphite (i.e. known as graphene)rotated in design of the cylinder. Thickness of SWCNTs is almost 1nm with a distance from the tube that comprises of several millions of times long. Electric properties have been displayed by SWCNTs unlike MWCNTs that the properties are not partaken [9]

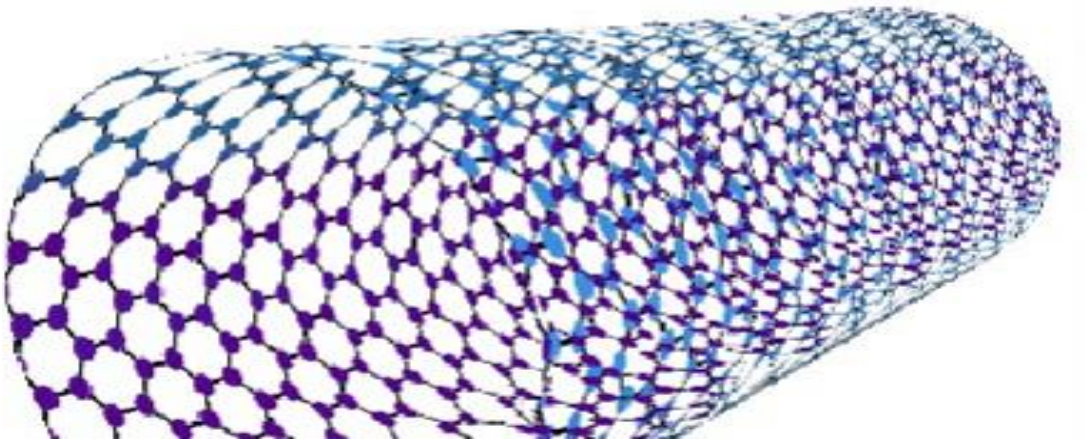


Fig 1.6. SWCNTs

1.3.3.2. Multi-walled Carbon nanotube (MWCNTs)

It comprises of several rotated sheets (concentric tubes) of graphite. The structure of multi wall carbon nanotube can explain by using two models. Model of Russian Doll composed of layers of graphite, that are aligned in the cylindrical concentric. Parchment model composed of a particular graphite layer, which is coiled in around it, looking like a newspaper rotated or parchment coil.

Inner shells have the telescopic motion capability, and also have novel mechanical properties, which allow the usage of MWCNTs as a key portable arm in approaching devices of nano mechanical [9].

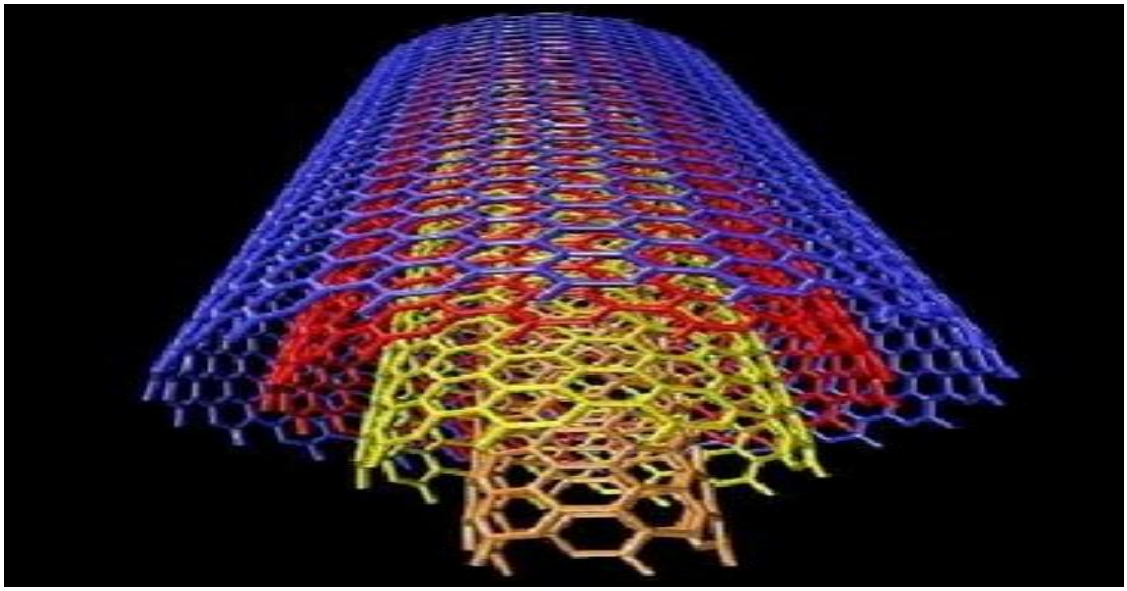


Fig 1.7. Multi wall carbon nanotube

1.3.4. Synthesis Of carbon nanotubes

The manufacturing of CNTs usually occurs from various methods such as method of chemical vapour deposition, method of arc discharge and method of laser ablation. It takes place in gases process or vacuum. Development of carbon nanotubes in method of chemical vapour deposition usually occurs at atmospheric pressure or vacuum. The large quantities of CNTs synthesized can be achieved via this process: catalytic growths as well as advancement of continuous processes, which make CNTs possible for commercial scale. Two different classes of CNTs (i.e. MWCNTs and SWCNTs) can be manufactured from these methods [9].

The diameters of the carbon nanotubes are between 0.4nm to 3nm for SWCNTs and between 1.4nm to approximately 100nm for MWCNTs. Hence, of CNTs' properties can be modified through altering the diameter. Woefully, now a day SWCNTs are manufactured only on a small-scale and are very costly. The well-known methods for synthesis of SWCNTs arise in main concentrations of impurities. Acid treatment removal of aforementioned impurities establishes other impurities to impair length of nanotube and excellence as well as adding cost to CNT. Manufacturing of MWCNTs via pyrolysis of gas-phase using the catalyst, such as nanotubes Hyperion, have high imperfection densities in comparisons to those manufactured by other costly methods [9].

1.3.4.1. Arc discharge Method

Manufacturing of carbon nanotubes involves three elements: heat, metal accerator and carbon feed.

The discoveries of CNT were, firstly, by using this method. The method of arc discharge is one of the most useful methods of nanotube synthesis. It comprises of two electrodes of graphite, which are sited in atmospheric inert helium. CNTs are manufactured on the cathode when DC current passed anode and consumed [9].

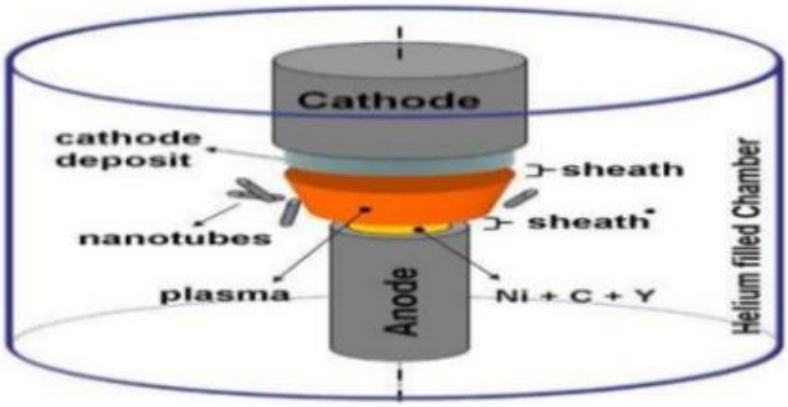


Fig 1.8. Arc discharge

1.3.4.2. Method of Laser Ablation

A reactor (laser vaporizes graphite) is used during this method whereas an inert gas is passed through it under extreme temperature. At the cooler surface of the reactor, the vaporized carbon condenses and also CNTs are manufacture here.

The system comprises of a water-cooled surface where the CNT collected. About 70% of the yields are obtained during this process. The chief yield is SWCNTs and the diameter is determined by the reaction temperature. The major disadvantages in this method are that it is associated with high cost when compared with method of arc discharge or method of chemical vapour deposition [9].

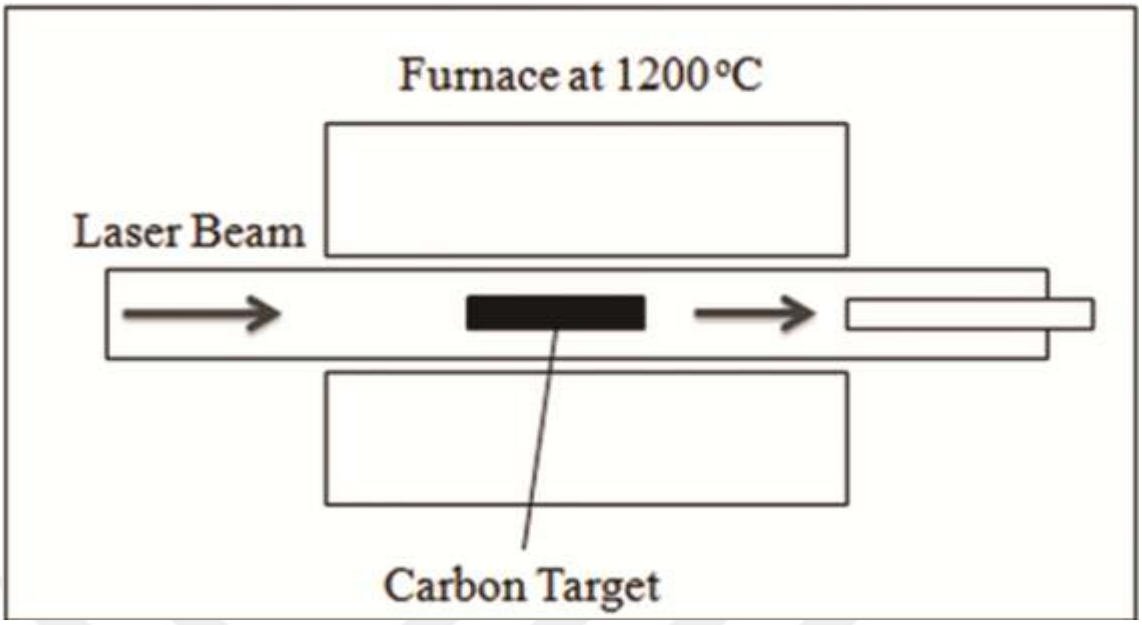


Fig 1.9. Method of Laser Ablation

1.3.4.3. Chemical Vapour deposition (CVD)

In this method iron, cobalt, nickel, or a combination which served as the catalyst is used to manufacture a substrate with a layer of metal. The substrate is heated to about 700C. The nanotube diameters, is dependent on size of metal particles. Gases are allowed to pass through the reactor; the site of the metal catalyst is where CNTs developing.

At the surface of the catalyst the carbon containing substances is broken apart, and the nanotubes are designs to the ends of the particle [9].

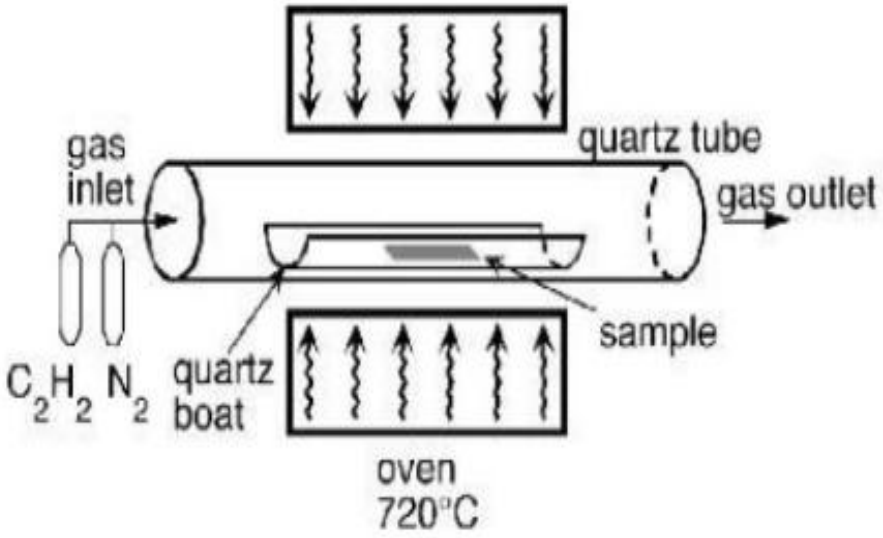


Fig 1.10. Chemical Vapour Deposition

1.3.5. Milestones in carbon nanotubes evolution

The longest carbon nanotubes are 18. 5cm long were revealed in 2009. The substrates of the CNTs were produced on Si by enhancement method of chemical vapour deposition, and it was found to have electrically uniform arrays of SWCNTs.

Early 2009, Organic compound cycloparaphenyleene are the shortest carbon nanotubes that were synthesized and the armchair CNT (2.2) was found to be the thinnest yield with a diameter of 3A. The types of CNTs are determined using advance HRTEM, DFT and Raman's spectroscopy.

SWCNT of freestanding is approximately 4. 3A and was found to be the thinnest. Researchers have suggested that it can either be arch discharge method or single-walled nanotube. However, the exact type of carbon nanotube remains unclear. Though, other CNTs structures and method of arch discharge were identified clearly using advance high-resolution transmission electron microscopy [9].

1.3.6. CNTs Properties

1.3.6.1. CNTs Strength

The flexibility and stiffest in terms of tensile strength and elastic modulus of CNTs are yet to be understood. Its strength is derived from covalent sp2 bond formed between the carbon atoms, and this was found to be stronger than the sp3 bond found in diamond and alkanes.

Due to their hollow structure and high aspect ratio, they exhibit torsional or bending stress under compression, which makes it to be weak. They also tend to undergo bucking when placed under compressive [9].

1.3.6.2. CNTs Hardness.

CNTs are found to be harder (152Gpa) and bulker modulus (1462-546) than diamond considered before as the hardest material on earth of 150Gpa and 420Gpa [9].

1.3.6.3. Kinetic property

The multi-walled nanotubes have telescopic property, which makes them to slide without friction between the inner and outer shells of CNTs [9].

1.3.6.4. Electrical property

The electrical properties of nanotubes are strongly affected by its structure as a result of exceptional electronic structure of graphene and symmetry, which make them to have very high electrical conductivity [9].

1.3.6.5. Thermal conductivity

Most of the CNTs are found to be very good thermal conductors related to copper at room temperature.

1.3.6.6. E M wave absorption

Carbon nanotubes are used in enhancement of aircraft and military vehicle secrecy, which make them hard to detect by radar as the CNTs are served as radar absorbing material. However, research is still going on with metals such as iron, nickel, cobalt, etc. to enhance the absorption effectiveness of MWCNTs in the microwave system [9].

1.3.6.7. CNTs thermal properties

Along the tube, CNTs have good thermal properties while along its axis have good insulator properties. SWNT was found to have good thermal conductivity along its axis at a room temperature of about $35000WM^{-1}K^{-1}$ compared with copper metal, which has a good thermal conductivity of about $385WM^{-1}K^{-1}$ [9].

1.3.7. CNTs Defects properties

1.3.7.1. CNTs Toxicity

The major short coming of CNTs it causes harmful effects such as fibrotic reaction as well as inflammatory when the cross membrane barrier that reaches the organ under some condition [9].

1.3.7.2. Crystallographic Defect

Another short coming of CNTs is that it causes material properties in form of atomic vacancies, which result in the crystallographic defect [9].

1.3.8. Applications of carbon. Nanotubes

1.3.8.1. Nano-electronics

It was believed that the most important use of single-walled nanotubes was in the nano-electronics field due to its high conductivity [10]. Thus, the greatly conductive

carbon fibres known are single-walled nanotube ropes. Silicon as semi conductive materials is another form of SWCNTs. Degree of chirality determines the conductivity in nanotubes, i.e. the size from the diameter of the actual nanotube and the degree of twist resulted in a forming the nanotube, which highly conductive or non-conductive.

1.3.8.2. Interconnect

The metallic compound is being required by the chip manufacturers to function for connectivity among transistors on chips. Aluminium was mostly used seven years ago but later at some point, copper was introduced. Nevertheless, the resistivity of copper increases electricity flow as the dimensions of metals decreases; making the copper-based interconnects size slightly bound. It is anticipated that, by 2012 [11] the performing chips joint with more closely arranged transistors is better needed interconnects less than 40 nanometres' wide. Resistivity of copper at that point will show to be futile as technological interconnection.

The small dimensions as well as high conductivity of CNTs may offer another interconnecting possibility with copper. Result was published by Stanford University as well as Toshiba indicating a based carbon nanotube interconnected working at 1 GHz on a chip comprising transistors of 11000 the chip size of square inch of 1/100th[3] [12].

Other benefits of carbon nanotubes are that different from copper, the demand to insert interconnects into channels on the board of circuit are not needed, which might result in making fabrication method easier.

1.3.8.3. Transistors

It designs the base for new integrated circuits, which act as digital switches. Different of CNTS' configurations will cause existing of defects, which permit SWCNT to function as transistors. The switches made up of CNT composed of electron had been anticipated, nevertheless, a cryogenic such as temperatures initially had been needed. A molecule can be placed in such a switch inside a CNT to influence flowing of current through it. The molecular-scare gate is the outcome whereby the flow of electrical current is control by the position within the molecule. The gate is around the size of 1nm or magnitude of three

orders slighter than a chip of silicon in the model. Scientists in 2001[38] [13] revealed that the CNT based transistors might be recognised which operated at room temperature. Furthermore, IBM has certified the manufacture of CNT based transistors [39] [14].

1.3.8.4. Energy production and storage

Technology of Carbon Nanotube keeps potential for wide-ranging energy-related applications.

1.3.8.5. Batteries

Many of the portable electronic devices use a rechargeable lithium-ion battery, in which the batteries discharge when lithium ions transfer among the two electrodes (comprises of graphite and metal oxide). Researchers [11] have revealed that if SWCNT is been used instead of graphite the storage capacity may be increase to twofold. CNTs based electrodes can be increase to ten times lighter as well as thinner than amorphous carbon based electrodes and have greater conductivity. The decrease in weight can make a substantial decrease in the requirement of battery power in some cases, as in electric vehicles. It can also be function in super-capacitors which might make the time taken to recharge devices such as cell phones and laptops to decrease significantly.

CNT infused paper have been used to manufacture ultra-thin flexible batteries [15]. The electrolyte of the battery is ionic liquid, which is soaked through the paper. Electrolytes in sweat, urine and human blood, can similarly assist to give the battery power that are beneficiary for device use in medically implantable. The batteries may be cut without efficiency loss, folded and/or coiled.

Likewise, their power output can also be increase. Though, the substance function in the batteries such as cellulose, are actually cheap and low-cost methods of method of huge fabrication has not been advanced.

1.3.8.6 Solar cells

Scientist at Tech Research Institute, Georgia discovered that a cell that uses solar energy containing high tower of 100mm composed of carbon nanotubes developed on iron

coated with wafer of silicon [16]. It comprises towers of the surface of 40,000cm2; every tower is a collection of millions of vertically well-arranged CNTs. The light on the towers is absorb by the two peaks of the cells at 45°, function with high efficiency throughout the day dissimilar to the usual solar cells that their efficiency is at peak when the sun is at 90º. This marks them suitable for using in space that eradicated the obligation a mechanical resource of orienting the cells to face the sun.

They can also be used in tablet production as glidants or lubricants due to the sliding nature as well as the nanosize of the graphite sheets held together with Vander Waal's forces [17].

1.3.8.7. Hydrogen storage

CNT can be used as hydrogen storage. It comprises of substances and nano fibres. It has been predicted as a result of their cylinder in shape and geometrical hollow, as well as diameters of nanometre-scale, that the CNT is storing liquid and gas in the inner cores through effective capillary [18].

1.3.8.8. Genetic engineering

Carbon nanotubes and nanohorns can be used for influencing the genetic and atomic advance of bio-imaging proteomics, genomes, as well as manufacturing of tissue. Because of their noble cylindrical structure and properties of Nanotubes can also be used as genes carrier (gene therapy), cancer treatment and genetic disorders. They act as gene therapy's vector. Effect of Antiviral in respiratory syncytial virus (RSV), a virus associated with harsh asthma 34 and bronchitis has been observed in Nanostructures. The therapy is normally completed by joining the slicing technology of gene and nanoparticles. Nanotubes are recognised for embryonic rat brain growth of their neurons and helical crystallisation of proteins [17], [19].

1.3.8.9. Biomedical applications

Soluble CNT's covalently linked with biologically active peptide have been prepared by Biancoetal, which was revealed for viral protein VP1 of foot mouth disease virus

(FMDV) displaying the response of immunogenicity and eliciting antibody. Drug embedded nanotubes in chemotherapy work by attacking viral ulcers directly and thus, eradicates the viruses. There were no formations of antibodies against the backbone of CNT alone, proposing that the CNT possess no intrinsic immunogenicity. Furthermore, CNT prove to have a significant role in the novel as well as effective vaccines construction by combining all the designated characteristics for the vaccine system. Infact, the abilities of the anti-peptide antibodies for neutralization of FMDV have been improved. In vitro studies, Pai P *et al*., 2006 and Kam NWS et al, .2005 disclosed that the killing of cancer selective cell is attained by hyperthermia because of the internal cells thermal conductivity of CNT [17], [19].

The critical stage for the treatment of cancer is its early stage detection. Presently, diagnosis as well as cancer detection rest on the cells and tissues changes detected by a physically touching or imaging expertise physicist. There is great possibility that the requirement for nanostructures to pass and analyse single cells could be met [17].

1.3.8.10. Artificial implants

Body normally displays implants rejection reaction with the administration pain post. However, the attachment of nanohorns and miniature sized nanotubes with other proteins and amino acids take place for the rejection prevention. Furthermore, they can also function in artificial joints in which host rejection reaction is negligible. In addition to that, calcium filled CNT arranged in the bone structure can served because of their tensile strength as the replacement of the bone [20].

1.3.8.11. Preservative

The antioxidant properties of Carbon nanotubes and nanohorns make them to be used as preservatives in drugs' formulations prone to oxidation. They can also be served in anti-aging cosmetics and to prevent oxidation of significant skin constituents if combine with zinc oxide as sunscreen dermatological [44, 45] [19], [17].

1.3.8.12. Diagnostic tool

Because of the nanotubes are filled with protein-encapsulated or protein/enzyme, as well as their ability of fluorescence together with specific biomolecules, proved to be useful in making of implantable biosensors. Radioisotope filled enzyme into nanocapsules as well as magnetic substances, functions as biosensors, robots, motors and nanosize. CNTs can also be useful in biological systems, and cells studying [20].

1.3.8.13. As catalyst

Large surface area of Nanohorns makes it possible to be combined into CNT significantly and instantaneously can be discharge in a desired proportion at the time specific. Henceforth, frequency reduction and addition of specific quantity of catalyst can be attained via Carbon nanotubes and nanohorns [20].

1.3.8.14. As Biosensors

CNTs can also function as materials for sensing pressure, flow, stress, strain, thermal, gas, optical, mass, position, chemical, as well as biological sensors.

Other uses of sensors made up of CNTs are as follows.

The revolutionary changes are expected by biomedical industry CNT-combined sensors in many fields and particularly in the biomedical industry sector. For e.g in the application of sensing of glucose, whereby testing of glucose level regularly by diabetic patients are necessary for measuring as well as control the levels of sugar. It can be used to monitor the hazardous radiation exposed from reactors/nuclear plants and/or factories or in laboratories chemical. The detection of the exposure is the chief purpose in all these cases in diverse steps for the control of suitable treatment. The suitability of nano sensors as implantable device make them useful to monitor and measure pulsation, temperature, glucose level in the blood (this is one of the examples that would permit patient suffering from diabetes to check the levels of sugar in which piercing of finger is unnecessary to collect samples), as well as for disease diagnoses [17].

Another carbon nanotubes application is as follows;

Nanotube reinforced composites, semiconductors and micro-electronics, conducting composites, thermal protection, artificial muscles, molecular quantum wires, super capacitors, nano gear, field emission flat panel, data storage and display, nanotube space magnetic elevator and nanotube actuator. Noble radioactive, gas storage and electromagnetic shielding.

1.3.9. Advantages of carbon nanotubes

i. It can also as a new technology boost economy.
ii. It has actual lightweight and small size.
iii. It has many required resources to produce them, and some can be produced through only a small amount of substance.
iv. It has the properties to enhance conductivity of the mechanical and flame barrier of composites and plastics.
v. It allows clean, bulk micro-machines and assembly of parts.

1.3.10. Disadvantages of carbon nanotubes

i. The execution of this new technology would be costly and has been becoming dated thus; it may be gambled to wager on this technology.
ii. Scientists, up until now, don't understand exactly how they work regardless of all the effort in making research.
iii. CNTs are hard to work as a result of their extremely small size.
iv. The production of nanotubes is presently quite costly

1.4. Polyvinyl chloride

Vinyl chloride was first discovered by scientist known as Regnault in 1835. Polyvinyl chloride was used as a polymer a year later. Firstly, polymerization of monomers of vinyl halide in the presence of sunlight, known as poly vinyl chloride leads to the formation of white powder. Since then USA and Germany scientist improved the advancement of PVC polymerization in larger commercial scale through emulsion polymerization in 1930s.

