



**PRODUCTION OF CERAMIC
NANOPARTICULATE REINFORCED POLYMER
MATRIX COMPOSITES AND
CHARACTERIZATION OF THEIR MECHANICAL
PROPERTIES**

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**PRODUCTION OF CERAMIC NANOPARTICULATE REINFORCED
POLYMER MATRIX COMPOSITES AND CHARACTERIZATION OF
THEIR MECHANICAL PROPERTIES**

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January 2022**

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Milad Omar Salem ALSADAIE

ABSTRACT

M. Sc. Thesis

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In recent studies, to improve thermal and antibacterial properties of polyethylene, copper is added to matrix. However, addition of copper to polyethylene matrix cause to decrement of mechanical properties despite its positive effect on thermal properties. Thus, mechanical properties of copper/polyethylene composites are needed to be enhanced using a reinforcement material. This study aims to investigate effect of vanadium carbide content on thermal, mechanical and tribological properties of copper/polyethylene matrix composites. For this purpose, copper/polyethylene composites containing nano vanadium carbide particles in different ratios (0.5 wt.%, 1 wt.%, 2 wt.%) were produced by injection molding. In order to examine thermal properties of samples, differential thermal analysis (DTA) was conducted. Tensile, flexural and impact tests were applied to understand mechanical properties of

composites. Reciprocating wear test was performed under different loads (20N, 30N, 40N) to examine tribological properties. Also, the cross-sectional surfaces of samples were investigated by Scanning Electron Microscope (SEM). The results show that thermal stability of PE/15Cu composite was improved by addition of VC. Also, hybrid composites including 1 wt.% VC had nearly 17% and 15% higher flexural strength and impact energy, respectively. However, VC addition caused to decrease of wear resistance when the reinforcement ratio is more than 0.5 wt.%.

Keywords : Polyethylene matrix composites, injection molding, copper, vanadium carbide.

Science Code : 92503

ÖZET

Yüksek Lisans Tezi

SERAMİK NANOPARTİKÜL TAKVİYELİ POLİMER MATRİS KOMPOZİTLERİNİN ÜRETİMİ VE MEKANİK ÖZELLİKLERİNİN KARAKTERİZASYONU

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Son zamanlarda yapılan çalışmalarda, polietilenin termal ve antibakteriyel özelliklerini iyileştirmek için matrise bakır eklenmektedir. Ancak polietilen matrise bakır ilavesi, termal özellikler üzerindeki olumlu etkisi göstermesine rağmen mekanik özelliklerin azalmasına neden olmaktadır. Bu nedenle, takviye malzemesi kullanılarak bakır/polietilen kompozitlerinin mekanik özelliklerinin iyileştirilmesi gerekmektedir. Bu tez çalışması, vanadyum karbür içeriğinin bakır/polietilen matrisli kompozitlerin termal, mekanik ve tribolojik özellikleri üzerindeki etkisini araştırmayı amaçlamaktadır. Bu amaçla, enjeksiyon kalıplama ile farklı oranlarda (ağırlıkça %0,5, ağırlıkça %1, ağırlıkça %2) nano vanadyum karbür partikülleri içeren bakır/polietilen kompozitleri üretilmiştir. Numunelerin termal özelliklerini incelemek için diferansiyel termal analiz (DTA) yapılmıştır. Kompozitlerin mekanik özelliklerini anlamak için

çekme, eğilme ve darbe testleri uygulanmıştır. Tribolojik özellikleri incelemek için farklı yükler (20N, 30N, 40N) altında aşınma testi yapılmıştır. Ayrıca numunelerin kesit yüzeyleri Taramalı Elektron Mikroskopu (SEM) ile incelenmiştir. Sonuçlar, PE/15Cu kompozitinin termal stabilitesinin VC ilavesiyle iyileştirildiğini göstermektedir. Ayrıca, ağırlıkça %1 VC içeren hibrit kompozitler, sırasıyla yaklaşık %17 ve %15 daha yüksek eğilme mukavemetine ve darbe enerjisine sahiptir. Ancak VC ilavesi, takviye oranı ağırlıkça %0,5'ten fazla olduğunda aşınma direncinin azalmasına neden olmuştur.