Initially, Sermon import Plasticizer that was modified in 1932 to attain heat strength and handling issues associated to application of polyvinyl chloride. The industrialization of Stabilizer practice was in 1930s; at that instant polyvinyl chloride was the world's main product that was formed on a large scale with its interest rising day by day [1].

PVC is third key manufacture thermoplastic after PP and PE. Its properties, which include antichemical, mechanical and corrosion resistance, brand it to be an adorable substance suspension method is aspect assignment manufacture carried using a raw substance of vinyl chloride monomer (VCM) [1].

It comprises of 42% hydrocarbon and 58% chlorine. It's also manufactured encompasses of industrially produced chlorine of about 35 percent and world's contributed gas and oil of about 0.3 percent. Other polymeric substances are generally hydro-carbons in which it manufactured depends on gas and oil supplies, while PVC production is hardly depended on oil and gas (Fig 1.4). Polymerizations of monomers of vinyl chloride's free radical or techniques of emulsion polymerization are used in the production of Poly vinyl chloride. Solution technique is not applicable due to the polarity of both monomers, and polymers, However, solvent of low boiling point is difficult to achieve. Technique of Emulsion is mostly used due to the supreme adaptation present with no single complexity.

At low temperature, Polymerization of vinyl chloride monomer, can take place using the redox initiator and by water-soluble initiator, sodium persulfate, potassium persulfate, as well as emulsion using hydrogen peroxide. This process gives greater results in contrast to other methods [1].

Polyvinyl chloride can combine with many thermal-organic stabilizers, which includes N-substituted maleimides, pyrazolones, hydroxyl benzylthioether as well as eugenol for specific use in different fields. Its lower mechanical strengths are improved by adding plasticizers. The PVC chemical stabilizers matrix can overcome imaginable decline within the content of the matrix. Stabilizers of lead-based are used for decrease of dehydrochlorination [1].

PVC can be modified by using some chemical and physical methods that included vapour depositing of metals, treating of flame, electrical discharge, plasma-ion beam, and technique of surface-grafting [8]. Chemical modification in PVC matrix is supported via dechlorination, substitution and elimination, grafting and reduction, crosslinking, and degradation mechanisms. By all of the aforementioned mechanisms, reaction by nucleophilic substitution is the most universally common and widely used method [1].

Frequent HCl molecules' loss takes place from the initial stage of monomer of its thermal degradation method. Finally, the formations of conjugated polyene having diverse lengths occur. This structural defects in the matrix of the polymer is as a result of low thermal strength [1].

Polyvinyl chloride can be used in the followings; pipes, wire, imitation leather, consisting of wallpaper, window blinds, coating, plastisols, cables, credit cards, blood bags, audio records, bottles, medical tubing, window frames, packaging and hoses.

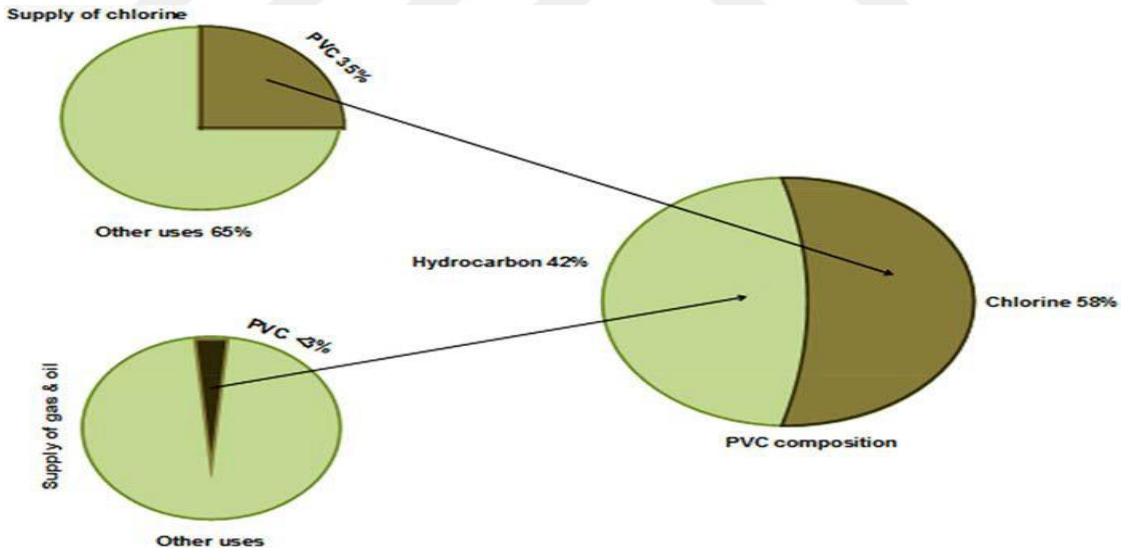


Fig 1.11. Composition of Polyvinyl Chloride. In Percentage

1.5. Polyvinyl Chloride- Based Composites

According to Prusty Et. al. (2013) nanocomposite is defined as a substance of an advanced class in which particles nanosize (filler) are offered into the matrix to enhance

thermal, electrical and mechanical properties. Common fillers examples are; clay, CNT, alumina, GO, silica and graphite [1].

Polymer nanocomposites can be used in the industrial sector. The classification of nanomaterials is based on the nanofillers it composed. These are classified into nanotubes, nanolayers and nanoparticles. These nanofillers are: nanowire, nanodiamond, metal oxide, nanosheet and metal

The remarkable transformations of Polyvinyl chloride-based nanocomposites have been shown as a result of their characteristic properties. Their uses are enough, owing to their light weight, tuneable conductivities, and process ability with simple, straightforward techniques. Such distinctive demonstrate their possibility for use in new applications. Preparation and synthesizing of nanocomposites conducting polymer/graphite presently gained attention due to their potential use in technological field applications, which comprise of super capacitors, electromagnetic shielding, thermistors, catalysis, electrochemical displayers, sensors, rechargeable batteries, antistatic textiles, conductive inks, aerospace, biopolymer plates tin the polymer electrolyte membranes fuel cell, and radar evasion. Aerospace, biopolymer plates tin the polymer electrolyte membranes fuel cell and radar evasion.

In Microelectronic industry, Polyvinyl chloride has a significant role to play because proved to have properties of insulators with lower loss in dielectric [49]. The enhancement of the strength of PVC chain occurs by the link level of molecules between carbon nanotube as well as matrix polymer. However, the mechanical features of polymer/CNT composites are defined. An impossible number of membranes of polyvinyl chloride and selective ion electrodes have produced for the purpose of distinct ions' types [1].

Recently, Nano-substances of carbon-origin attracted considerable attention because they are smaller in size, light in weight, larger and precise surface area, and are in conformity with polymer. Particularly polymer/carbon nanotube improved the hinderance of spreading microcracks. Polyvinylchloride/composites of based carbon nanotube have high prospective for usage. In many.fields, as resistant to wear, composites.can be used in industrially .because of their.strong flame retardancy [49]. The high perspective of PVC

and carbon nanotube-based composites makes them useful in many fields as wear-resistant composites. It can also be used in industries because of their strong flame retardancy [1].

One of the environments friendly uses of PVC is the recycling of plastic materials. The combinations of polyvinyl chloride/graphite oxide nanocomposites have suitability for radar and communication towers performing as a radiation shield. It displayed the conductivity (7.9 x103 s-1m1) for 2wt% GO loading, polyvinyl chloride/graphite oxide nanocomposites because of improvement in charge carriers. They make contact with charge transporter and chain of polymer. Formation of water purification membrane is one of its uses as a result of its excellent properties of polyvinyl chloride (high flux) [1].

Polymeric substance of Poly (vinylidene fluoride) (PVDF) is used for water desalination. Moreover, the nature of PVDF crystal was affected because of unsuitability of PVC. Declination of Porosity, angles contact, and water flux occurred. The polyvinyl chloride/nanomaterials of carbon – based can conquer these shortages and improve the effectiveness of the membrane. Cu composites and polyvinyl chloride/graphite were found to have higher electrical and lower threshold percolation. In addition, it was found that dispersal of fillers of Cu and graphite over the matrix of PVC can serve as semiconductor of n-type. Through the use of neutral carrier base and ion selective electrodes these composites were accomplished for substituting entrants, which are used for electrons' interference and transition of heavy metals [1].

1.6. Methods of polymer/CNT nano-composites preparation

CNTs dispersion in matrix polymer is important matters in the preparation of composites of polymer/CNT. To attain a better CNTs dispersion in the matrix polymer when aggregates do not exist in polymer composites. CNTs dispersion in the matrix polymer can be enhanced by used of many methods such as method of in-situ polymerization, mixing of solution method and blending of melt method.

1.6.1. Mixing of solution

CNTs dispersion is favourable in blended solution of polymers as well as solvent in mixing of solution method. By solvent evaporation or by precipitation composite of polymer/CNT is produced. Formation of CNTs dispersion is a more effective by using a high-power ultrasonic processor rather than dispersed by simple stirring method, which is very difficult. Methods of ultrasonication are broadly used in particles activating, emulsifying, dispersion and crushing. CNTs entanglements, aggregates and ultrasound multi-effects can be effectively prevent their formation by taken its advantage [21].

1.6.2. Blending of melt

The matrix polymer must be soluble in at least one solvent in mixing of solution method, which many polymers are having this difficult. Thermoplastic polymers are generally used for blending of melt method due to it simple as well as common. Dispersion of CNTs in blending of melt methods are by mechanically dispersing into a matrix polymer by the used of high forces of shear blender as well as high room temperature [50]. The blending of melt method is suitable with the present industrial system due to the simple of the method [50]. The forces of shear assist to avoid the creation of aggregates in the nanotube. Different CNT/polymer composites are prepared by using the method of blending of melt such as CNT/polyoxymethylene, CNT/polypropylene, CNT/polyimide and so on. The major short coming of this process is that CNTs dispersion in a matrix polymer is very poor in comparison with dispersion attained by mixing of solution method. Moreover, due to the high viscosities of the composites at higher loaded of CNTs, the CNTs must be lower [21].

1.6.3. Method of in-situ polymerization

Dispersion of CNTs into a matrix polymer is carried out by polymerization. In this method, dispersion of CNTs may be simply dispersing at the higher percentage, which results in the strong interaction with the matrix polymer. The methods of in-situ polymerization are very important during the preparation of composites with polymer, which do not treat by blending of melt method or mixing of solution method. In addition,

attachments of the CNTs to the surface of conducting polymers are by method of in-situ polymerization, which enhances CNTs optical properties, magnetic, processability as well as electrical conductivity [21].

1.7. Graphite

The word graphite is originated from Greek word graphein meaning to write. Plumbago or black lead is also known as graphite. It naturally existed in forms of flake-like or powder comprises of flat, fibrous or spherical shapes. They are categorized into distinct groups based on their sizeable nature, colour, luster, habit and cleavage. The flake graphite comprises a thin sheet of thickness less than 100nm [1].

Alpha (hexagonal) and beta (rhombohedral) are two common types of graphite and are similar in terms of physical properties but differ slightly in terms of grapheme sheets stack. Alpha graphite can either be buckled or flat. It can be transformed into beta forms through mechanical treatment. Beta forms can also be converted into alpha forms when heated above 1300^0C.

The discrete layer and sheet between the covalently carbon atoms found in graphite known as graphene are joined by Vander Waal's forces. The characteristic properties of graphite make it useful for the manufacture of nanocomposite polymer for the acquiring properties that have variety of functions. Carbon atoms in graphite layers are aligning hexagonally. Every layer of the hexagon has 1. 42A distances linking two atoms of carbon and the layers' distance are 3.35A [1].

The distance between the graphite layers is so minor in such a way that compound intercalation is not accessible. Hence, its determination is by distinct physical and chemical processes. The production of graphite intercalating compound using intercalating agent is through distinct chemical species between the layers (Fig 1.6). The Vander Waal's forces decrease as a result of the chemical intercalation of atoms, ions, and molecules into graphite's layers.

Because of the Vander Waal's bond, sigma bond, as well as delocalized pi electrons existed among the graphite's layers, it shows the property of good thermal, electrical conductance as well as a lubricant [1].

Graphene as another form of allotrope of carbon is singularly composed of sheet of atoms of carbon aligned hexagonally in lattice pattern. It is the elementary structural form of several other allotropes of carbon, which include graphite, charcoal, carbon nanotubes and fullerenes [1]. Its strength is owing to its closely packed carbon atoms and sp2 orbital hybridization. The hybridised structure of every carbon atom is joined by covalent bond to three carbon atoms, and each atom has a distance of 1.42 Å [1].

Graphene has unique properties which include electrical conductivity, high thermal conductivity, Young's modulus, mechanical strength, optical transmittance and high theoretical surface area [1]. Graphene has many applications as follows; in energy (solar cells), membranes, composites, coatings, electronics, sensors and biomedical [1].

The following is applications of graphite; refractories, batteries, steel making, brake linings, pencils, electrodes, neutron moderator, expanded graphite, foundry facings and lubricants, scientific research, invention of a process to produce synthetic graphite, powder and scrap.

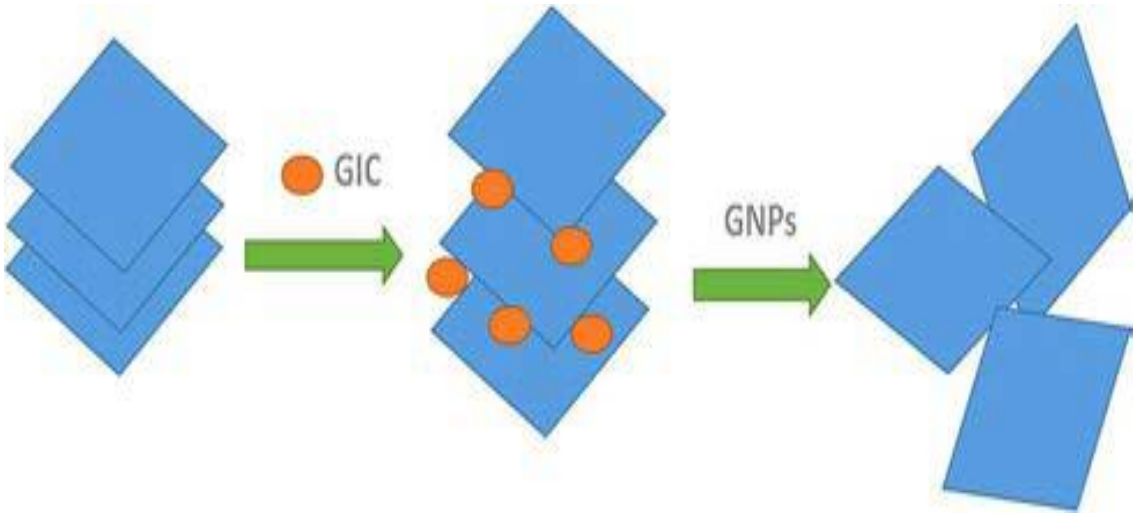


Fig 1.12. Graphite powder prepared through GIC.

GIC note: GNP, grapheme nano-platelet; GIC, .graphite-intercalating.

Table 1.1. Distinct types of nanomaterial have different physical properties1

Properties	Graphite	Diamond	Fullerene	SWCNT
Electrical Conductivity ($S\ cm^{-1}$)	4,000	10^{-2}-10^{-15}	10-5	10^{2}-10^{6}
Electron Mobility ($cm^2\ V^{-1}\ s^{-1}$)	2.0x 10^{4}	1,800	0.5x10^{-6}	~10^{5}
Thermal Conductivity ($Wm^{-1}\ K^{-1}$)	298	900-2,320	0.4	6,000

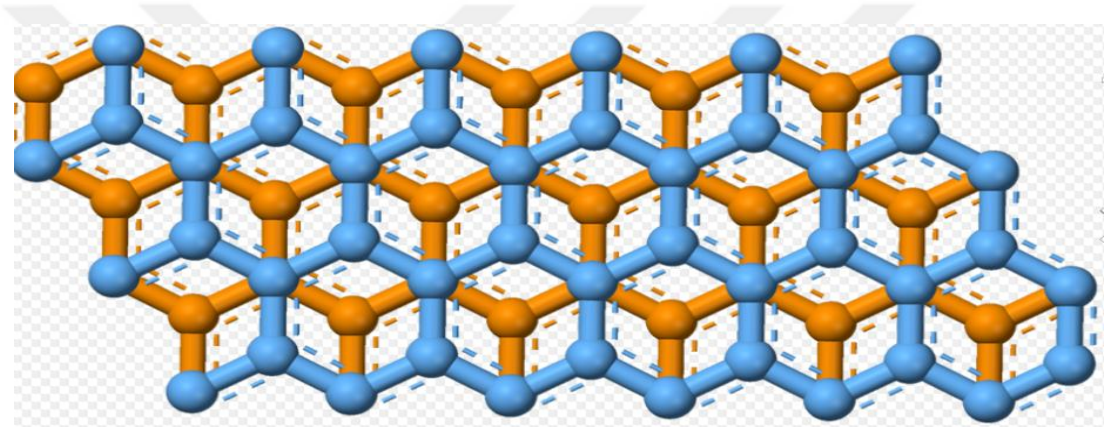


Fig 1.13. Stacking layer of plane view of graphite

1.8. Advantages of graphite

i. They have built-in lubrication and non-galling
ii. They have good thermal conductivity
iii. They are good corrosion resistance
iv. They have Porosity
v. Machinability
vi. They have high compressive strength

1.9. Aim and objectives

The aim of this research is to prepare composites composed of aminated PVC, oxidized multi-wall carbon nanotubes (MWCNT), graphite as well as nanographite. The properties such as electrical, thermal as well as mechanical of these composites are to be measure after homogeneity is achieved. Thus, these properties such as mechanical, electrical as well as thermal stability of PVC, which are very common in uses and production, which will be examined to improve material properties.

The objectives of this research will be achieved through the followings:

i. By preparing composites of PVC carrying functional groups (amine and carboxylic acid), graphite and nanographite.

ii. The composites will be prepared in different percentages in PVC matrix.

iii. The prepared composites will be characterized by FT-IR spectra, DSC, TGA and SEM EDEX.

iv. The properties such as electrical conductivities (in AC and DC), dielectrical constant, thermal and mechanical of these composites will be measured.

1.10. Literature Review

Scientists have made several studies on the mechanical, thermal and electrical properties of aminated polyvinyl chloride as well as oxidized MWCNTs composites.

The flexibility and conductivity of thin film of composite of CNTs/PVC and its mechanical strength as well as how stable it may be thermally was investigated by Annu *Et al* (2014). He found that thermal characteristics of films' composite indicate the polymer glass transition temperature to increase, affirming the enhancement of their thermal stability. The percolation threshold of films of the composite is as low as 0.6vol% with a highest electrical conductivity (0.058 S/cm) at 6.47 percentage volume on carbon nanotube loading [22].

The electrical and mechanical properties of loaded PVC was studied by Aljaafari *Et al* (2011). He observed the increase in Young's modulus concentrations of 1wt% CNTs and 2wt% CNP by up to 2.3 times and 1.4 times of their mechanical properties respectively. There is an initial increase in the tensile strength to the concentrations of 1wt% of CNTs

and 2wt% CNP, which later decrease with increasing of nanofiller. moreover, he observed the decreased strain at the break with increase in both filler in host of the PVC. The storage modulus' character, viscosity complex at 0.1 Hz frequency at room temperature with that of the elastic modulus for PVC/CNTs composites are comparable. The PVC/CNP modulus' storage was different according to the study. The transition temperature of the glass varies with the addition of filler or PVC [23].

Saeed *Et al* (2012). Studied the preparation and properties of PVC/MWCNT nanocomposite. He observed how the neat PVC and nanocomposite showed rapid solvents uptake at the initial stage, and turn to equilibrium with time. He realized that solvent uptake of F-MWNTs was lower than pure PVC [24].

Trommer *Et al* (2014). Investigate on the properties and processing of CNTs/PVC composites [25].

I.S., Elashmawi *Et al,* (2017) investigated the nanocomposite based PVDF/PVC characterization and Preparation based on doped with nanoparticles-based graphene. They prepared nanoparticles of PVDF/PVC based nanocomposites containing oxide of graphene (GO). This analysis showed that by the use of sonicator, the addition of graphene oxide prompts crystal transformation of ά¬ phase of PVDF. the decrease in activation energy with an increase in GO content was observed [26].

Z. Al- Ramadhan *Et al (2012).* Synthesized carbon nanotubes polyvinyl chloride composites and study its electrical properties by using different weight percentage of carbon nanotubes. He observed that the electrical conductivity increases by increasing percentage weight of carbon nanotubes the change of the DC electrical conductivity with increase in temperature for different concentration of carbon nano tubes were observed, and the activation energy of the DC electrical conductivity decrease by increased in concentration of carbon nano tube as well [27].

Dielectric properties and Piezoelectric of PVC/MWCNT and graphite doped with PVC/MWCNT composites was studied by X.-f. Liu *Et al* (2006). He observed that dielectric constant value (£r) increases with the increase of MWCNT volume fraction (Fpzt) at 1000HZ and at a higher frequency, the value remains constant [28].

I.Jilassi *Et al* (2016). Investigate electrical conductivity and dielectric properties of PVC/MWCNT doped lithium phosphate glasses. He used complex impedance spectroscopy for measuring the properties of electrical and dielectric of PVC/MWCNT doped lithium phosphate glasses. IR investigation shows the PVC/MWCNT content

gradual increase in on the structure of the glass. For the impedance analysis he observes a good conformity between experimental and theoretical curve, the result also reveals that the glass sample's conductivity observed to increased by the increase in temperature. The activation energy increased as well by the increase of MWCNT [29].

Thermomechanical as well as electrical properties of segregated nanocomposites based on MWNT/PVC was studied by Ye.P. Mamunya *Et al* (2010). He investigates the electrical properties which are examined on thermomechanical properties, DC and AC of MWCNT/PVC nanocomposites. He segregated the composites of MWCNT/PVC by the method of hot compacting. Conductivity measurement of DC and AC reveals an existence of threshold ultralow percolation (QC value of 0.045vol %). Conductivity temperature dependence shows a switch from the pure PVC ionic conductivity and in filled PVC below percolation threshold to the electron type of the charge transport along the MWCNT phase in the composites above percolation threshold. The analysis of thermo-mechanical of the polymer matrix indicated the rigid network of filler in the volume of the polymer matrix to be formed, whereby the formation of the polymer into the plastic state is inhibited [29].

The thermophysical and electrical behavior of MWCNT/PVC nanocomposites was studied by Ye.Mamunya *Et al* (2007). They investigate the concentration dependence of electrical and thermophysical behavior of composites based on MWCNTs filled with PVC [30].

The MWCNT/PVC nanocomposites electrical as well as thermophysical behaviour was investigated by Ye.Mamunya *Et al* (2008). They investigate the concentration dependence of electrical and thermophysical composites behaviour of composites based on filled MWCNTs with PVC [30].

The transition temperature of plasticized PVC correlation of glass using a model of lattice fluid was investigated by Todd M. *Et al* (2003). He developed a model for estimating the transition temperature of glass polymer + mixtures of plasticizer. He discovered that the modelled polymer matched the experimental data composition dependence of equal to 30 w percent di(2¬ethylhexyl) phthalate. The model was also capable to match the efficiency parameter of the parabolic dependence qualitatively on the length of alkyl chain for linear alkyl phthalates. He concluded that from methyl to n¬butyl phthalate, the efficiency increases which decreases by the increase in molecular weight. However, the efficiency of plasticizer was also able to be estimated by the model for a

wide range of plasticizers e.g PS (R2 = 0.87). Moreover, 0.62 slope of the regression line shows that the plasticizer efficiency was underestimated consistently by the model [31].

Abdullah *Et al* (2012). Study the optical properties of PVC¬ MWCNT nano composites. He fabricates the nano composites by casting method, whereas the optical properties of PVC-MWCNT nano tubes were investigated using different concentration in wt %. His results indicate that the absorbance within near uv region is high. However the Absorption coefficients, extinction coefficient, refractive index, real and imaginary part of dielectric constants were increased with the MWCNT content increase as well [32].

A.T. Abdullah *Et al* (2012). Worked on AC electrical and dielectric properties of PVC-MWCNT nanocomposites. He studied AC electrical conductivity of PVC and PVC/MWCNT were studied as a function of Frequency, Temperature and concentration, the concentration in wt% MWCNT nanocomposites was shown a slight increase as compared with the pure sample. The dielectric investigations reveal decrease in the real and imaginary parts of the composite with increasing the MWCNTs concentration [32].

Nafees A. *Et al* (2016). Make a review of perspective on PVC and nanofiller of carbon composites. Their paper mainly reviewed concentrate on PVC/carbon nanofiller composites they observe that the present study focused on synthetic strategy and significance of polyvinyl chloride/graphite, PVC/graphene, PVC/ graphene oxide, and PVC/carbon nanotubes nanocomposites. They observe that nanofiller of graphene oxide nanofiller depicted enhanced dispersion in matrix of the PVC [1].

X.L. Wu *Et al* (2010). Study on grafted polyvinyl chloride MWCNTs through alkylation process of Friedal¬ Crafts. A model approach was developed for the modification of MWCNTs surface with high grafting percentage (PG %) by the grafting of polymerization by means of Friedal craft alkylation. The graft reaction condition were optimize with PVC, and catalytic anhydrous aluminium chloride in chloroform. The analysis by using FT-¬IR, Raman, and TGA indicated the successful grafting of PVC onto MWCNTs at the ends and on the sidewalls by the Friedal crafts alkylation proposed process. Suggestion has been made that the organic solvents could serve as dispersal medium and would be more stable for PVC grafted MWCNTs [33].

Asrar A. S. *Et al* (2015). Study on dielectric properties of PVC¬MWCNTs polymer nanocomposites. In the study the ranges of 5% 10% 15% up to 40% of weight percentage of MWCNTs were added to PVC powder , measurement of properties of dielectric at room temperature carried out at different frequencies. It was observe that there is increase in

dielectric constant with the increase in carbon nanotube content and decrease with increase of frequency as well. The AC conductivity was also increased with the increased in concentration of MWCNTs [34].

Enhancement study of the thermal and mechanical properties of polyurethane/polyvinyl chloride by A.M. Hezma *Et al* (2017) blended by loading MWCNTs was investigated. They studied the mechanical, structural, as well as thermal properties of pure blended nanocomposites polyurethane-based (PU) and PVC doped with low different content of single walled¬carbon nanotube (SWCNTs). The different concentration of nanocomposites was prepared via casting techniques. X¬ Ray Diffraction results revealed that there is increase in amorphous domain of nanocomposites with increase in the content of SWCNTs. Observation through Transmission electron microscope (TEM) revealed that the polymer wrapped the surface of SWCNTs with the improvement of thermal properties of nanocomposites. The nanocomposites mechanical behaviour was evaluated as a function of content of SWCNTs [35].

A.N Naje *Et al* (2013). Study on the CNT-PVC composites preparation at 5%, 10% up to 20% of weight percentage of CNT and DC electrical conductivity was measured. It was observed that electrical conductivity increase significantly after 8% CNT.

S.M Hasan *Et al* (2013). Study on the CNT-PVC composites preparation and also optical properties of PVC-MWCNT composites were measured. It was observed that absorption around UV region is high and also absorption coefficient, dielectric constant and refractive index increases with increase in MWCNT content.

Katarzyna Skorezewska *Et al* (2011). Study on mechanical and thermal properties of PVC-MWCNT composites prepared using dispersant additives which includes methyl oleate and oleate acid. It was observed that methyl oleate is very effective in dispersing even at low concentration [36].

Girish M. Josh *ET al* (2014), used GO (grapheme oxide) as reinforcement for PVC nanocompound by colloidal mixing method and he show the fabrication of high performance nanocomposites matrix PVC reinforced is attained by interaction strongly between GO and PVC through covalent bonding. It was also observed that thermal composite film stability is enhanced by the dispersion of GO to the matrix PVC as shown by analysis of TGA and also examined properties of dielectric in the frequency of 50¬35Hz as well as temperature between 40¬150^{0}C . The composite films electrical properties shows stability in polarization all over temperature and frequency ranges [37].

Yashpal singh *Et. al* (2010) studied effect of temperature and frequency on dielectric properties of PVC/MWCNT. He observed that the dielectric constant values increases with increase in temperature. The dielectric loses and dielectric constants maximum value was due to the polymer transition phase. The net effect of some internal field within the polymer along with the external A.C. field was due to the variation in loss tangent t and dielectric constant [37].

The variation of properties of PVC-MWCNT composites depending on MWCNT concentration when volume percentage of nanotube was above 0.05%.

Athar Javed Et al (2005), studied the dielectric loss as well as the dielectric constant in polymer composite and pure polyester at temperature and frequency dependence with various types of glass fiber in the temperature range 25-150 ^{0}C as well as in the frequency range 300 - 3 MHz. He observed that in polyester resin both dielectric constant loss and dielectric constant increased with the concentration of glass fiber increases. By increasing the temperature the value of dielectric constant increased and it is due to the orientation polarization. At high temperature , Tg of polyester shows that the dielectric constant loss peaks in the composite materials. Due to greater movement freedom of the dipole within the polyester chains at high temperature that causes the value of dielectric constant increased with increasing the temperature [38].

2. MATERIAL AND METHOD

Avery Berkel VA 304 digital scale instrument was used to carried out all weighing in this experiment, ultrasonic sonicator FY-US-01 digital instrument was used to carried out sonication, Perkin Elmer spectrum One was used to record the infrared spectra for aminated PVC with oxidized MWCNTs/nanographite composites, Bruker XFlash 6110 digital instrument was used for SEM, which is used to determine the surface structures of the composites as well as elementary analyser, THF was used as a solvent, ethanol was used as a reagent for precipitate, phenolphthalein as indicator, PVC was used as a polymer, MWCNTs as nanotube, nanographite as well as graphite are used as doping agents, functional groups such as amine and carboxylic acid are added to the surface of PVC matrix and MWCNT respectively.

The apparatus used in this experiment are magnetic stirrer, filter paper, oven, round bottom flask, conical flask, measuring cylinder, automatic pipette, spatula, burette, pipette, conical flask and so on.

2.1. Experimental

Firstly, functional group such as amine was dispersed on to the PVC matrix in this method. The composite was prepared in various percentages containing aminated PVC matrix and oxidized MWCNT. Aminated PVC was dissolved in tetrahydrofuran (THF), oxidized MWCNT is added and blended with a sonicator providing sound wave for 1 h 30 min and homogeneous mixture is then formed [32].

The prepared homogeneous composites was then used to make a disk by heating under pressure after thoroughly crushing a powder mixture of aminated PVC with oxidized MWCNT in a proclaim mortar [30] and their properties such as electrical conductivities (AC and DC), dielectrical constants, as well as thermal of the prepared composites were examined. The composites prepared was characterized by SEM, TGA, DSC and FT-IR spectra.

The second phase of the experiment, the prepared composites was doped with nanographite and their properties such as dielelctrical, electrical conductivities (AC and DC) as well as thermal were also be measured and compared them with former composites. The prepared doping composite was characterized by TGA, DSC, SEM and FT-IR spectra.

2.1.1. Preparation of composite of PVC/ 5% aminated MWCNT and doped with 5% graphite

0.05 g of aminated MWCNT was dispersed in 15 mL THF with sonicator providing the wave sound for 24 h and then 0.45 g of PVC was added to the aminated MWCNT disperse solution. The mixture was sonicated for 48 h, then precipitated in ethanol, dried at room temperature for 24 h and then dried again in a vacuum at 45 ^{0}C.

The prepared composite was mixed with 0.05 g of graphite, and then a disk was made by heating under pressure after thoroughly crushing a powder mixture of PVC with aminated MWCNT and graphite in a proclaim mortar [30] and their properties such as AC conductivity, dielectrical constants, as well as thermal properties of the prepared composites were examined. The composite prepared was characterized by SEM, TGA, DSC and FT-IR spectra.

2.1.2. Synthesis of aminated PVC

5 g of PVC was accurately weighted and mixed with 40.40 mL of 3-(Dimethylamino)-1- propylamine and 9.6 mL distilled water in 100 mL volumetric flask. The mixtures are sonicated for 5 min and then passed into argon gases. They are placed into oil bath for 1 h at 80 ^{0}C and then filter it by washing with distilled water. The mixtures are immersed in ethanol for overnight, dried it at room temperature for 24 h and then dried it again in a vacuum at 45 ^{0}C for 48 h [39].

2.1.3. Preparation of composite of aminated PVC with oxidized MWCNT

Firstly, we prepared aminated PVC composite with 2% MWCNT. 0.49 g of aminated PVC was accurately and then dissolved in 10 mL THF. 0.01 g of oxidized MWCNT is

added to aminated-PVC solution and mixed with a sonicator providing a sound wave for 1 h. The resulting composite is precipitated in ethanol and filtered [30].

We used same procedure to prepare aminated-PVC/oxidized MWCNT composites that containing 5%, 8% and 11% oxidized MWCNTs, respectively. All composites prepared were first dried at room temperature and then dried again in a vacuum at 45 ^{o}C for 48 h [40].

2.1.4. Preparation of composite of aminated PVC/ oxidized MWCNT doped with Nanographite

Firstly, we prepared aminated PVC / 8% oxidized MWCNT doped with 1% nanographite. 0.364 g of aminated PVC was accurately weighted and then dispersed in 20 mL ethanol with a sonicator providing sound waves for 1 h 30 minutes. 0.032 g of oxidized MWCNT and 0.004 g of nanographite are added to aminated PVC dispersed solution [30].The mixture was evaporated and then dried in a vacuum at 45 ^{0}C for 48 h.

We used same procedure to prepare composites of aminated PVC / 4% and 12% oxidized MWCNT doped with nanographite containing 1%, 5%, 8% and 12% nanographite, respectively.

3. RESULTS

3.1. Calculation of amination percentage of PVC

3.1.1. Preparation of standard HCl solution

A stock solution of HCl have concentration of (M = 36.46 g/mol), 8.29 mL of the stock solution of HCl was accurately measured and diluted in 1L volumetric flask filled with distilled water to the mark. Three different amount (0.2013, 0.3044 and 0.4123 g) of Na_2CO_3 were added to 10 mL distilled water. Each Na_2CO_3 solution was titrated with 0.1 M HCl. The processes of titration were repeated three times in order to obtain volume of the HCl used. The standard concentration of HCl is calculated by the following equation:

$$\frac{w \times 1000}{106} = V \times M$$

Where w is weight of the Na_2CO_3, V is titre value of the HCl, M is molarity of the HCl used.

The average concentration of HCl was obtained as 0.1108 M.

3.1.2. Preparation of standard NaOH solution

0.4 g of NaOH was accurately weighted and then dissolved in 100 mL volumetric flask filled with distilled water to the mark. Two drops of phenolphthalein are added to the 10 mL of 0.1108 M HCl and then it is titrated with prepared 0.1 M NaOH. The processes of titration were repeated three times in order to obtain the volume of the NaOH used. The standard concentration of NaOH is calculated by the following equation:

$C_1V_1 = C_2V_2$

Where C_1 is the concentration of NaOH, V_1 is the volume of the NaOH used, C_2 is the concentration of the HCl, V_2 is the volume of the HCl used. The average concentration of NaOH was obtained as 0.1079 M

3.1.3. Evaluation, calculation and analysis of percentages of amine in aminated PVC composite

Number of moles HCl $=$ Molarity $\times$ Volume

Number of moles HCl $= 0.1108\ M \times 10\ mL = 1.108\ mmol$

Number of moles NaOH $=$ Molarity $\times$ Volume

Back titration value of NaOH solution = 7.4 *mL*

Number of moles NaOH for back titration $= 0.1079\ M \times 7.4\ mL = 0.7985\ mmol$

Volume of aminated PVC solution used = 10 *mL*

Number of moles of HCl consumed for amine = (number of moles of acid) – (number of moles base);

$1.108\ mmol - 0.7985\ mmol = 0.3095\ mmol$ for two moles of amine

$$\text{Mole number of amine group} = \frac{0.395}{2} = 0.1548\ mmol$$

$$\text{Mass of amin containing unit} = 0.1548\ mmol \times 128 = 19.814\ mg$$

We used 100 mg of aminated PVC,

$$\text{Mass of unreacted PVC} = 100\ mg - 19.814\ mg = 80.186\ mg$$

$$\text{Number of moles of unreacted PVC} = \frac{\text{Mass of the PVC group}}{\text{Mer weight}}$$

$$= \frac{80.186}{62.5} = 1.283\ mmol$$

$$T\text{otal number of moles amin and chlorine group} = 0.1548 + 1.283 = 1.4378\ mmol$$

$$\text{Percentages of aminated group} = \frac{\text{Number of moles of amine group}}{\text{Total number of moles}} \times 100$$

$$= \frac{0.1548\ mmol}{1.4378\ mmol} \times 100 = 10.8\ \%$$

3.2. Characterization of PVC and Aminated PVC

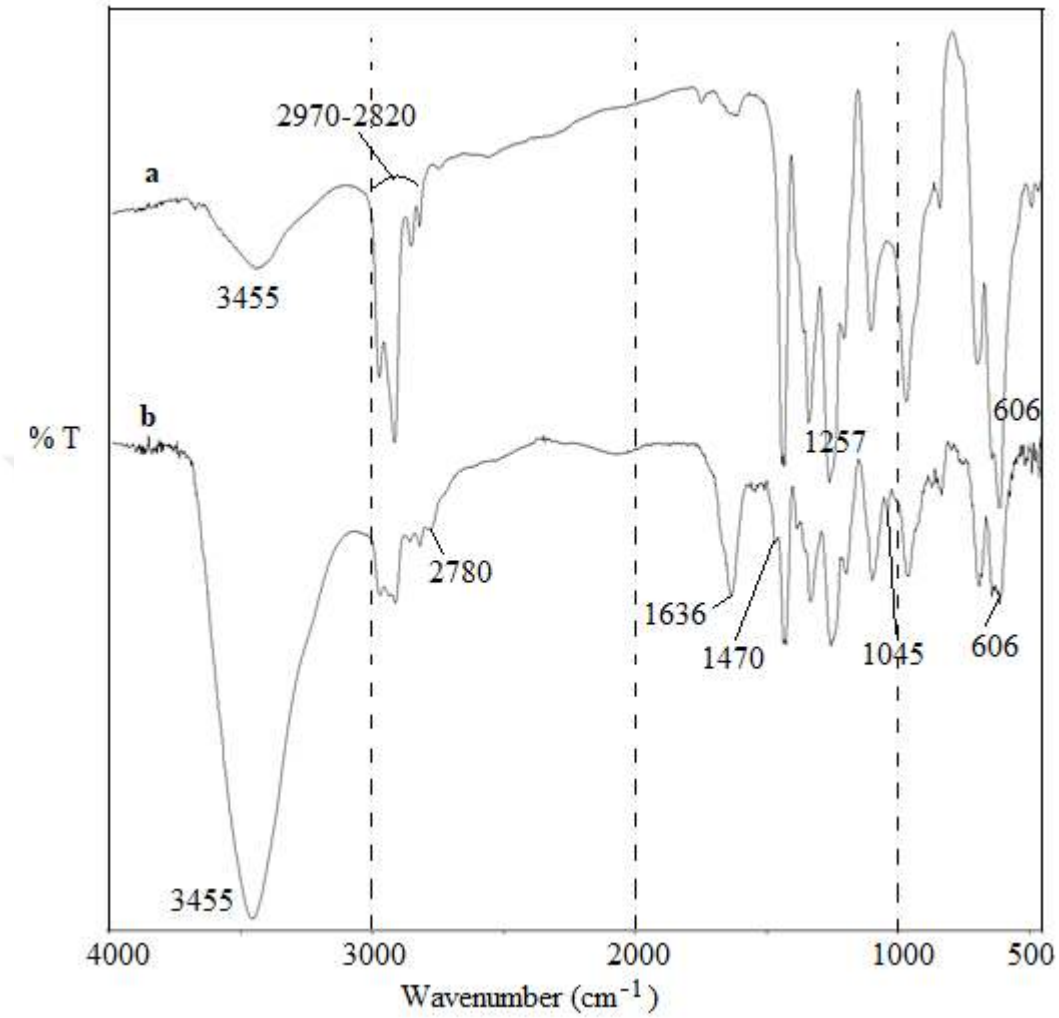
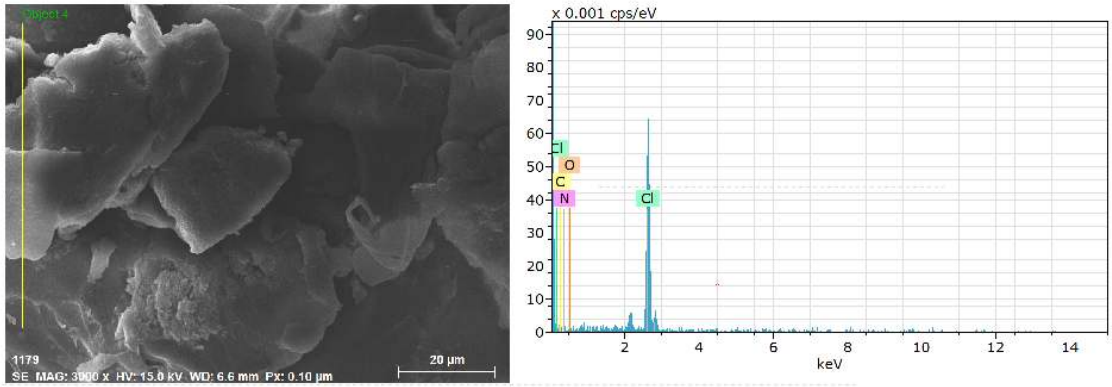


Fig 3.1. FT-IR spectra of (a) PVC and (b) aminated PVC (KBr disc)

FT-IR spectrum (Figure 3.1a) of PVC showed characteristic bands at 2970-2820 cm-1 (aliphatic C-H stretching vibration on CH_2, CH), 1257 cm^{-1} (C-H bending vibration on –CHCl-) and 606 cm^{-1} (C-Cl stretching vibration). In addition, bands of water coming from KBr, which is unrelated to the structure, is seen in the O-H stretching band at 3455 cm^{-1} and the O-H bending band 1636 cm^{-1}. FT-IR spectrum (Figure 3.1b) of aminated PVC showed characteristic bands at same wavenumbers with PVC including water bands. Aminated PVC showed weak bands related to the amine group attached to PVC at 2780 cm^{-1} (C-H stretching vibration on CH_3-N), at 1470 cm^{-1} (aliphatic C-N stretching vibration band) and at 606 cm^{-1} (C-Cl stretching band relatively weaker than that of PVC). The N-H band of aminated PVC is probably under the O-H stretching band.



Spectrum: Object 4

El	AN	Series	Net unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
Cl	17	K-series	695	24.58	55.17	30.19	1.40
C	6	K-series	20	14.95	33.55	54.19	14.97
N	7	K-series	6	5.02	11.28	15.62	8.42
O	8	K-series	0	0.00	0.00	0.00	0.00
		Total:		44.55	100.00	100.00	

Fig 3.2. SEM-EDX results of aminated PVC

SEMEDX Results of aminated PVC shown in Figure 3.2. From the SEM-EDX data it is seen that the atomic percentage of the nitrogen element in the aminated PVC is 15.62%. It can be estimated that the molar percentage of the aminated units in PVC is 7.81 since 2 nitrogen atoms are present in the aminated units in the aminated PVC. It is relatively comparable to the% 10.8 amination percentage found by titration. However, it can be said that the value found in the titration is closer to the true value. Because the SEM-EDX can be computed from the image taken from a specific region, it can be a zone where the amination is intense or less.

3.3. Aminated PVC/oxidized MWCNT Composites

Composites containing 2%, 5%, 8% and 11% (by weight) oxidized MWCNT of aminated PVC were prepared.

3.3.1. Characterization of aminated PVC/oxidized MWCNT Composites

FT-IR spectrum of oxidized MWCNT (Figure 3.3) showed characteristic bands at 3330 cm^{-1} (O-H stretching in carboxyl group), at between 2970-2860 cm^{-1} (aliphatic C-H stretching vibration), 1727 cm^{-1} (C=O stretching band), 1636 cm^{-1} (O-H bending in probably water), 1575 cm^{-1} (C=C stretching in MWCNT) and 1180-1030 cm^{-1} (C-O stretching).

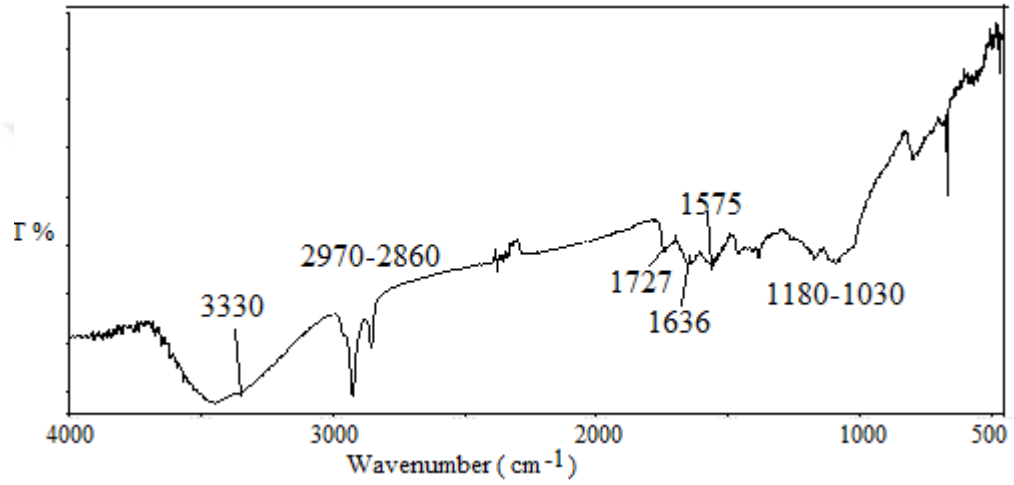


Fig 3.3. FT-IR Spectra of oxidized MWCNT

FT-IR spectrum of aminated PVC/11% oxidized MWCNT composite (Figure 3.4) showed characteristic bands at 3455 cm^{-1} (O-H stretching in water) 3300 cm^{-1} (as shoulder, O-H stretching in carboxyl group), at between 2970-2860 cm^{-1} (aliphatic C-H stretching vibration in aminated PVC and oxidized MWCNT), 1727 cm^{-1} (C=O stretching band in oxidized MWCNT), 1636 cm^{-1} (O-H bending in probably water), 1575 cm^{-1} (C=C stretching in MWCNT), 1180-1030 cm^{-1} (C-O stretching) and the 606 cm^{-1} band of PVC, which is not seen in the spectrum of MWCNT, is attributed C-Cl band in aminated PVC.

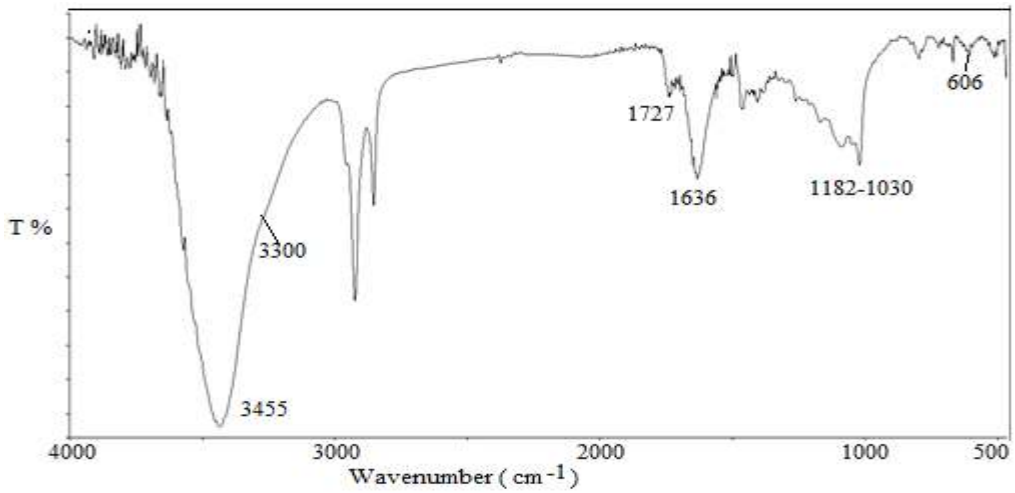
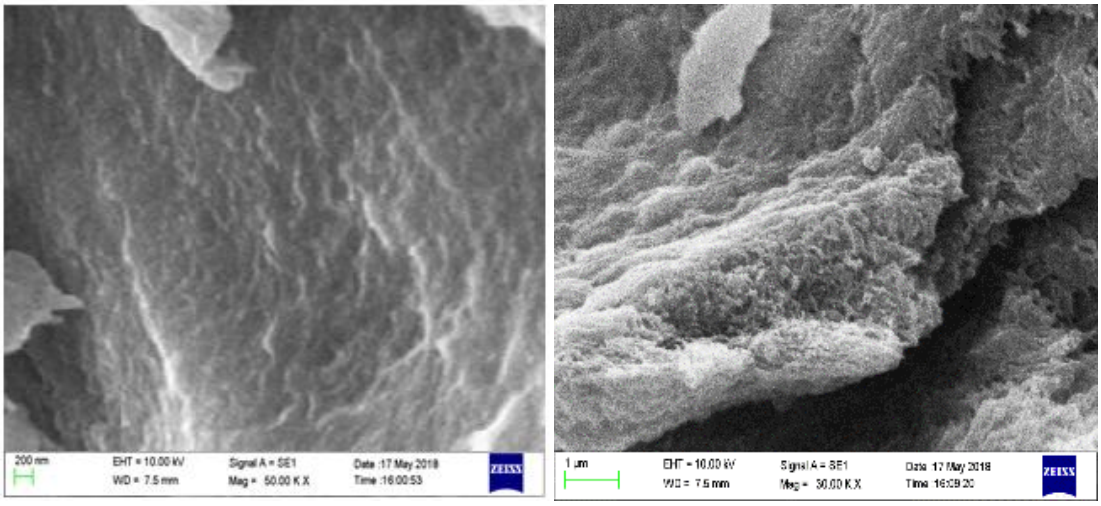


Fig 3.4. FT-IR Spectra of aminated PVC / 11% oxidized MWCNT Composite

SEM images of aminated PVC/5% oxidized MWCN composite (a) and aminated PVC/11% oxidized MWCN composite are seen in Figure 3.5a and b, respectively. The SEM image of the composite containing 5% oxidized MWCNT shows that most of the oxidized MWCNT rods are well dispersed in the aminated PVC matrix, with a few minor irregularities. Figure 4.5b shows one of the fractured zone, which reveals the well dispersed oxidized MWCNT rods in the composite. The length of the rods is not very well chosen because they are embedded in the polymer matrix.