Anahtar Sözcükler : Polietilen matrisli kompozitler, enjeksiyonlu kalıplama, bakır, vanadyum karbür

Bilim Kodu : 92503

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SYMBOLS AND ABBREVIATIONS INDEX

SYMBOLS

A : alkaline

Cu : copper

mm : milimeter

N : newton

O : oxygen

Si : silicon

wt : weight

ABBREVIATIONS

ABS : Acrylonitrile Butadiene Styrene

DTA : Differential Thermal Analysis

MMC : Metal Matrix Composites

Nylon : Polyamides

PC : Polycarbonates

PE : Polyethylene

PET : Polyethylene Terephthalate

POM : Polyoxymethylene

SEM : Scanning Electron Microscope

VC : Vanadium Carbide

PART 1

INTRODUCTION

Composite materials: They are materials that are formed by the combination of two or more components in macro size and show special structural properties. Composite material components come together not by dissolving in each other, but by forming a physical interface and behave like a single material. In addition to their high strength, composite materials can be preferred over other materials due to their properties such as low density, high corrosion, electrical insulation, lower tooling, and assembly costs. Considering that the strength to lightness ratio is better than other engineering materials, composite materials; aerospace and aviation, maritime, automotive, building and construction has found use in many sectors [1].

In addition to the advantages of composite materials that allow it to be used in many sectors, it is one of the topics that are studied to be able to give different properties to these materials by adding various additives. The composite material sector is a sector whose usage area is increasing with the increasing momentum in the world. Especially, thanks to the innovations made on the matrix materials that make up the composite material and the fibers as the reinforcement element, composite materials with new combinations are produced and new materials with higher physical, mechanical, and chemical properties are emerging compared to other conventional materials [2].

Polymer matrix composite materials are among the most used types of composite materials. There are many properties that composite materials are desired to have according to their usage areas and these properties should be known in advance. In addition, polymer matrix materials are modified with inorganic particles due to their low bending strength, rigidity and abrasion resistance. Particle reinforcement significantly affects the physical and mechanical properties of polymer materials. The

particle characteristics and physical properties of these additives have the same influence on the properties of composites as those of simple fillers [3].

Carbide ceramics has low thermal expansion coefficient, high thermal conductivity, creep and wear resistance, high hardness and corrosion resistance. These properties make carbide ceramics the base material for structural components [4]. Furthermore, carbide ceramics are high-temperature, wear-and-corrosion-resistant. It has high temperature and variable electrical conductivity, and is mainly used in mechanical and chemical engineering, power engineering and microelectronics as well as aerospace engineering [5-7].

The antibacterial properties of copper have been known since ancient times. Copper compounds in different forms are used in many applications to provide hygiene. Ancient Egyptians used copper to clean drinking water or to prevent wounds from getting infected. The Romans, on the other hand, used copper in the treatment of diseases in many medical studies. While the Aztecs used copper in the treatment of sore throats, copper was used in the treatment of eye infections in Iran and India. In recent years, high-performance copper-containing materials have been developed and their application areas are expanding day by day [8].

In recent studies, to improve thermal and antibacterial properties of polyethylene, copper is added to matrix [9,10]. However, addition of copper to polyethylene matrix cause to decrement of mechanical properties despite its positive effect on thermal properties. Thus, mechanical properties of copper/polyethylene composites are needed to be enhanced using a reinforcement material. This study aims to investigate effect of vanadium carbide content on thermal, mechanical and tribological properties of copper/polyethylene matrix composites. For this purpose, copper/polyethylene composites containing nano vanadium carbide particles in different ratios (0.5 wt.%, 1 wt.%, 2 wt.%) were produced by injection molding. In order to examine thermal properties of samples, differential thermal analysis (DTA) was conducted. Tensile, flexural and impact tests were applied to understand mechanical properties of composites. Reciprocating wear test was performed under different loads (20N, 30N,

40N) to examine tribological properties. Also, the cross-sectional surfaces of samples were investigated by Scanning Electron Microscope (SEM).

PART 2

COMPOSITE MATERIALS

Composite materials: It is a new material group formed by two or more components showing different properties by forming an interface between them. One of the most important advantages of composite materials is that the new material formed shows superior properties compared to the components from which it is formed [11].

Natural composites are mostly found in the structure of animals and plants. The wood formed because of reinforcing long cellulose fibers (polymer component) with a component called lignin is a natural composite structure. Cellulose is also found in the structure of cotton and shows weak properties, so although lignin and cellulose alone have weak properties, they have formed a durable and new material together.

Bone found in the human body is also a natural composite material. Bone structure: It consists of hydroxyapatite crystals, a hard and brittle calcium phosphate ceramic, and collagen fibers, a ductile protein. Another composite material used since ancient times is concrete. Concrete containing gravel, sand and cement in its structure has good compressive strength values. The most common example of modern composite materials formed by supporting the matrix component with the reinforcement component is plastic reinforced glass fiber composite materials [12].