(a) **(b)**

Fig 3.5. SEM picture of aminated PVC/ 5% oxidized MWCNT composite (a) and aminated PVC/ 11% oxidized MWCNT composite (b)

3.3.2. Thermal investigation

Differential scanning calorimeter (DSC) curves of PVC, aminated PVC and composites heated at 10 °C/min are given in the Figure 3.6. Thermogravimetric analyisis (TGA) curves of PVC and aminated PVC are in Figure 4.7, and those of the composites are in Figure 4.8. DSC measurements show that amine substitution reaction in PVC reduced the glass transition temperature (T_g) of PVC from 84.7 °C to 79 °C. The addition of oxidized MWCNT to aminated PVC increased the T_g temperature of PVC, as details are shown in Table 3.1.

TGA curve of aminated PVC shows that after a weight loss of 4.7% from 100 °C to 133 °C, rapid decomposition started at 176 °C. In case of PVC rapid decomposition began at 248 °C (T_i, initial decocmposition temperature). Amination reaction reduced thermal stability of PVC. Oxidized MWCNT increased slightly thermal stability of aminated PVC, but still thermal stabilities of the composites are lower that of PVC. The results obtained from TGA curves are given in Table 3.1 in detail.

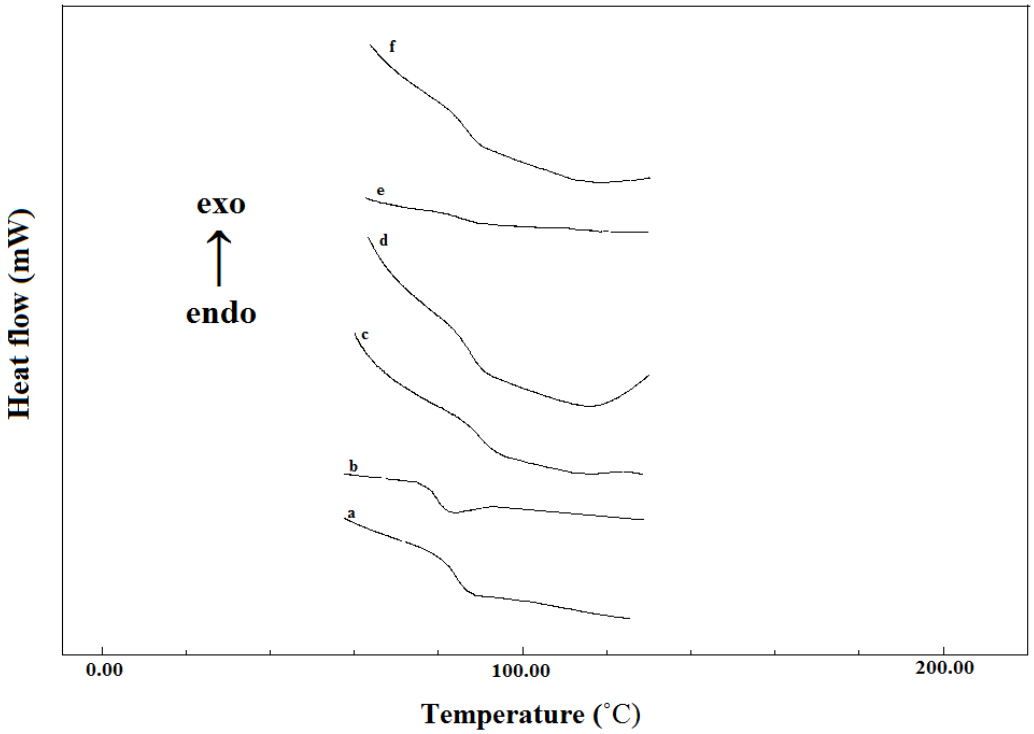


Fig 3.6. DSC curves of PVC (a), aminated PVC (b) and oxidized MWCNT concentration (wt%) in aminated PVC/ oxidized MWCNT composites, 2% (c), 5% (d), 8% (e), 11% (f) (second cycle heating temperatures)

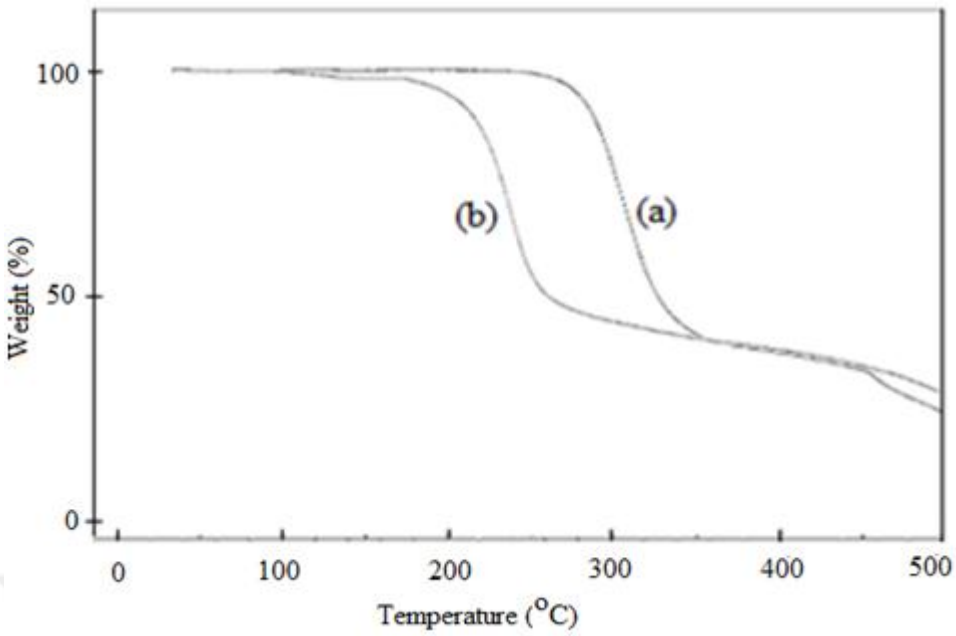
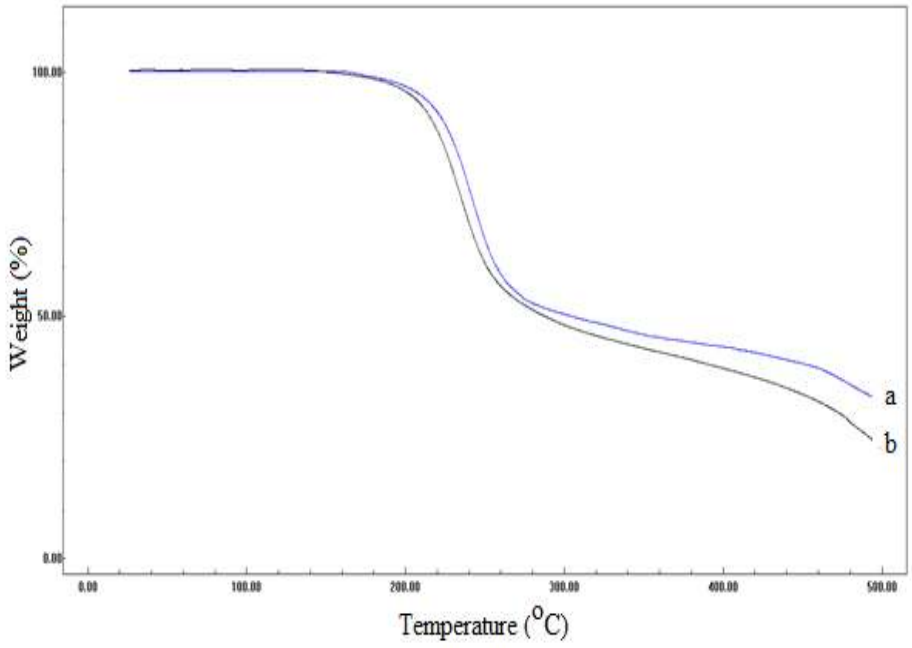


Fig 3.7. TGA curves of a) PVC and b) aminated PVC



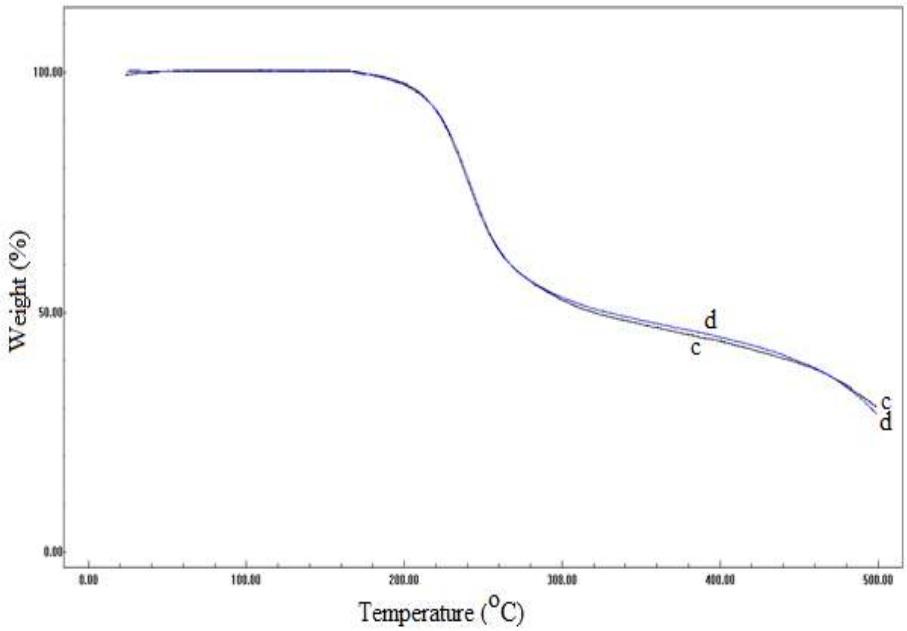


Fig 3.8. TGA curves of composites of aminated PVC/ oxidized MWCNTs, Oxidized MWCNT contens (wt%) : (a) 2%, (b) 5%, (c) 8% and (d) 11%

Table 3.1. Thermal investigation of PVC, aminated PVC and composites containing oxidized MWCNT at different percentages

Material	Tg (oC)	Ti* (oC)	50 % wt loss at tem. (oC)	Weight loss (wt) at 300 oC	Residue (wt%) at 500 oC
PVC	84.7	260	329	22.9	24.2
Aminated PVC	79.0	209	262	53.3	29.0
Aminated PVC/Oxidized MWCNT (wt %)					
2	90.3	217	307	51.0	33.8
5	89.5	209	286	47.6	25.0
8	85.5	212	319	52.5	29.8
11	87.1	212	331	53.3	28.3

*Initial decomposition temperature: The temperature at which rapid decomposition begins

3.3.3. Elecrical investigation

For dielectric constant, dielectric loss and AC conductivity measurements, samples having 1.32 cm^2 area were used for all composites. Sample thickness is 0.051 cm, 0.067 cm, 0.067 cm and 0.040 cm for the composites containing 2%, 5%, 8% and 11% (by weight) oxidized MWCNT, respectively.

3.3.3.1. Dielectric constant, Dielectric loss

One of the most important properties used to determined A.C. conductivity and it is determine by using the following equation

$$\boldsymbol{\varepsilon'} = \frac{C_p}{C_o} \qquad \boldsymbol{\varepsilon'} = \frac{C_p.d}{\varepsilon_0 A}$$

Where **ε'**= dielectric constant, C_p= capacitance of dielectric filled capacitor, C_o= free space capacitor, d= thickness of sample, A= surface area of sample and the dielectric constant is measured at different frequency and temperature. Variation of dielectrical constant of composites of aminated PVC/oxidized MWCNTs with frequency at room temperature are seen in Fig 3.9. This figure shows that dielectric constant values decrease with increasing of the applied field frequency increasing , between 100-500 Hz rapid for composites contaning 2% and 5% of oxidized MWCNT, and between 100-800 Hz rapid for compsites containing 8% and 11% of oxidized MWCNT, then remain constant.

Dielectric loss is also one of the parameter used to determined electrical properties and can be measured by using the following equation

$$\text{Tag } \delta = \frac{\varepsilon''}{\varepsilon'}$$

Where tan δ= dielectric loss factor, **ε''**= dielectric loss and **ε'**= dielectric constant. Dielectric loss result from the inability of polarization process in a molecules to follow the rate of change of the oscillating applied electric field. This arise from the relaxation time (τ) in a polymer which is the time taken for the dipoles to return to its original random orientation. It does not occur instatntaneously but the polarization diminished exponentially. If the relaxation time is smaller or comparable to the rate of oscillating electric field, then there would be no or minimum loss.

The dielectric loss was measured by depending on frequency (Figure 3.10) and temperature (Figure 3.12). The dielecric loss of composite containing 11% of oxidized MWCNT which has higher dielectric constant decreases, rapidly up to 500 Hz, slowly from 500 to 2000 Hz, with increasing frequency. At the other composites, the dielectric remain nearly constant with increasing frequency.

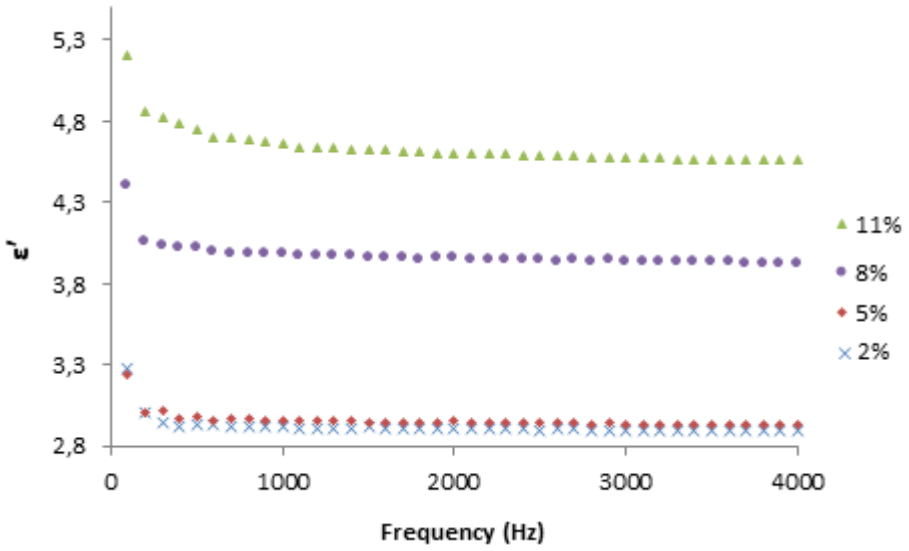


Fig 3.9. Variation of dielectrical constant of composites of aminated PVC/oxidized MWCNTs with frequency

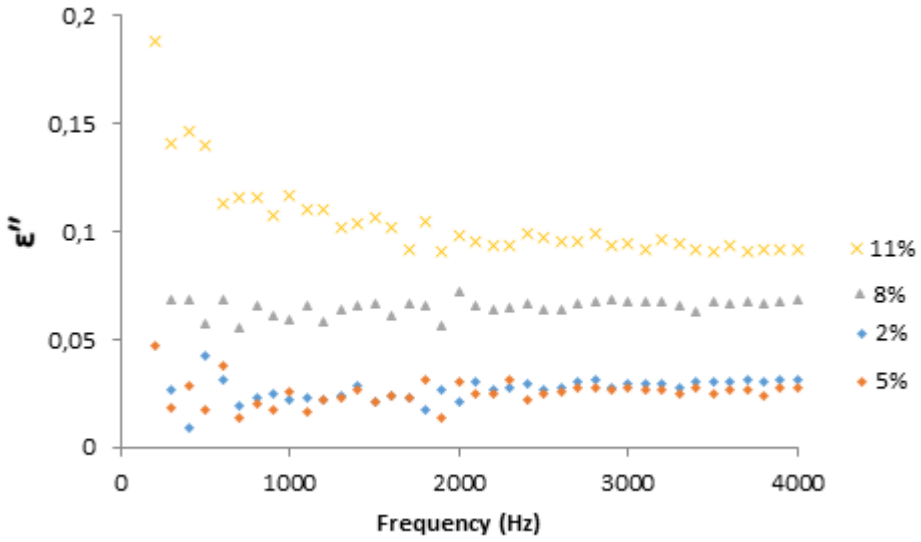


Fig 3.10. Variation of dielectrical loss of composites of aminated PVC/ oxidized MWCNTs with frequency

Temperature dependence of dielectric constants are seen in Figure 3.11. The Figure shows that the dielectric constant increase with increasing temperature for the composites, except composite containing 11% of oxidized MWCNT. For 8% composite the increase is relatively slow upto 93 ^{o}C, after that it is rapid. For 2% and 5% composites the increase is relatively slow upto 95 ^{o}C , then it is rapid. Thes temperatures are similar to the glass transition temperature of polymer in composites.

Temperature dependence of dielectric losses are seen in Figure 3.12. The Figure shows that the dielectric loss for containing 8% and 11% oxidized MWCNT remain nearly constant up to 105-110 ^{o}C, but it increases rapidly after nearly 92 ^{o}C for the composites containing 2% and 5% oxidized MWCNT.

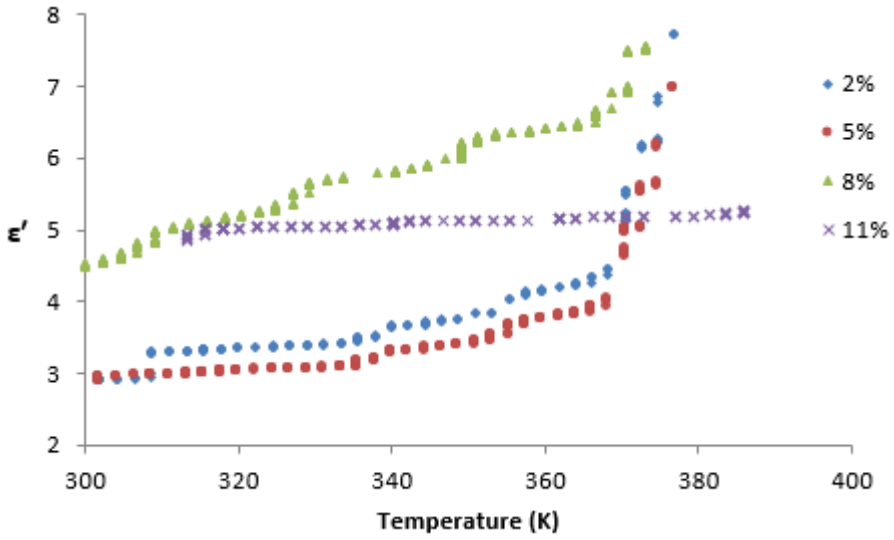


Fig 3.11. Variation of Dielectric constant of composites of aminated PVC/oxidized with temperature

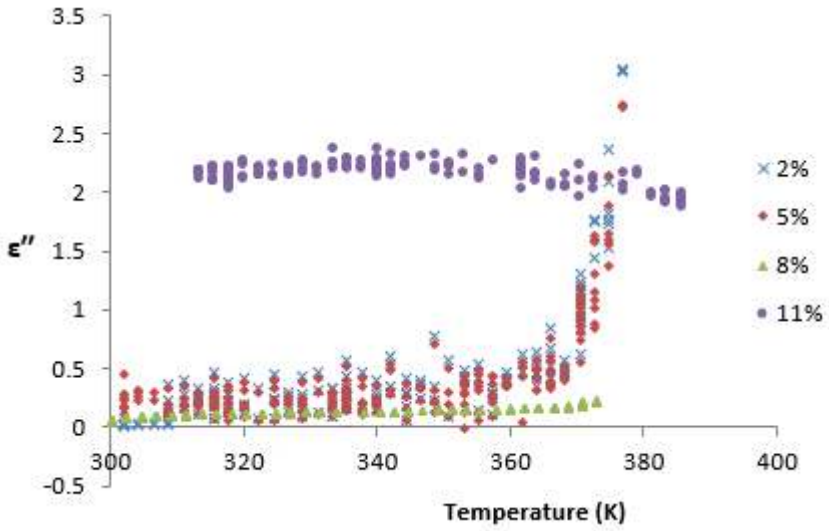


Fig 3.12. Variation of Dielectric loss of composites of aminated PVC/ oxidized MWCNT with temperature

3.3.3.2. A.C. Conductivity

A.C. conductivity is another parameter used to determine electrical properties and can be measured by using the following equation

$\sigma_{ac} = \omega \varepsilon'' \varepsilon_o$

where σ_{ac} = A.C conductivity, $\omega = 2\pi f$ and ε_o = permittivity at free space.

The A.C. conductivity is measured at different temperature and frequency. For the composites, variation of A.C. conductivity with frequency is shown in Figure 3.13. A.C. conductivities for the all composites increase rapidly with increasing frequency up to 800 Hz, then they increase slower up to 4000 Hz at room temperature.

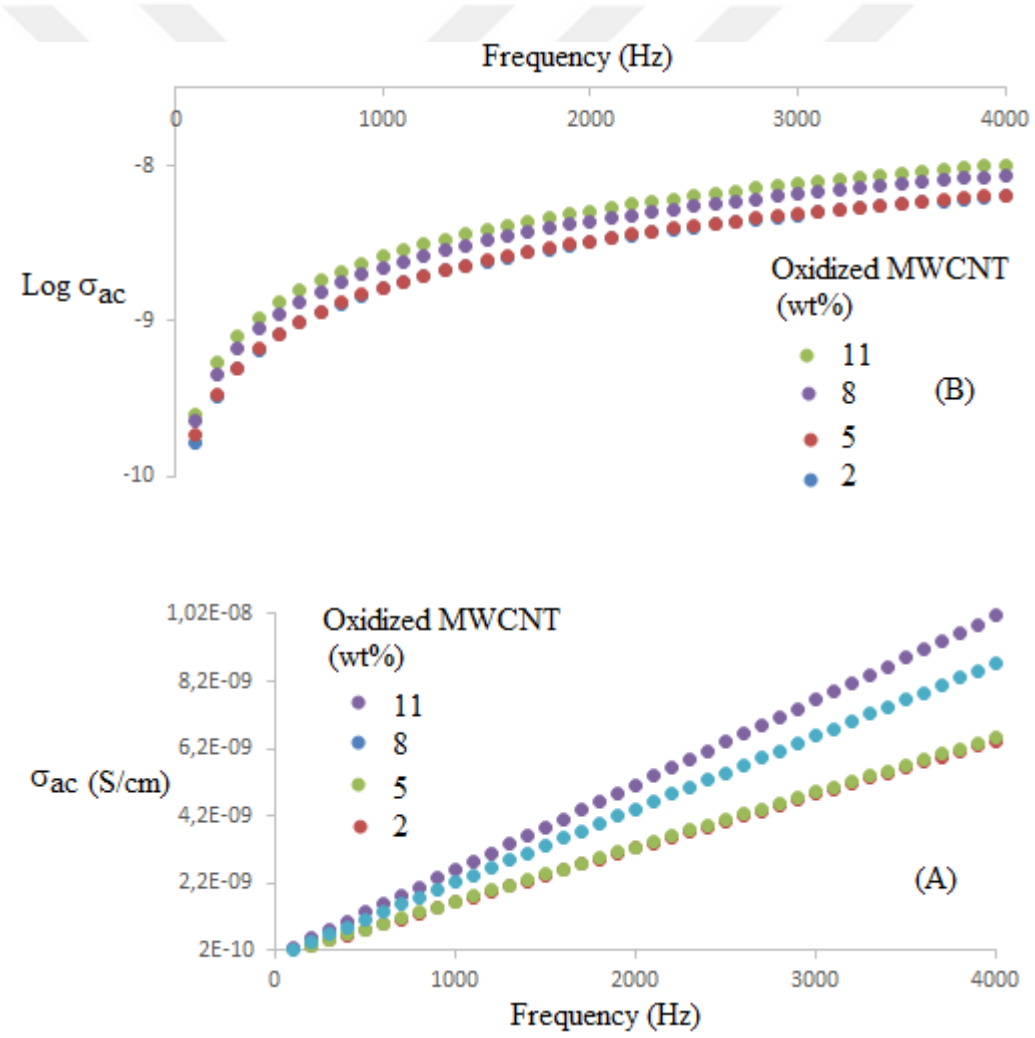


Fig 3.13. Variation of A.C. conductivity of composites of aminated PVC/ oxidized MWCNTs with frequency, (A) σ_{ac}-frequency, (B) Log σ_{ac}-frequency

Variation of A.C. conductivity of aminated PVC/ oxidized MWCNT composites with temperature is seen at Figure 3.14. The 2% oxidized MWCNT-containing composite showed a rapid increase in AC conductivity from room temperature to about 35 °C (308 K). Then, after a very slow increase between 35-92 °C, it was seen to increase again rapidly. The A.C. conductivity of the composite containing 5% oxidized MWCNT increased more rapidly after increasing slowly from room temperature to 92 ° C. A.C. conductivities of composites containing 8% and 11% oxidized MWCNTs have been observed that the temperature did not change with the temperature.

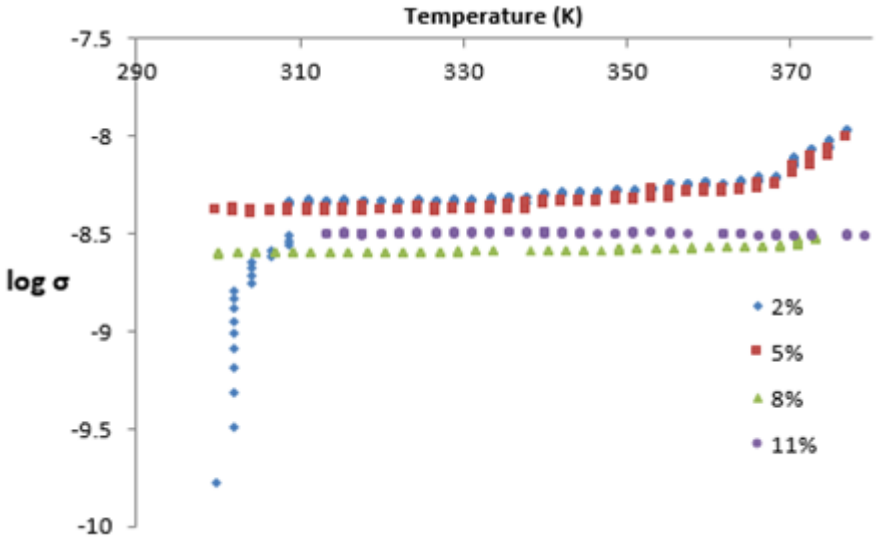


Fig 3.14. Variation of A.C. conductivity of aminated PVC/ oxidized MWCNT composites with temperature at 1 kHz

3.4. Doping of aminated PVC / oxidized MWCN composites with nanographite

Composites containing oxidized MWCNT in different weight percentages were doped with nanographite in different percentages (1 %, 3 %, 5 %, 8 % and 12% by weight).

3.4.1. Characterization of aminated PVC / oxidized MWCNT composites doping with nanographite

FT-IR spectrum of aminated PVC/11% oxidized MWCNT composite doped with 12 wt% nanographite is seen in Figure 3.15. The FT-IR spectrum of doped composite is

similar with that of undoped composite due to the both composite contains similar kinds of hydrocarbon groups [41].

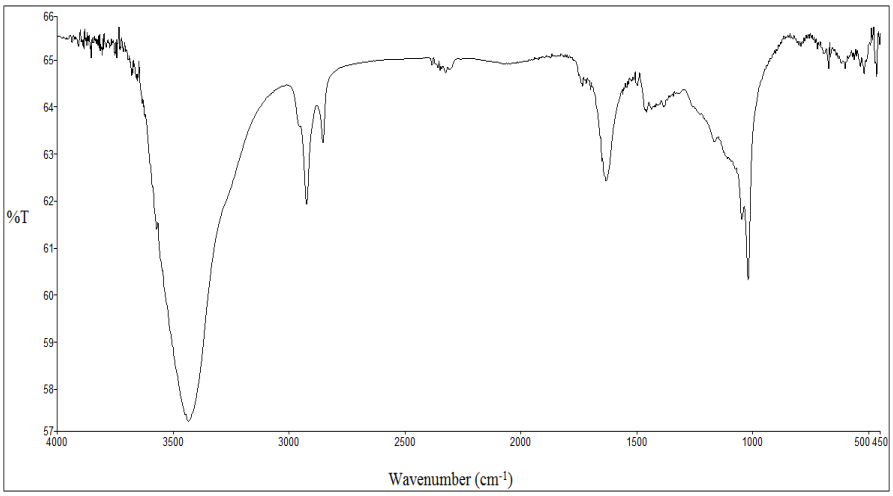


Fig 3.15. FT- IR Spectra of aminated PVC / 11% oxidized MWCNT composite doping with 12% nanographite

SEM pictures of aminated PVC/ 11% oxidized MWCNT composites doping with a) 3%, b) 12% nanographide are shown in Figure 3.16. SEM images for both composites were taken at 50,000 magnifications. SEM images show that the oxidized MWCNT and nanographite particles are homogeneously dispersed in the aminated PVC matrix.

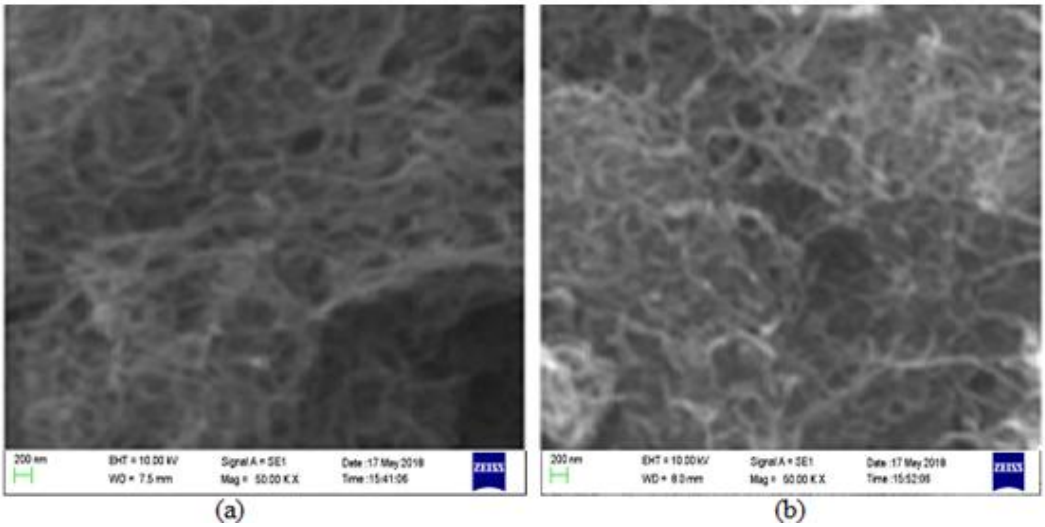


Fig 3.16. SEM pictures of aminated PVC/ 11% oxidized MWCNT composites doping with a) 3%, b) 12% nanographite

3.4.2. Thermal investigation

DSC curves of composites of aminated PVC/ 5% oxidized MWCNTs doped with nanographite are seen in Figure 3.17. The glass transition temperatures determined based on the midpoint of the glass transition zone are given in Table 3.2. While the T_g temperature before graphite addition was 89.5 ^oC, the Tg temperatures of graphite-doped composites at different percentages were found between 88.4 and 90.7 ^oC. In other word, graphite which added to composites, did not significantly affect Tg temperatures.

TGA curves of aminated PVC/5 wt% oxidized MWCNT composite doped at different percentages with nanographite are seen in Figure 3.18. Initial decomposition temperature (T_i) for all composites are around 164 ^oC and it is not depedent to the graphite content. It has been observed that the weight loss at 300 ^oC and the residual amount at 500 ^oC increased as depending on the increasing graphite content.

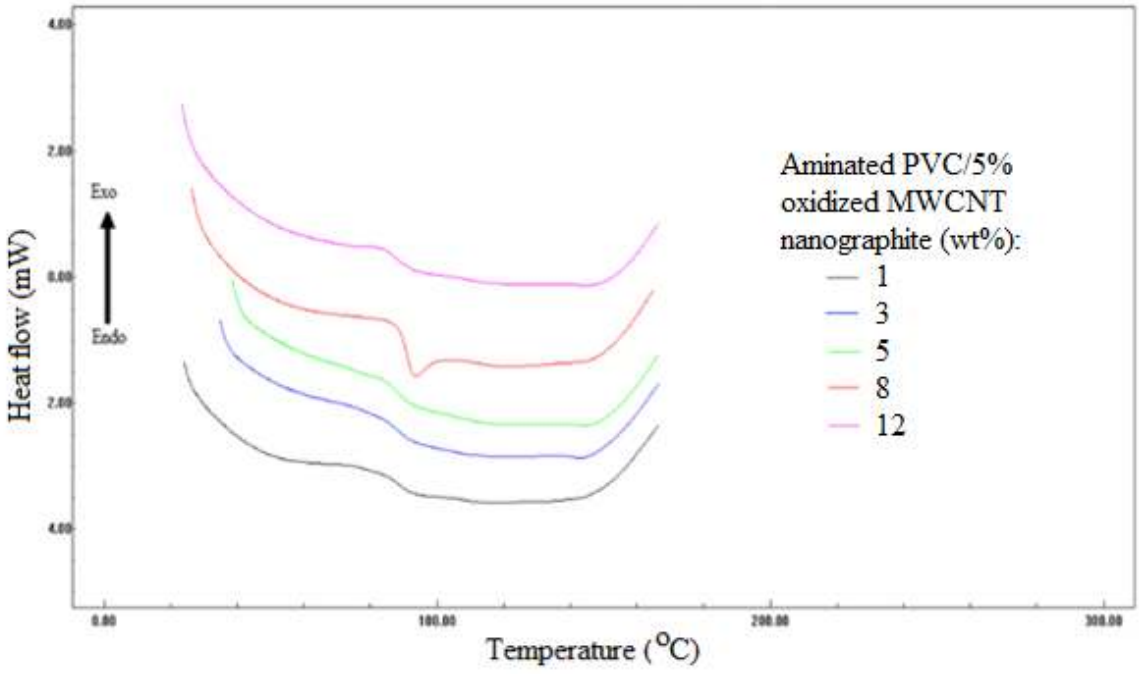


Fig 3.17. DSC curves of composites of aminated PVC/ 5% oxidized MWCNTs doped with nanographite

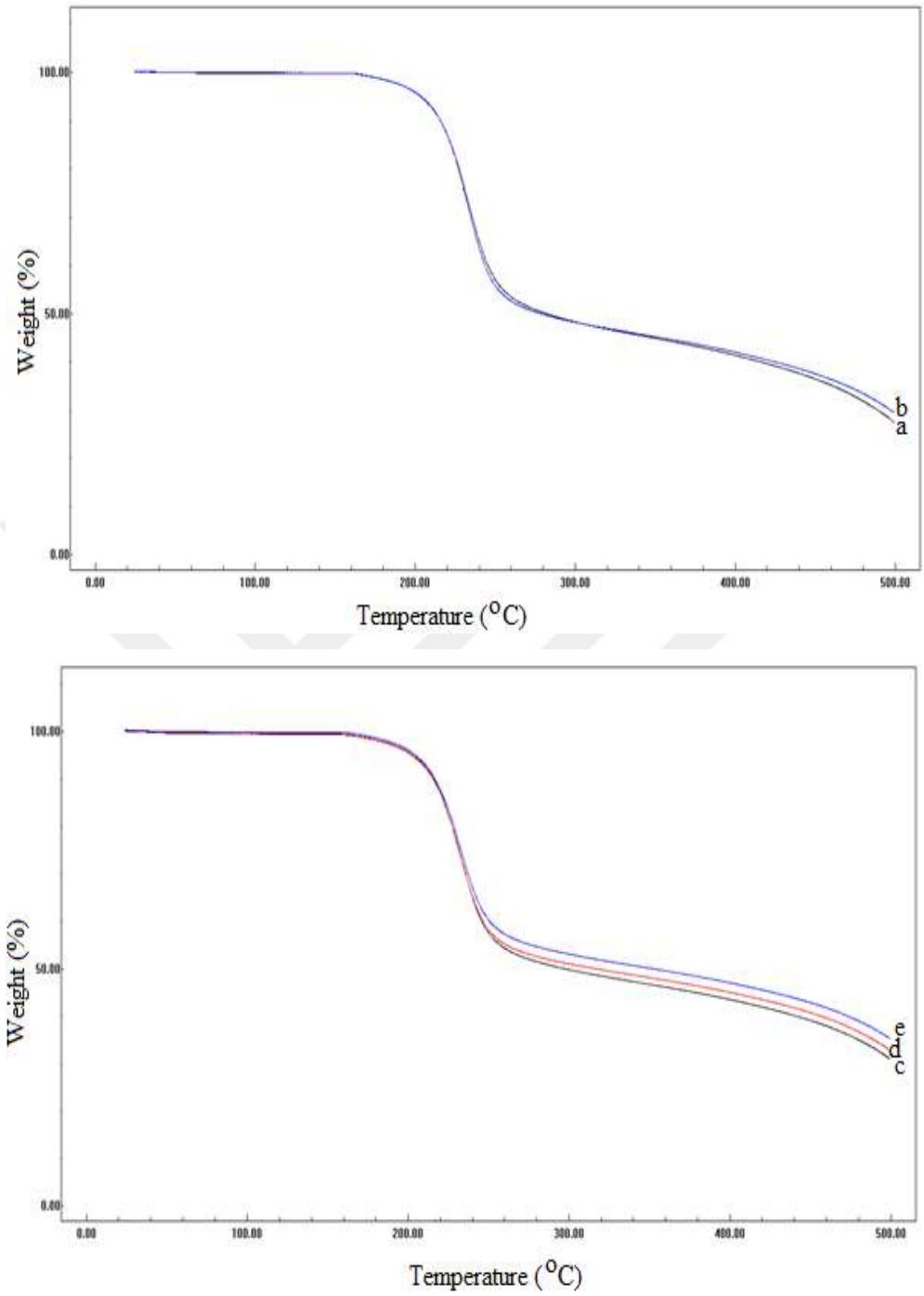


Fig 3.18. TGA curves of aminated PVC/5 wt% oxidized MWCNT composite doped at different percentages with nanographite : nanographite content (wt %): (a) 1 %, (b) 3 %, (c) 5 %, (d) 8 % and (e) 12 %

Table 3.2. Thermal investigation of aminated PVC / 5% oxidized MWCNT composites doping with nanographite at different percentages

Material	Tg (^{0}C)	Ti (^{0}C)	Weight loss (wt%) at 300 ^{0}C	Residue (wt%) at 500 ^{0}C
Amin. PVC/5% MWCNT/ nanograph. Comp. Nanographite (wt%)				
1	88.4	164.0	48.6	27.9
3	88.5	164.0	48.5	29.6
5	88.4	163.6	49.6	31.0
8	90.7	163.6	51.2	32.6
12	89.5	163.6	52.7	34.9

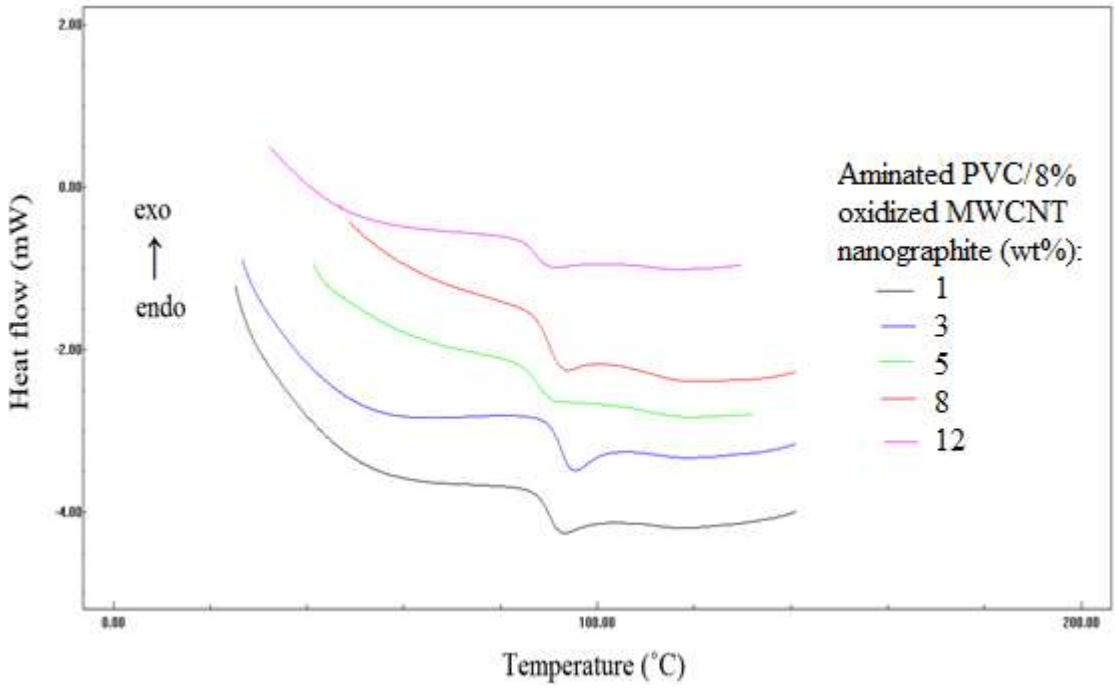


Fig 3.19. DSC curved of composites of aminated PVC/ 8% oxidized MWCNTs doped with nanographite

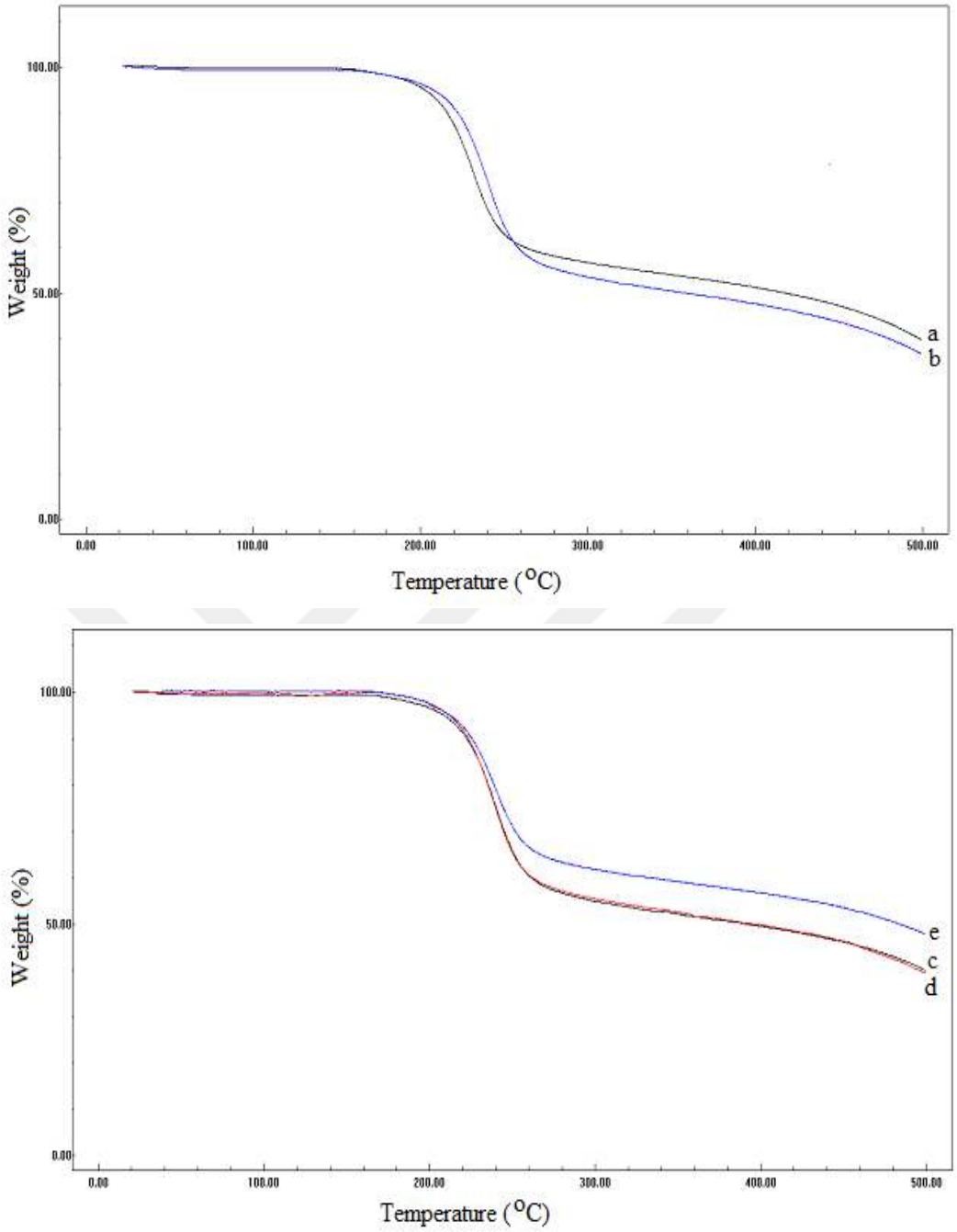


Fig 3.20. TGA curves of aminated PVC / 8 wt% oxidized MWCNT composite doped at different percentages with nanographite : nanographite content (wt %): (a) 1 %, (b) 3 %, (c) 5 %, (d) 8 % and (e) 12 %

Table 3.3. Thermal investigation of aminated PVC / 8% oxidized MWCNT composites doping with nanographite at different percentages

Material	Tg (^{0}C)	Ti (^{0}C)	Weight loss (wt) at 300 ^{0}C	Residue (wt%) at 500 ^{0}C
Amin. PVC/8% MWCNT/ nanograph. Comp. Nanographite (wt%)				
1	90.2	160	43.0	39.9
3	91.8	163	45.8	37.3
5	88.5	169	44.5	40.6
8	90.2	179	44.2	39.7
12	87.7	170	38.3	47.8

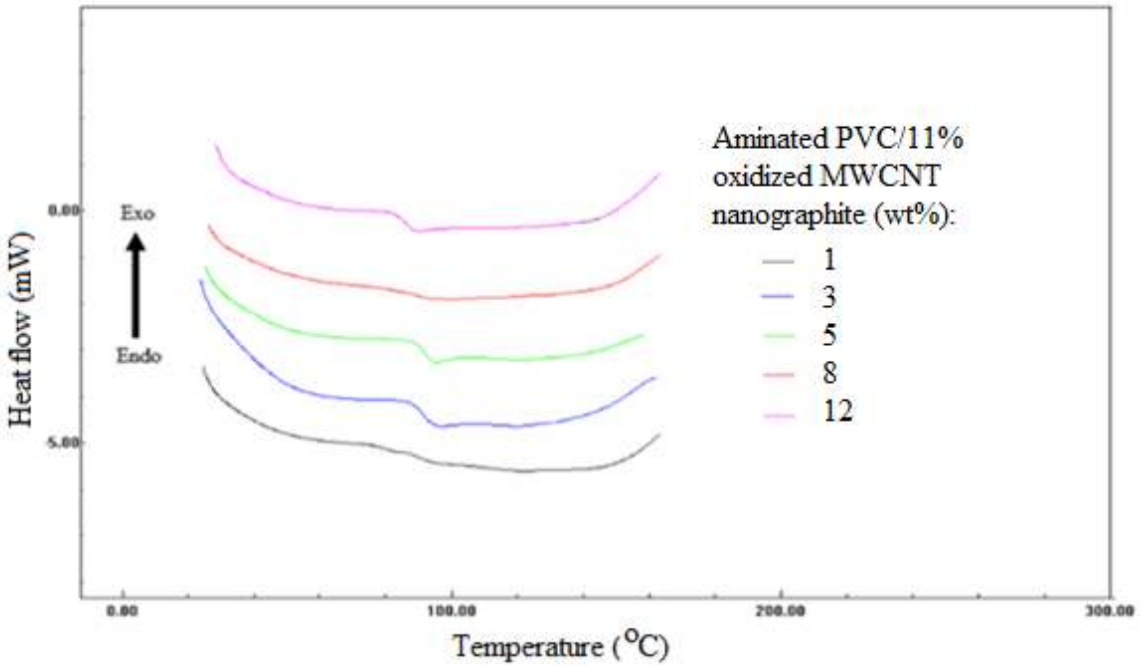


Fig 3.20. DSC curved of composites of aminated PVC/ 11% oxidized MWCNTs doped with nanographite

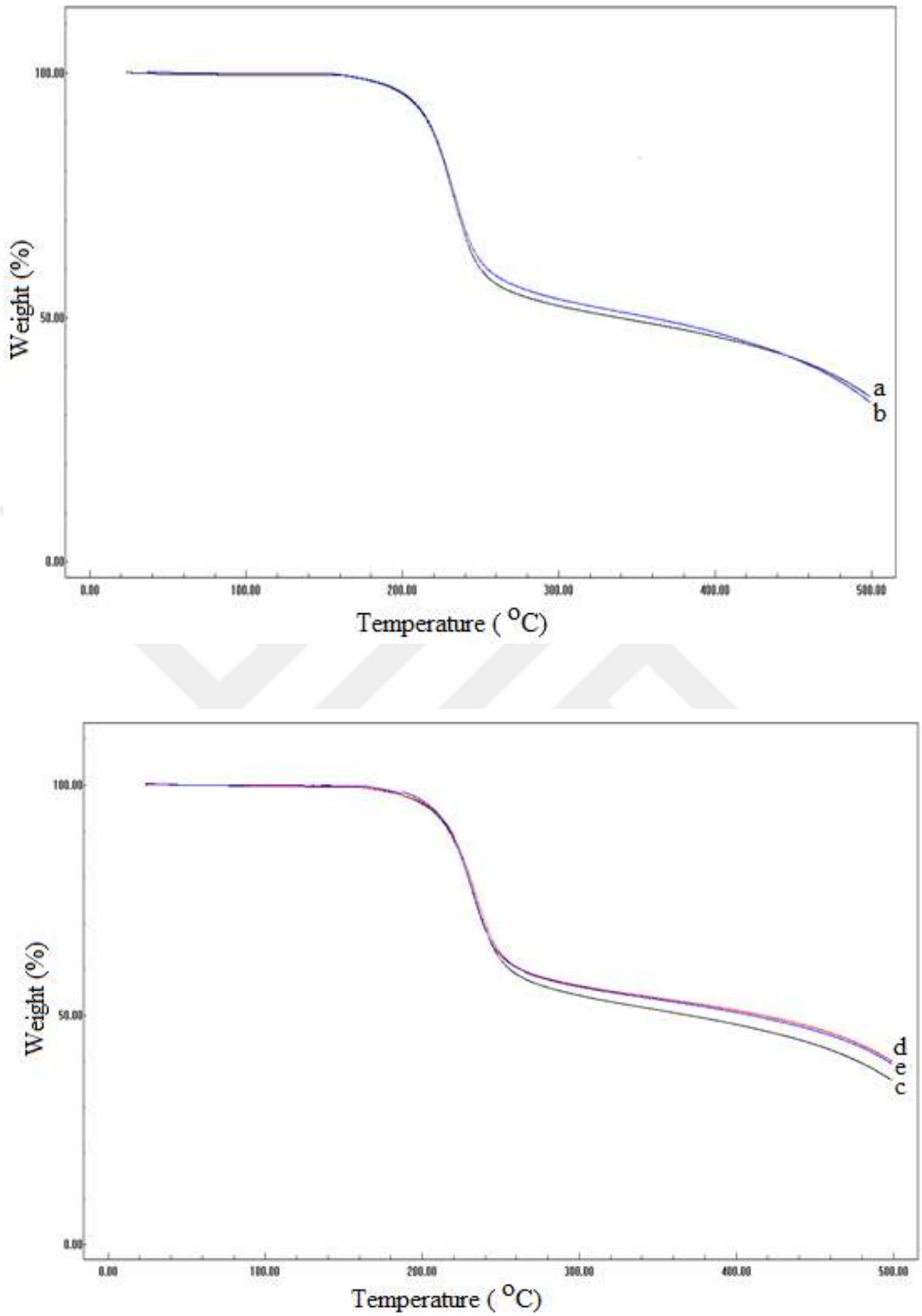


Fig 3.21. TGA curves of aminated PVC/11 wt% oxidized MWCNT composite doped at different percentages with nanographite : nanographite content (wt %): (a) 1 %, (b) 3 %, (c) 5 %, (d) 8 % and (e) 12 %

Table 3.4. Thermal investigation of aminated PVC / 11% oxidized MWCNT composites doping with nanographite at different percentages

Material	**T_g (°C)**	**T_i (°C)**	**Weight loss (wt) at 300 °C**	**Residue (wt%) at 500 °C**
Amin. PVC/11% MWCNT/ nanograph. Comp. Nanographite (wt%)				
1	86.9	165	47.1	34.2
3	91.7	159	46.1	32.7
5	90.5	159	45.5	35.9
8	88.1	165	43.0	40.4
12	85.7	167	43.6	39.6

3.4.3. Elecrical investigation

Discs with a surface area of 1.32 cm^2 were prepared using the aminated PVC / 8% oxidized MWCNT composites doped with nanographite at different percentages. The thicknesses of the discs were 0.061 cm for 1% nanographite, 0.067 for 3%, 0.062 for 5% and 0.071 cm for 12%.

3.4.3.1 Dielectric Constant, Dielectric Loss

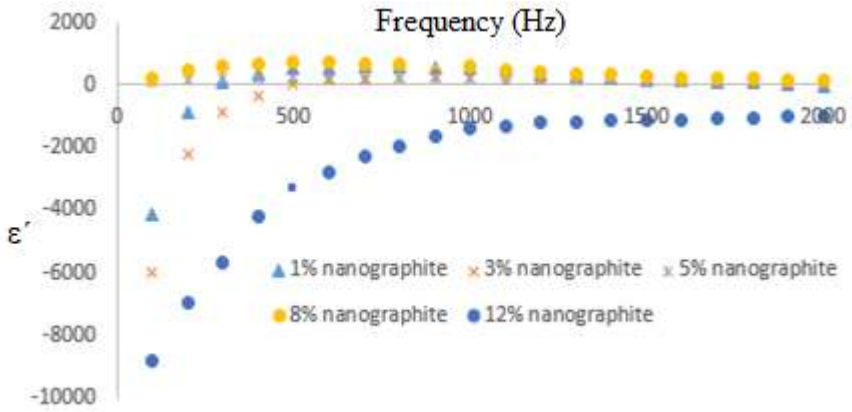


Fig 3.22. Variation of Dielectric constant of composites of aminated PVC/ 8% oxidized MWCNT/nanographite with change of frequency at room temperature

Variation of dielectric constant of composites of aminated PVC/8% oxidized MWCNT/nanographite with change of frequency is seen Figure 3.22. The Figure shows that dielectric constants of the composites containing 1%, 3% and 5% graphite started from the other negative ones and increased to the positive values with frequency increase and then changed again to negative values. The dielectric constant of composite with 8% of nanograph increased from 200 to 750 by increasing frequency from 100 Hz to 500 Hz, then it decreased to 100 at 2000 Hz. But for the composite containing 12% graphite, dielectric constant changed from -9000 to -1000 with increasing from 100 Hz to 2000 Hz in frequency.

Dielectric loss values are very high at lower frequencies. They decrease with increasing frequeny (Figure 3.23).

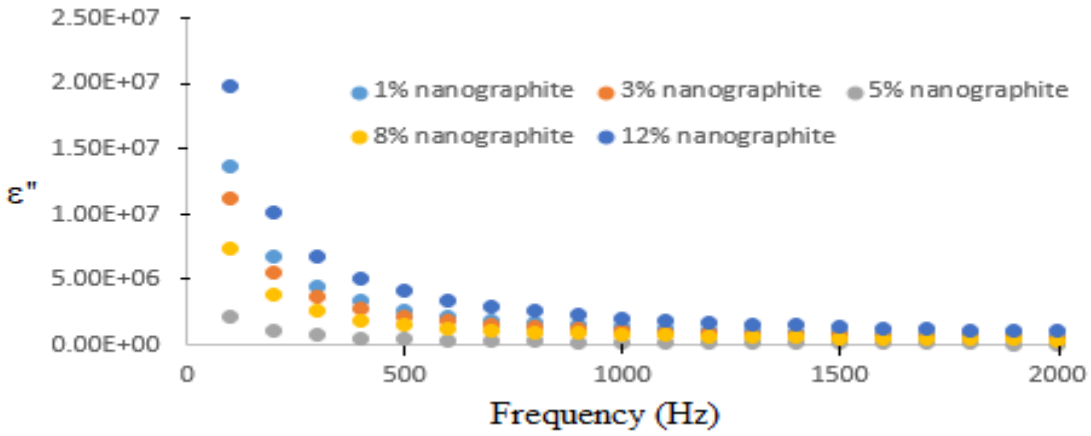


Fig 3.23. Variation of Dielectric loss of composites of aminated PVC/ 8% oxidized MWCNT/nanographite with change of frequency at room temperature

The frequency dependences of the dielectric constants of aminated PVC / 8% oxidized MWCNT/8% nanographite composites at different temperatures are shown in Figure 3.24. The Figure show that dielectric constans are negative large values at all temperature. It also showed that the dielectric constants at 20 ºC, 50 ºC, 65 ºC and 80 ºC have a maximum value at certain frequencies. The dielectric constant exceeded the maximum at around 650 Hz for 20 ºC, 50 ºC and 65 ºC, and around 1300 Hz for 80 ° C.

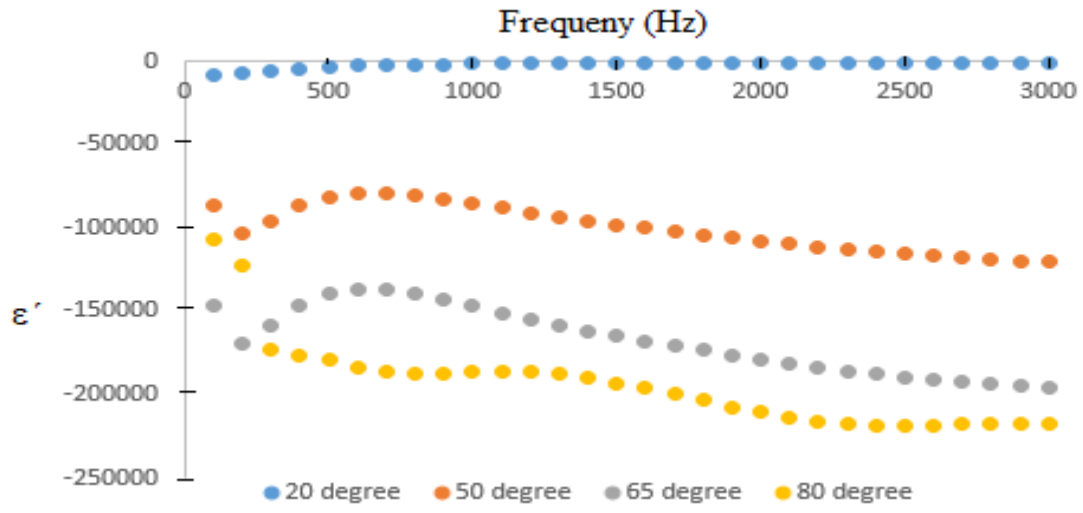


Fig 3.24. Variation **of** Dielectric constant of composites of aminated PVC/ 8% oxidized MWCNT/ 8% nanographite with frequency at different temperatures

Variations in dielectric loss of composites of aminated PVC/ 8% oxidized MWCNT/ 8% nanographite with change in temperature are shown in Figure 3.25. At low frequencies, dielectric losses are very high values at 50 ^{o}C, 65 ^{o}C and 80 ^{o}C, as the frequency increases, they fall and approach each other.

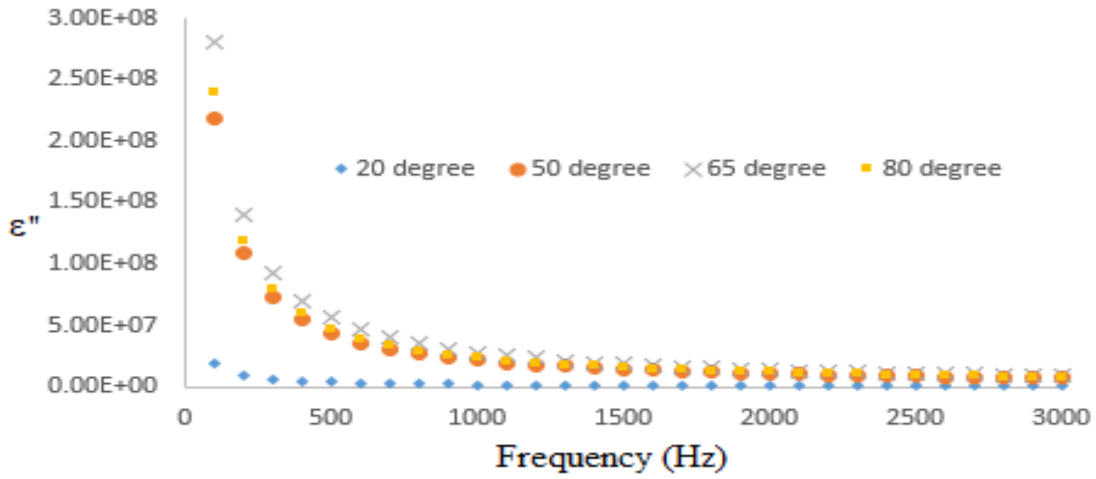
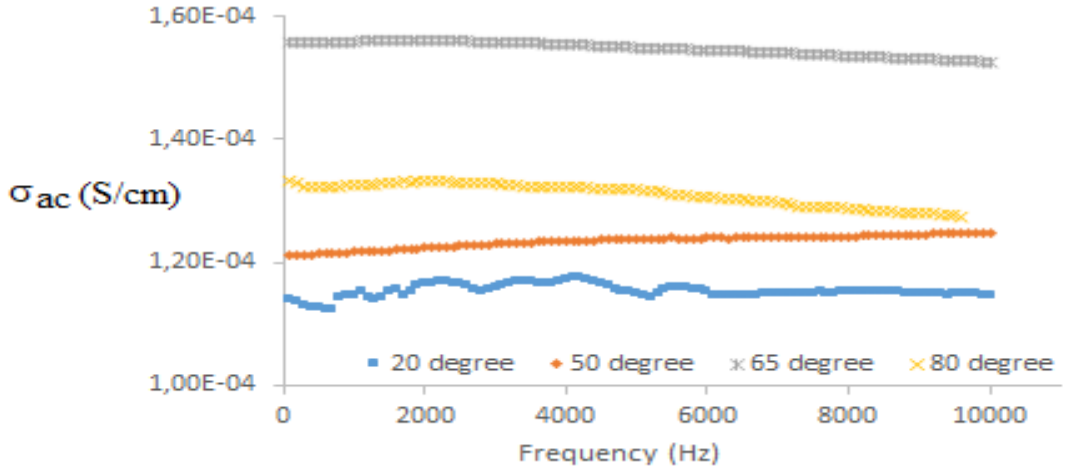


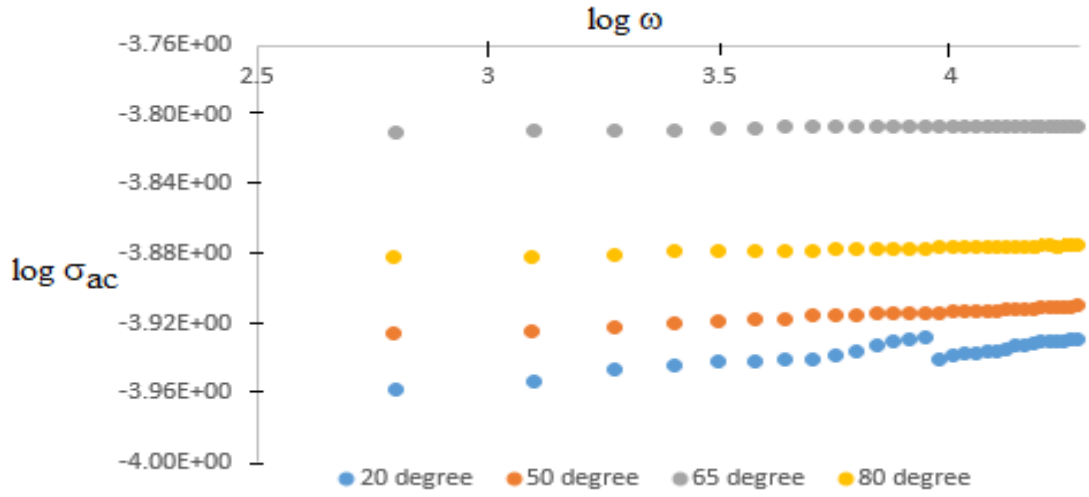
Fig 3.25. Variation of Dielectric loss of composites of aminated PVC/ 8% oxidized MWCNT/ 8% nanographite with frequency at different temperatures

3.4.3.2. A.C. conductivity

Variation of A.C. coductivity of composites of aminated PVC / 8% oxidized MWCNT / 8% nanographite with frequency in different temperatures is shown in Figure 3.26. At angular frequency at 630 Hz (linear frequency 100 Hz), A.C conductivities are $1.12x10^{-4}$ S/cm, $1.20x10^{-4}$ S/cm, $1.32x10^{-4}$ S/cm and $1.56x10^{-4}$ S/cm at 20 ^{o}C, 50 ^{o}C, 80 ^{o}C and 65 ^{o}C, respectively. At angular frequency 19963 Hz (linear frequency 3177 Hz), A.C conductivities are $1.19x10^{-4}$ S/cm, $1.24x10^{-4}$ S/cm, $1.35x10^{-4}$ S/cm and $1.58x10^{-4}$ S/cm at 20 ^{o}C, 50 ^{o}C, 80 ^{o}C and 65 ^{o}C, respectively. These values indicate that A.C conductivity is almost not dependent on the frequency and that there is little increase with temperature.



(A)



(B)

Fig 3.26. Variation **of** A.C. coductivity of composites of aminated PVC / 8% oxidized MWCNT / 8% nanographite with frequeny at different temperatures. (A) σ_{ac}- f, (B) log σ_{ac}- log ω.

3.5. PVC/aminated MWCNT composites

From PVC and aminated MWCNT, the composites containing aminated MWCNT in different concentrations (5%, 14% and 26% by weight) were prepared.

3.5.1. Characterization of PVC/aminated MWCNT (5 wt%) composite

FT-IR spectrum of aminated MWCNT is shown in Figure 3.27. The spectrum shows characteristic bands at 2920 and 2840 cm^{-1} (asymmetric and symmetric C-H stretching

bands in -CH_2- group), at 1635 cm^{-1} (C=C stretching vibration in MWCNT), at 1175 cm^{-1} (C-O stretching vibration), at 1080 cm^{-1} (Si-O-C, Si-O bands) and 794 cm^{-1} (it may be attributed N-H wagging)

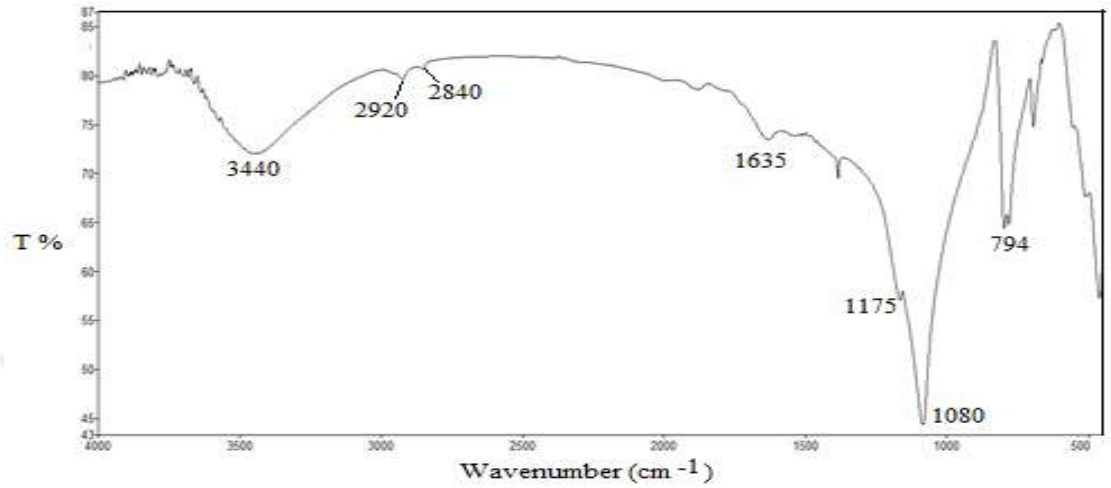


Fig 3.27. FT-IR Spectra of aminated MWCNT

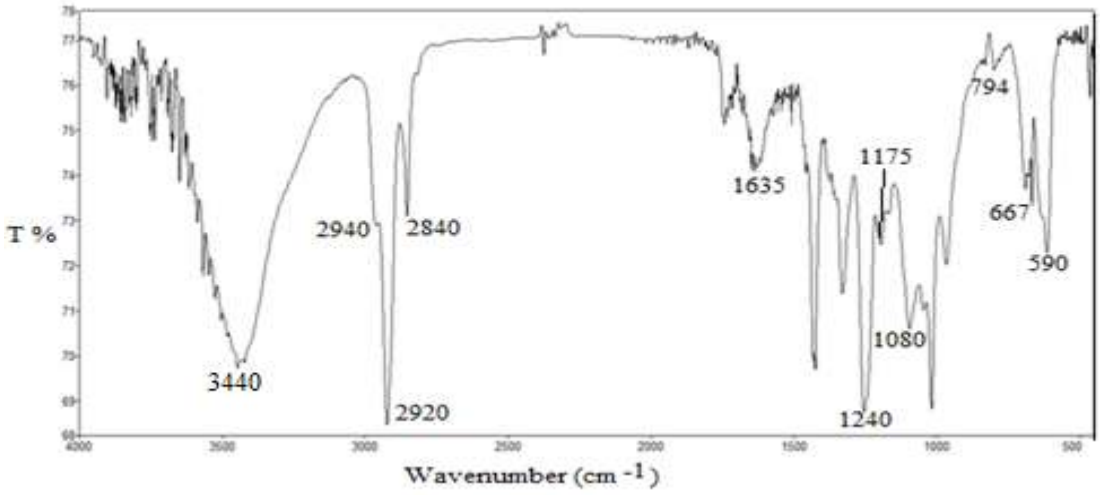


Fig 3.28. FT-IR spectra of PVC / aminated MWCNT (5wt%) composite

FT-IR spectrum of PVC/aminated MWCNT (5 wt%) composite is shown in Figure 3.28. In addition to the FT-IR spectrum bands (Fig. 3.27) of the aminated MWCNT, FT-IR spectrum of the composite showed characteristic bands at 2940 cm^{-1} (C-H stretching vibration in CHCl group), 1240 cm^{-1} (C-H wagging band in CHCl group), 667 and 590 cm^{-1} (C-Cl stretching vibraion in CHCl group) belonging to PVC. Relative intensities of CH_2 bands at 2920 and 2840 cm^{-1} have increased because PVC also contains CH_2 group.

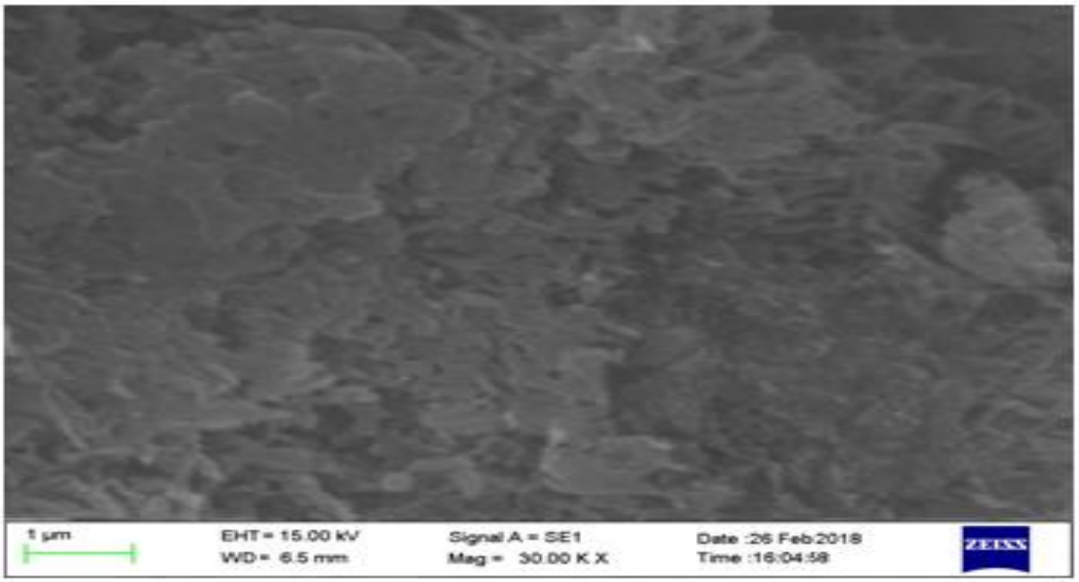


Fig 3.29. SEM image of PVC / aminated MWCNT (5wt%) composite

SEM image of PVC/aminated MWCNT (5 wt%) is shown in Figure 3.29. The SEM image shows that the MWCNT bars are dispersed relatively homogeneously in the PVC matrix and the bars are mostly embedded in PVC.

3.5.2. Thermal investigations

DSC curves of pure PVC and PVC / aminated MWCNT composites containing aminated MWCNT at three different concentrations are shown in Figure 3.30. The glass transition temperatures for PVC and the PVC/aminated MWCNT composites (5 %, 14 % and 26 % by weight) are obtained as 87.2 ºC, 79.5 ºC, 89.7 ºC and 92.3 ºC from DSC curves, respectively. TGA curves of pure PVC and PVC / aminated MWCNT composites containing aminated MWCNT at three different concentrations are shown in Figure 3.31.

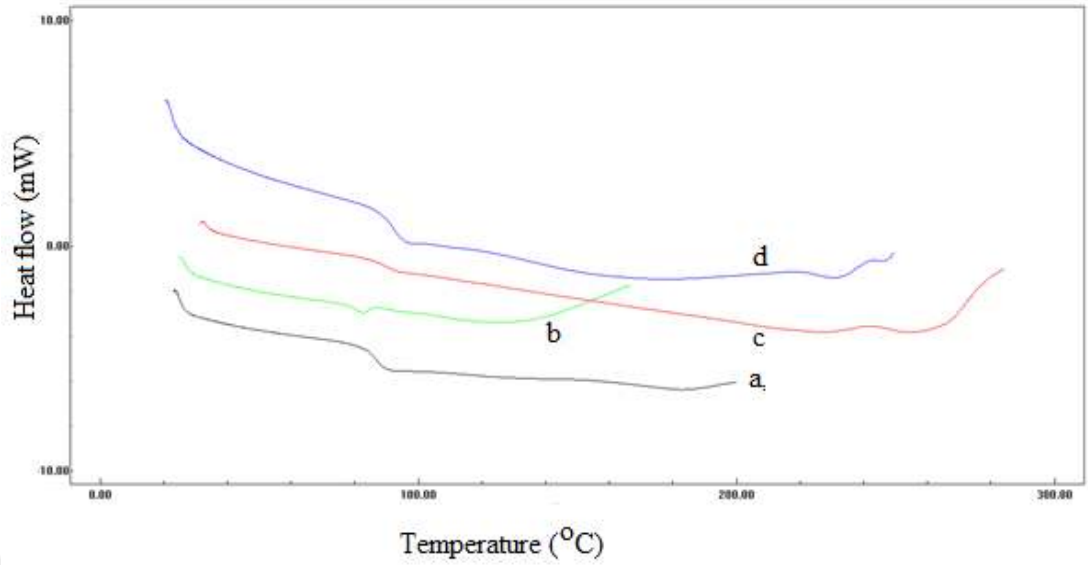


Fig 3.30. DSC curves of PVC (a) and PVC/aminated MWCNT [wt % : 5 % (b), 14 % (c) and 26 % (d)]

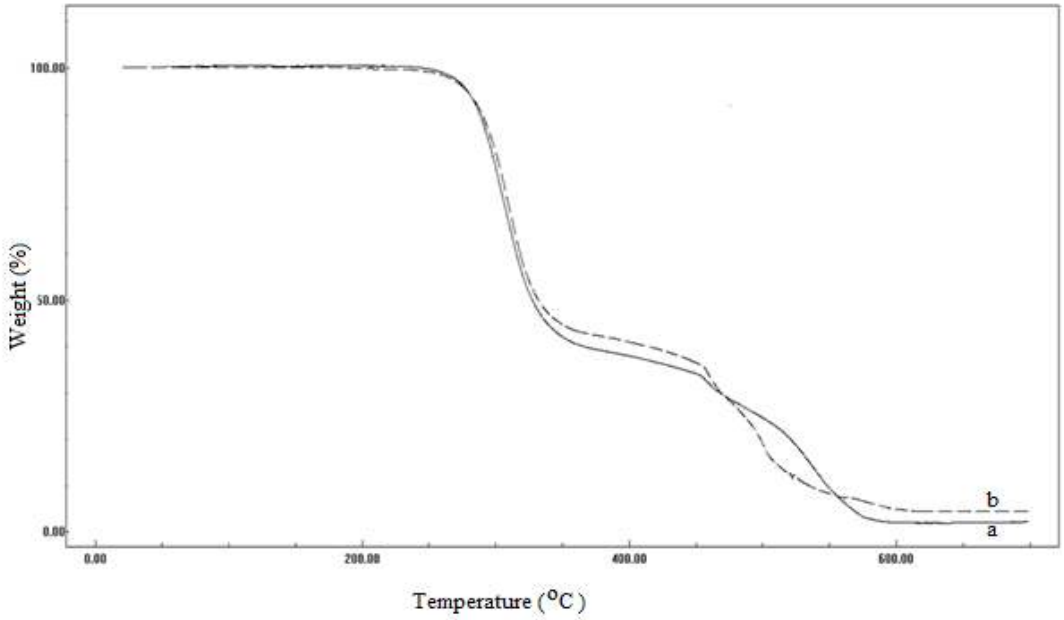


Fig 3.31. TGA curves of PVC (a) and PVC/ 5% aminated MWCNT composite (b)

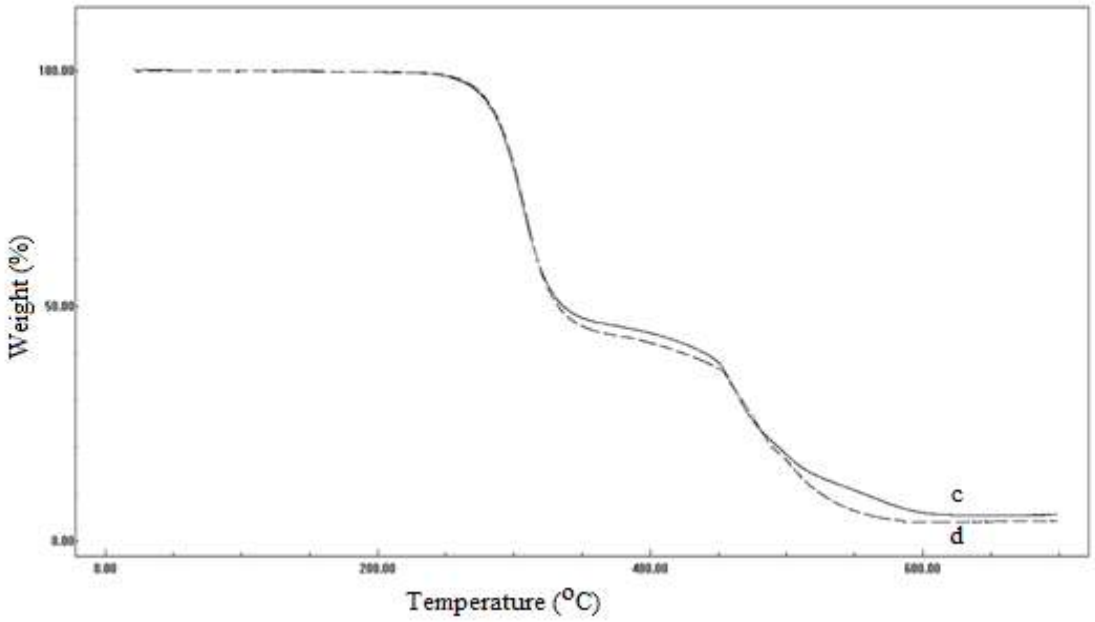


Fig 3.32. TGA curves of PVC/ 14 % aminated MWCNT (c) and PVC/ 26 % aminated MWCNT (d)

3.5.3 Elecrical investigation

3.5.3.1. A.C conductivity investigation in PVC/aminated MWCNT (5wt%) composite

The variation of the AC conductivity of the PVC / aminated MWCNT composites containing 5% MWCNT was studied. Graph of ln σ_{ac} vs 1000/T(K) is seen in Figure 3.33. The plot shows that A.C conductivity increases slowly with increasing temperature in range of low temperatures, it increases rapid with increasing temperature in range of high temperatures.

The variation of the AC conductivity of the PVC / aminated MWCNT composites containing 5% MWCNT doped with graphite (5 wt%) was also studied. Graph of ln σ_{ac} vs 1000/T(K) is seen in Figure 3.34. The plot shows that A.C conductivity increases slowly with increasing temperature in range of low temperatures, it increases rapid with increasing temperature in range of high temperatures.

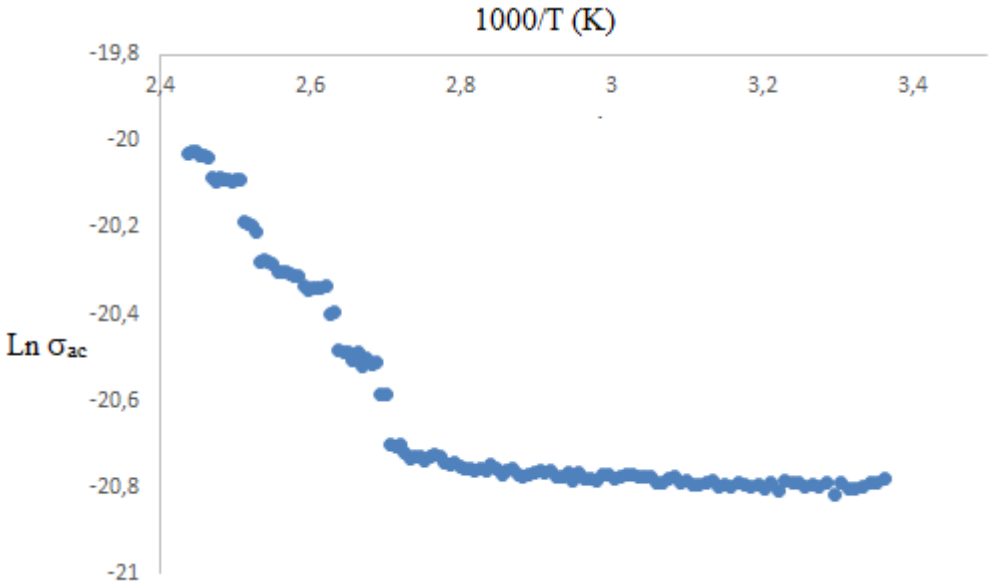


Fig 3.33. Plot of ln σ_{ac} versus 1000/T for PVC/aminated MWCNT (5wt%)

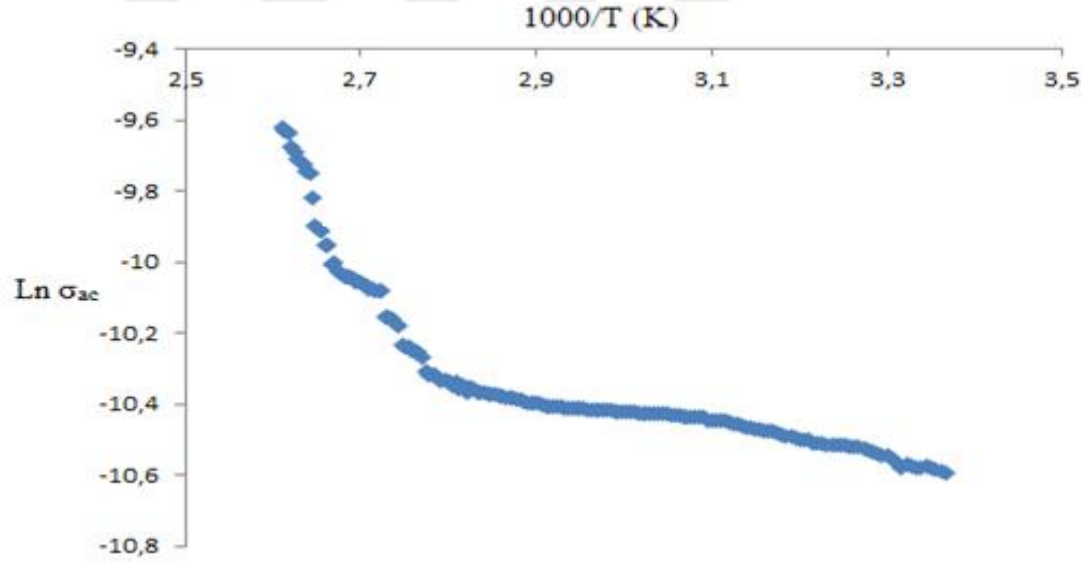


Fig 3.34. Plot of ln σ_{ac} versus 1000/T for PVC/aminated MWCNT (5wt%) composite doped with graphite (5 w%)

3.5.3.2 Dielectric investigation in PVC/aminated MWCNT (5wt%) composite

The dielectric constant is the ratio of the permittivity of a substance to the permittivity of free space. One important property of a dielectric material is its permittivity. Permittivity (ε) is a measure of the ability of a material to be polarized by an electric field.

Relative permittivity (ε′) (dielectric constant) is difined as ε′= ε/ ε_o, where ε_o is the permittivity of vacuum (8.85 x 10^{-12} F/m).

Dependence on temperature of dielectric constant and dielectric loss values of PVC/aminated MWCNT (5wt%) composite is shown in Figure 3.35 and Figure 3.36, respectively. Figure 4.36 shows that dielectric constant of the composite increase with increasing temperature, as faster above Tg and slower below Tg. Figure 4.37 shows that the dielectric loss values are constant at a value of 0.025 but do not change with temperature below Tg, while it increases with increasing temperature above Tg and reaches such as 0.2 value at 390 K. From Figure 4.36 and Figure 4.37, Glass transition temperature is estimated as 92 and 90 ^oC, respectively.

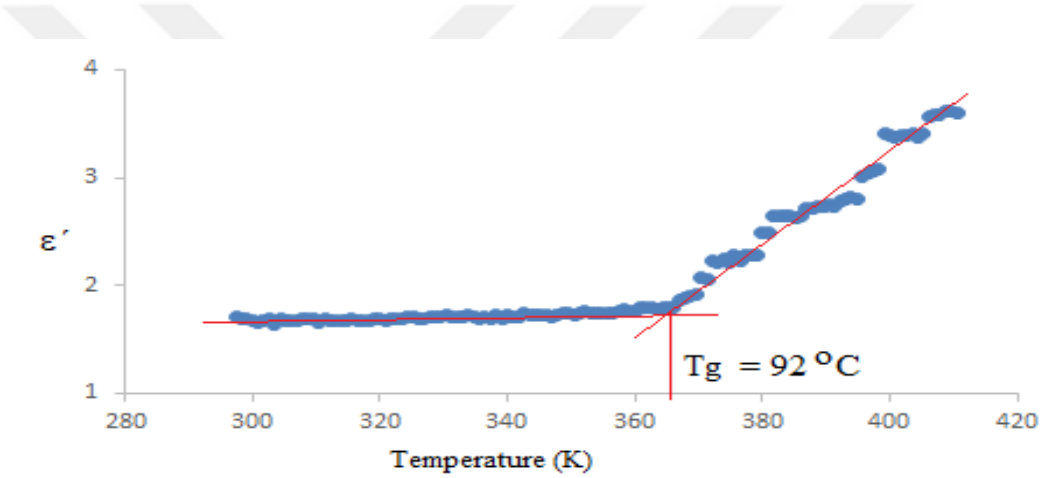


Fig 3.35. Dependence on temperature of dielectric constant for PVC/ 5% aminated MWCNT

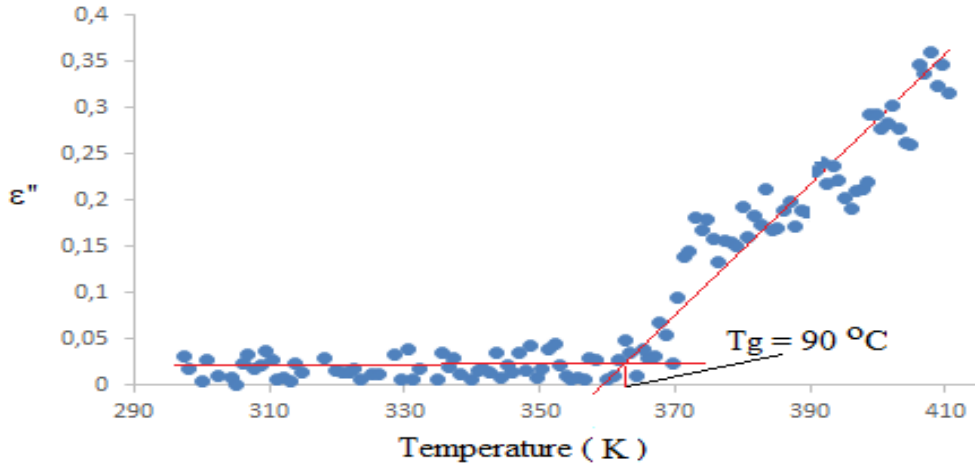


Fig 3.36. Dependence on temperature of dielectric loss for PVC/ 5% aminated MWCNT

3.6. Electrical properties of PVC and Aminated PVC

Dependence on frequency of dielectric constant, dielectric loss and log σ_{ac} of PVC and aminated PVC are shown in Figure 3.37, Figure 3.38 and Figure 3.39, respectively. Figure 3.37 shows that The dielectric constant of PVC and aminated PVC decreased very slowly while the frequency increased from 100 Hz to 2000 Hz and remained constant at 2.25 for PVC and 3.5 for aminated PVC. At all frequencies, aminated PVC showed higher dielectric constant than PVC.

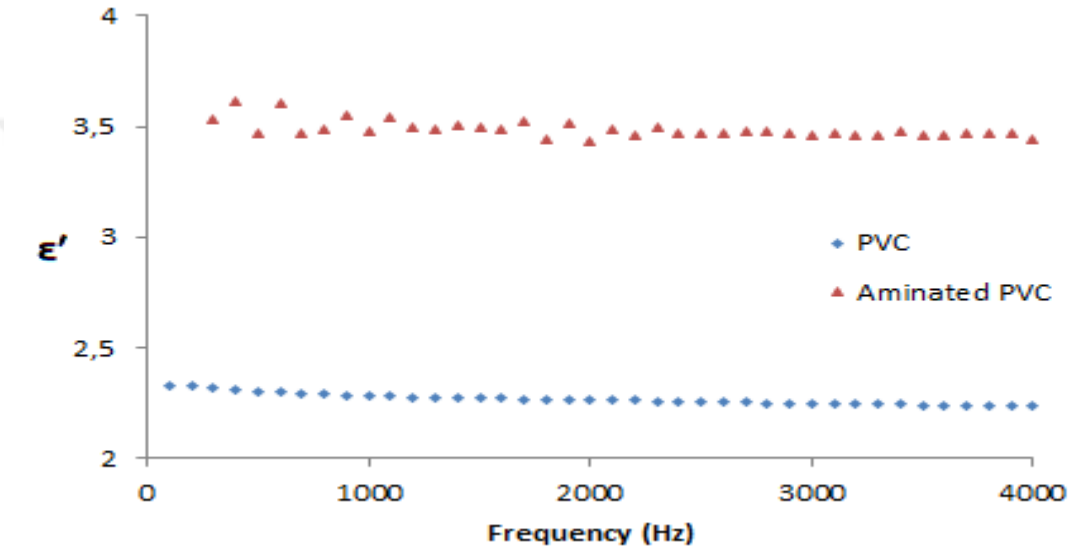


Fig 3.37. Variation of dielectric constant of PVC and aminated PVC with frequency

Figure 3.38 shows that The dielectric loss of aminated PVC decreased rapidly while the frequency increased from 200 Hz to 1000 Hz and remained constant at about 0.075. For PVC, the dielectric loss decreased before between 100-1000 Hz, then increased, and then remained constant at about 0.050. At all frequencies, aminated PVC showed some higher dielectric loss than PVC.

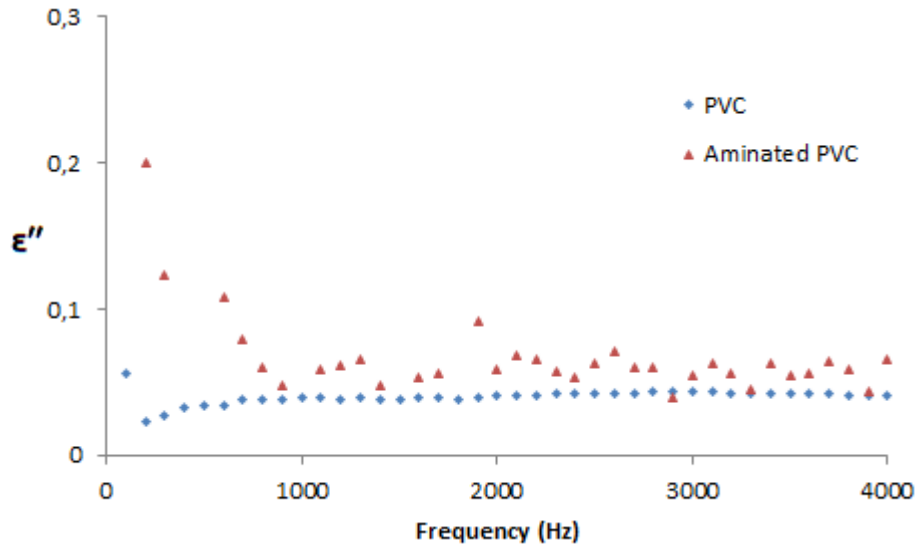


Fig 3.38. Variation of dielecric loss of PVC and aminated PVC with frequency

Figure 3.39 shows that A.C. conductivity (as log σ_{ac}) of PVC and aminated PVC has increased more slowly with increasing frequency in the range of 1000-4000 while showing a rapid increase with frequency in the range of 100-1000 Hz. At all frequencies, aminated PVC showed higher conductivity than PVC.

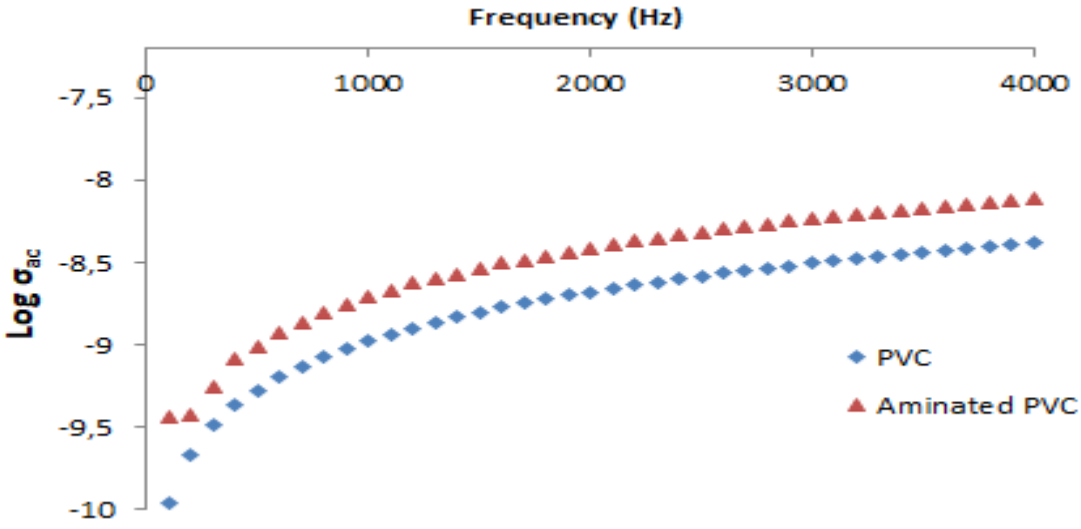


Fig 3.39. Variation of Log σ_{ac} of PVC and aminated PVC with frequency

Dependence on temperature of dielectric constant, dielectric loss and log σ_{ac} of PVC and aminated PVC are shown in Figure 3.40, Figure 3.41 and Figure 3.42, respectively.

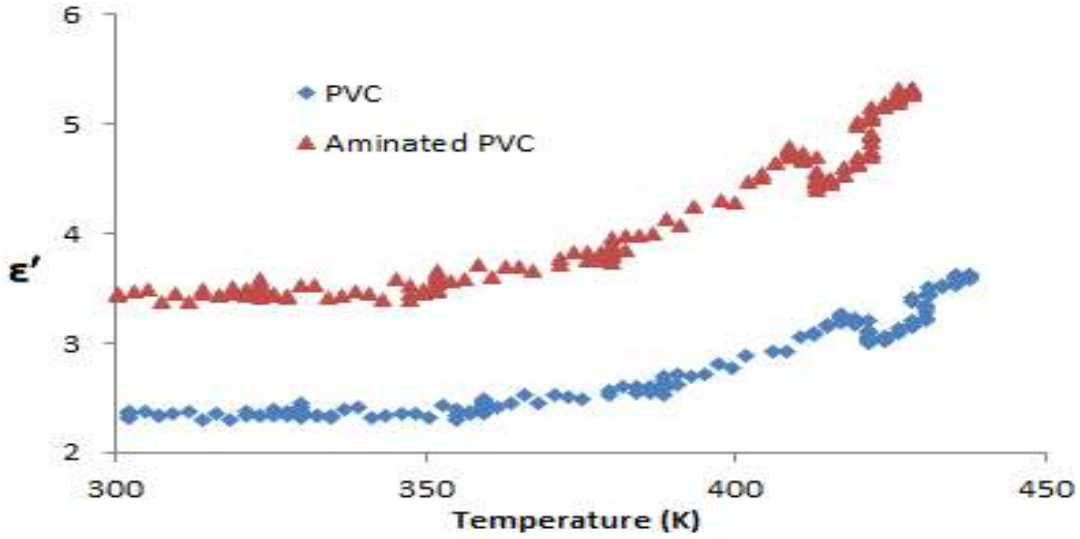


Fig 3.40. Variation of dielectric constant of PVC and aminated PVC with temperature

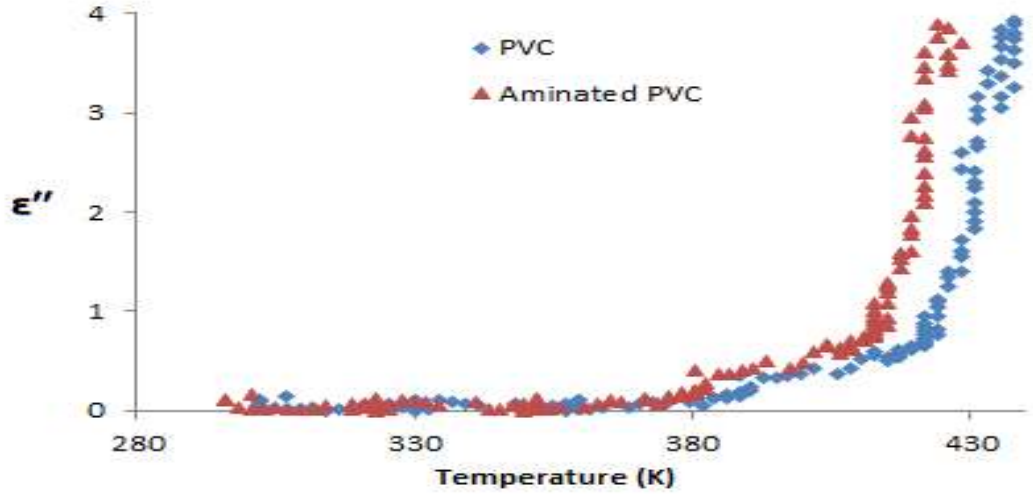


Fig 3.41. Variation of dielectric loss of PVC and aminated PVC with temperature

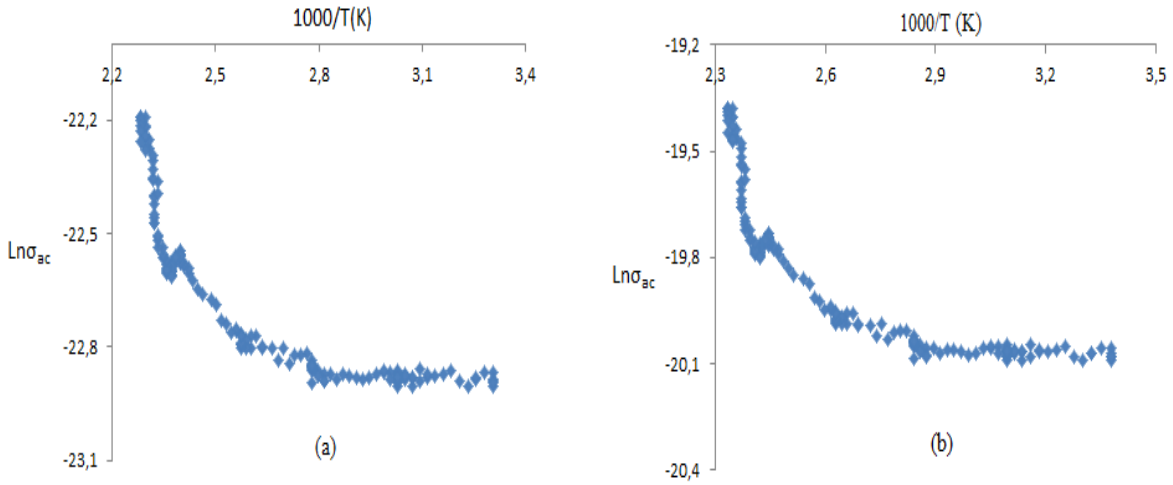
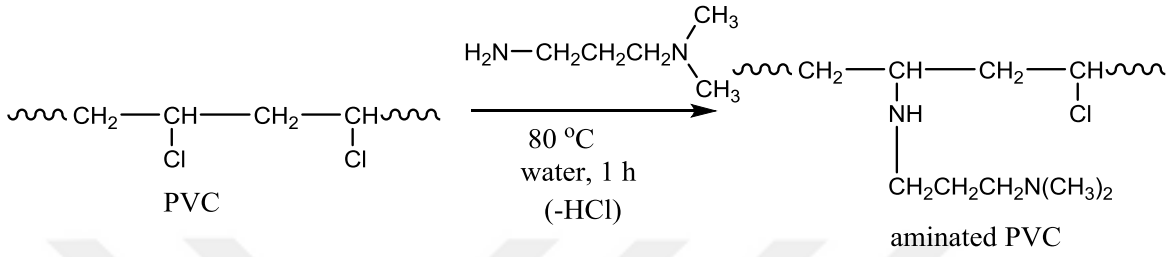


Fig 3.42. Variation of conductivity of (a) PVC and (b) aminated PVC with temperatur

4. DISCUSSIONS

This study is concerned with the preparation and characterization of composites of oxidized MWCNT with aminated PVC. Aminated PVC was prepared from nucleophilic substitution reaction of PVC with 3-(Dimethylamino)-1- propylamine, as follow:



Oxidized MWCNT was buyed and contained 2.5 % by mol carboxylic acid group. FT-IR spectrum (Fig. 3.1b) of aminated PVC showed characteristic bands at same wavenumbers with PVC including water bands. Aminated PVC showed weak bands related to the amine group attached to PVC at 2780 cm^{-1} (C-H stretching vibration on CH_3-N), at 1470 cm^{-1} (aliphatic C-N stretching vibration band) and at 606 cm^{-1} (C-Cl stretching band relatively weaker than that of PVC). The N-H band of aminated PVC is probably under the O-H stretching band.

Percentage of aminated group in PVC was found by titration. For this purpose, a certain volume of HCl solution at a certain concentration was added to the PVC , and then a certain volume of NaOH solution at a certain concentration was added . The excess of NaOH solution was back titrated with HCl solution in presence of phenolphthalein. At the end of this process, 10.8 % (by mol) amine compound was found to bind to PVC. SEM-EDX results (Fig. 3.2) showed that aminated PVC contained 11.28 % (by mol) amine compound. The value found with titration is thought to be a more accurate result because SEM-EDX represents only a specific region of the polymer. Nevertheless, these two values can be regarded as more or less close to each other.

Composites containing 2%, 5%, 8% and 11% (by weight) oxidized MWCNT of aminated PVC were prepared. FT-IR spectrum (Fig. 3.4) of the composite containing 11 wt% oxidized MWCNT shows characteristic bands of both aminated PVC and oxidized MWCNT. The SEM image (Fig.3.5a) of the composite containing 5% oxidized MWCNT shows that most of the oxidized MWCNT rods are well dispersed in the aminated PVC

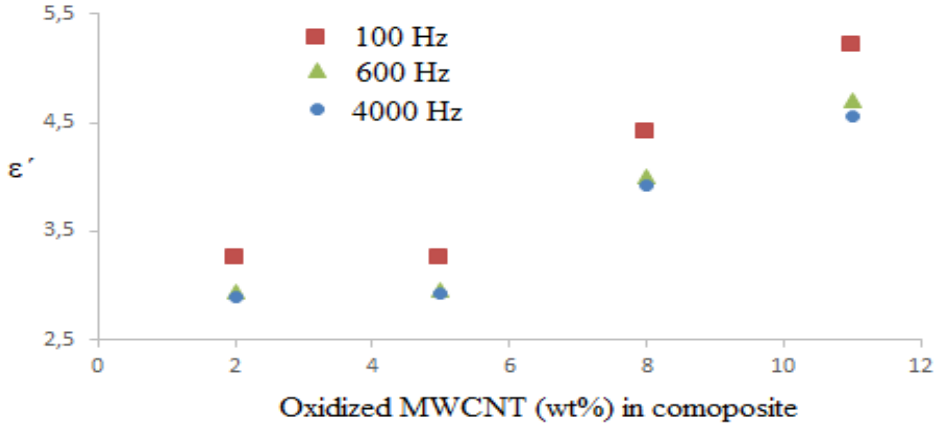
matrix, with a few minor irregularities. The SEM image (Fig.3.5b) of the other composite (11% oxidized MWCNT) shows one of the fractured zone, which reveals the well dispersed oxidized MWCNT rods in the composite. The length of the rods is not very well chosen because they are embedded in the polymer matrix.

DSC results showed that amination of PVC reduced the glass transition temperature (T_g) of PVC from 84.7 to 79.0 ^oC. This result can be interpreted as the fact that the amine compound separates the PVC chains partially, thereby increasing the free volume in the polymer. Increasing free volume means decreasing T_g. The Tg values of all composites are higher than those of aminated PVC. The interaction of the oxidized MWCNT-bound acid group with the amine group in the aminated PVC is believed to result in a reduction in free volume by bringing the chains closer together.This means that Tg increases.

Initial decomposition temperature (T_i) are 260 ^oC for PVC and 209 ^oC for aminated PVC. Based on T_i at which it begins to degrade rapidly, it can be seen that the amination process reduces the thermal stability of PVC (Table 3.1). T_i values of all composites close to that of aminated PVC.

Dielectric constant values of all composites decrease with increases of the applied field frequency, rapid at low frequencies and at high frequencies, then remain constant. The increase of frequency results in decreasing of the polarization, at sufficiently high frequencies, the dipoles can no longer follow the rapid changes in field direction [42]. Dielectric constant of the composites increases with increasing in oxidized MWCNT concentration of the composites.

At all frequencies, dielectric constant increased with percentage of oxidized MWCNT in the composites, as shown in the following graphic.

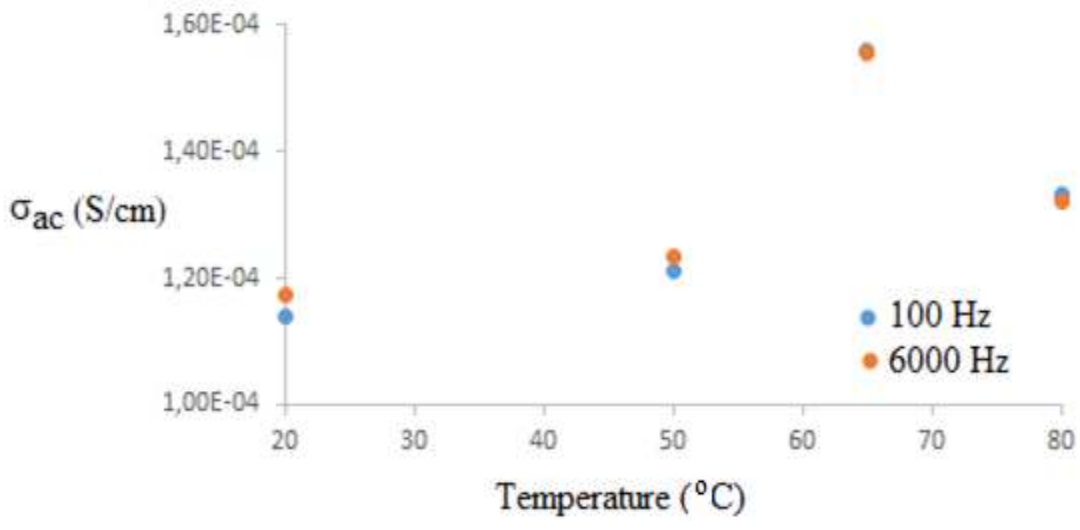


The dielectric constant of the composites increases with increasing temperature in range of 300-390 K, except composite containing 11% of oxidized MWCNT. The increase has accelerated at around 365 K (92 ^{o}C) . This temperature is thought to be the T_g of the polymer.

A.C. conductivities for the all compsites increase rapidly with increasing frequency up to 800 Hz, then they increase slower up to 4000 Hz (Fig.3.13) at room temperature. M. Pollak has demonstrated that if A.C conductivity increases as a function of frequency, then conduction is taking place by hopping [43].

Composites containing oxidized MWCNT in different weight percentages were doped with nanographite in different percentages (1 %, 3 %, 5 %, 8 % and 12% by weight). FT-IR spectrum of aminated PVC/11% oxidized MWCNT composite doped with 12 wt% nanographite is similar with that of undoped composite due to the both composite contains similar kinds of hydrocarbon groups. SEM images show that the oxidized MWCNT and nanographite particles are homogeneously dispersed in the aminated PVC matrix (Fig. 3.16). it has been seen that the doping with nanograph does not have much effect on the thermal properties. The main effect of grafting is on A.C. conductivity.

For example, aminated PVC / 8% oxidized MWCNT composite indicates an A.C conductivity of 2.37×10^{-9} S / cm at around room temperature , A.C. conductivity of doped composite with 8% nanographite increased to a value of 1.18×10^{-4} S / cm. These numbers for conductivity show that the doping increases the conductivity by about 45,000-50000 times. Conductivity increased slightly with temperature at all frequencies and showed a maximum value at 65 ° C, as show in the following graph.



from the other negative ones and increased to the positive values with frequency increase and then changed again to negative values at room temperature. The dieleccric constant of the composite containing 12% graphite gave negative values at all frequencies (Fig. 3.22).

Variation of A.C. conductivity of any material with angular frequency at different temperatures is important in terms of giving information about the conductivity mechanism. A.C conductivity, $\sigma_{ac}(\omega)$, is related to the total conductivity, $\sigma_{total}(\omega)$ and D.C. conductivity, σ_{dc}, according to the relation [44], [45]:

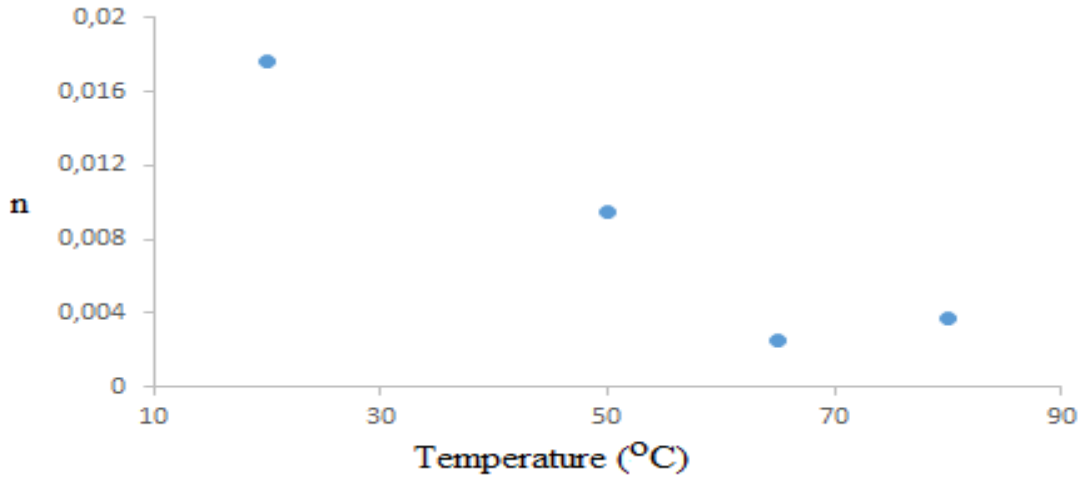
$$\sigma_{ac}(\omega) = \sigma_{total}(\omega) - \sigma_{dc}$$

However, the dc component σ_{dc} is negligibly small compared to the $\sigma_{total}(\omega)$. Thus $\sigma_{total}(\omega)$ considered to be $\sigma_{ac}(\omega)$. On the other hand, A.C conductivity is related to the angular frequency by the following equation (Universal power Law) [44], [45]

$$\sigma_{ac}(\omega) = A.\omega^{n}$$

where *A* is constant, *ω is* the angular frequency, and n is frequency exponent ($0 \leq n \leq 1$). The plot of log $\sigma_{ac}(\omega)$ against log ω gives a straight line with n slope.

The plots of log $\sigma_{ac}(\omega)$ against log ω at different temperatures (20 ^{o}C, 50 ^{o}C, 65 ^{o}C and 80 ^{o}C) For each temperature, the slopes of the straight lines against the temperature are as follows. For each temperature, the slopes of the straight lines against the temperature are as follows:



The n values are between zero and one and close to zero. They decrease as the temperature increases. The fact that n values are close to zero indicates a significant contribution of D.C conductivity. The fact that n values decrease with temperature means that the corraleted barrier hopping (CBH) conductivity mechanism dominates. In CBH

model, the conduction occurs via single polaron or bipolaron hopping process over the Coulomb barrier separating two defect centers [46].

Some part of this work is related to PVC / aminated MWCNT composites. FT-IR spectrum (Fig.3.28) of PVC/aminated MWCNT (5 wt%) composite shows the characteristic bands at 2940 cm^{-1}(C-H stretching vibration in CHCl group), 2920 and 2840 cm^{-1} (asymmetric and symmetric C-H stretching bands in -CH_2- group), 1240 cm^{-1} (C-H wagging band in CHCl group), 667 and 590 cm^{-1} (C-Cl stretching vibraion in CHCl group) belonging to PVC, 1635 cm^{-1} (C=C stretching vibration in MWCNT), at 1175 cm^{-1} (C-O stretching vibration), 1080 cm^{-1} (Si-O-C, Si-O bands) and 794 cm^{-1} (it may be attributed N-H wagging), The N-H stretching vibration is probably within 3440 cm^{-1} band.

The SEM image (Fig. 3.29) shows that the MWCNT bars are dispersed relatively homogeneously in the PVC matrix and the bars are mostly embedded in PVC.

DSC curves (Fig. 3.30) of pure PVC and PVC / aminated MWCNT composites containing aminated MWCNT at three different concentrations shows that the Tg (79.5 ^oC) of the composite containing 5% MWCNT was lower than that (87.2 oC) of PVC and Tgs of the composites containing 14% and 26% MWCNT (89.7 ^oC and 92.3 ^oC, respectively) were higher than that of PVC.

TGA curves (Fig. 3.31 and 3.32) of pure PVC and PVC / aminated MWCNT composites containing aminated MWCNT at three different concentrations show that initial decomposition temperature for PVC and the composites are around 280 ^oC, the residue at 500 ^oC are 26% for PVC, 19.6% for PVC/5% aminated MWCNT composite, 19.1% for the composite containing 14% aminated MWCNT and 17.4 for the composite containing 26% aminated MWCNT.

The glass transition temperature (T_g) of the polymer and the activation energy of AC conductivity were estimated from conductivity-temperature relationship. The AC conductivity depends on the temperature to the following equation [47]

$$\sigma_{ac} = \sigma_o exp\left(\frac{-E_a}{kT}\right)$$

Where σ_{ac} is AC conductivity, σ_o is a constant, E_a is activation energy of AC conductivity, k is Boltzmann´s constant (k = 1.38 x 10^{-23} J/K) and T is the absolute temperature. If the logarithm of both sides of this equation is taken,

$\ln \sigma_{ac} = \ln \sigma_o - E_a/kT$

equation is obtained. Slope of plot of $\ln \sigma_{ac}$ versus 1/T is E_a/k. (as seen in Fig. 3.33)

The equation of the straight line above T_g is y = -2,1888 . x – 14,667 (R^2= 0,964).

The equation of the straight line below Tg is y = -0,1051 . x – 20,459 (R^2= 0,819).

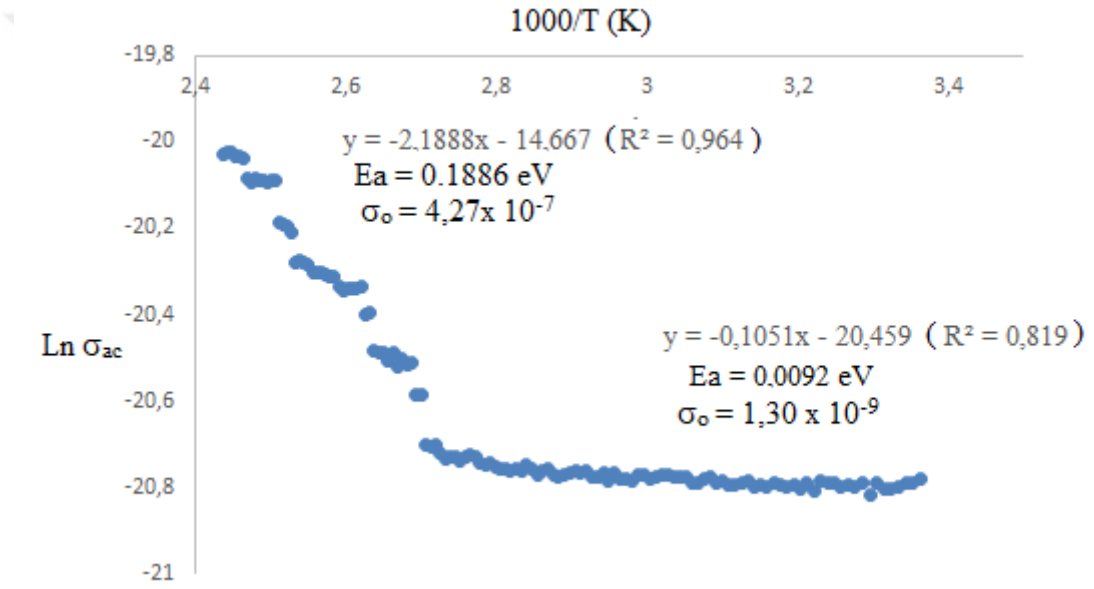
For both equation, x corresponds 1000/T.

The joint solution of these two lines showed that the Tg temperature for the composite (5% aminated MWCNT) was 86.8 ºC.

For above T_g, slope = $-E_a/k$ = -2.1888 x 1000

E_a= (2.1888 x1000) $1.38x10^{-23}$ J x 1 eV/$1.602x10^{-19}$J = 0.1886 eV

For below T_g, E_a = 0.0092 eV. The activation energies of A.C. conductivity show that A.C. conductivity above Tg depends more on the temperature than conductivity below Tg



After doping of PVC/aminated MWCNT (5% wt) composite with graphite (5 wt%), plot of ln σ_{ac} vs 1000/T is seen in Fig. 3.34.

The equation of the straight line above Tg is y = -3.4798 . x – 0.6298 (R^2= 0,941).

The equation of the straight line below Tg is y = -0,3853 . x – 9.2686 (R^2= 0,960).

For both equation, x corresponds 1000/T.

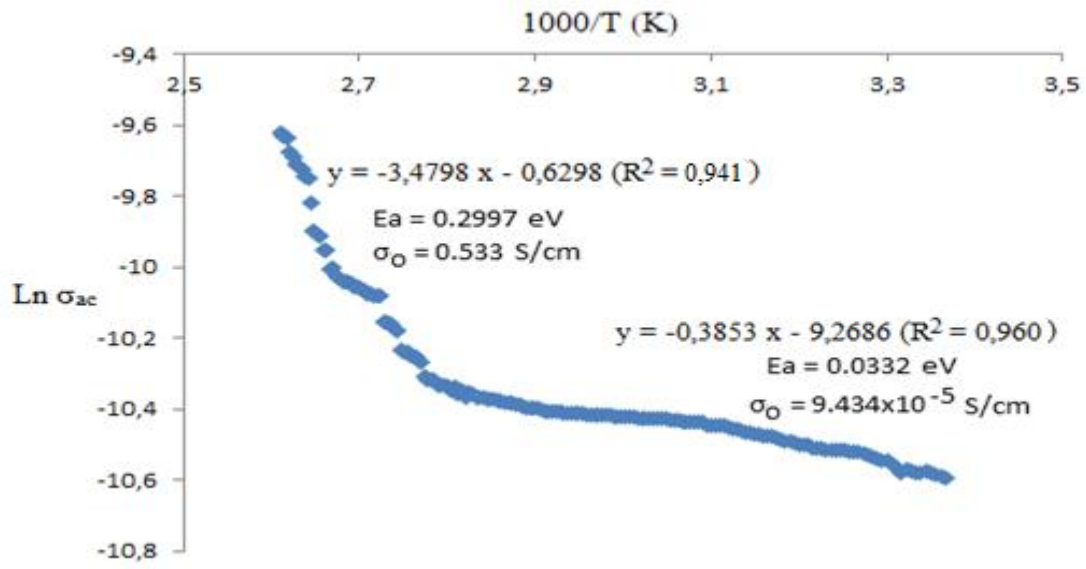
The joint solution of these two lines showed that the Tg temperature was 85.2 ºC.

For above T_g, slope = $-E_a/k$ = -3.4798 x 1000

E_a= (3.4798x1000) $1.38x10^{-23}$ J x 1 eV/$1.602x10^{-19}$J = 0.2998 eV.

For below T_g, E_a= 0.0332 eV. The activation energies of A.C. conductivity show that A.C. conductivity above Tg depends more on the temperature than conductivity below Tg.

In addition, doping with graphite increased slightly the activation energies both below Tg and above Tg.



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