2.1. COMPOSITE MATERIALS AND TYPES

The composite materials are generally formed by the fact that the load-bearing reinforcing fibers are surrounded by the matrix component, which is a continuous phase with a certain location and orientation; They consist of three main components: reinforcing component, matrix component and interface. When the reinforcement component, which is another main component of composite materials, is taken as

reference, composites; fiber-reinforced composites, particle-reinforced composites, layered composites, scale-reinforced composites, and filled-structured composites [13]. The most common group is fiber reinforced, also called fiber composites. When these materials are produced in the form of fiber (fiber) in engineering, they are more preferred because they have higher strength values than the mass state. Particle reinforced composites: It is formed by random distribution of various particles in an alloy or ceramic matrix. For this reason, they are generally isotropic in nature. It has advantages such as improved strength, high operating temperature, and oxidation resistance. Pebble, sand, and cement used in reinforced concrete can be given as examples. They provide advantages such as high strength and low cost. However, they are difficult to manipulate and the materials that can be used are limited. Another component of composite materials is the interface. The stronger the interface, the better the mechanical properties of composite materials. Thanks to the interface, matrix molecules are attached to the reinforcement surface by creating a chemical reaction or adsorption. Thanks to its controllability, especially by atomic force microscopy (AFM), developments and changes in the interface have recently been monitored [13].

2.1.1. Metal Matrix Composites

Metal matrix composites are generally composite structures formed by the support of low-density metals such as magnesium and aluminum by a ceramic material with particles or fibers. Materials such as silicon carbide and graphite are often preferred as ceramic reinforcement. Compared with other metals, metal matrix composites show high strength and hardness, higher operating temperature, very good abrasion resistance [14].

Reinforcement of aircraft engines with ceramic whiskers and the development of 'in-situ' eutectics in the 1960s helped to improve high temperature applications. The automotive industry began to take metal matrix composites more seriously in the late 1970s. As metal matrix composites have matured over the last two decades, they have become a widely used and commercially viable material class [15].

The main methods used in the production of metal matrix composite materials are powder metallurgy and the casting method by squeezing liquid metal alloys into a porous ceramic structure. Metal matrix composites produced by these methods are preferred due to their good physical and mechanical properties in areas such as automotive and aircrafts and in defense technology [16].

2.1.2. Ceramic Matrix Composites

Ceramics have high melting point values and have superior properties than metals because they have good oxidation and corrosion resistance. In addition to these, although they are brittle, they are insufficient against thermal shocks [17].

Ceramic matrix composites are obtained by embedding ceramic fibers in a ceramic matrix. Various ceramic materials with oxide or non-oxide; They can be used as ceramic fiber and ceramic matrix. Due to the wide variety of fiber structures available, the mechanical and thermal properties of ceramic based (matrix) composites may differ for special constructions [18].

Glass ceramic matrix such as borosilicates and aluminosilicates, traditional ceramic matrix such as SiC, Si₃N₄, Al₂O₃, ZrO₂; Examples of ceramic matrix types used in ceramic matrix composites [19].

2.1.3. Polymer Matrix Composites

Polymer matrix composites are one of the most studied material groups used in the twentieth century. It is preferred in space, automotive, building, marine and sports applications due to its low density and high strength properties. Polymer matrix composites; It is formed by the presence of various short or continuous fibers in an organic polymer matrix. In contrast to the supporting component that improves fracture toughness in ceramic matrix composites, the supporting component in polymer matrix composites increases high strength and hardness [20].

Polymer matrix composites can be divided into two categories. These are reinforced plastics (polymers) and advanced composites. The distinction between the two categories can only be made in terms of mechanical properties generally in terms of strength and hardness. Reinforced plastics usually consist of polyester or epoxy resin reinforced with carbon or glass fibers. Advanced composites that have been used in the last fifteen years and have high strength and hardness properties are relatively more expensive. The most important advantage of these materials is that they are lightweight yet have high strength and hardness values. Compared to metals, corrosion resistance and fatigue resistance are better [20].

2.2. MECHANICAL PROPERTIES OF COMPOSITE MATERIALS

While most of the mechanical properties obtained by adding continuous reinforcing materials to the metal matrix are determined by the law of mixing ratios, this is not the case for composite materials with discontinuous reinforced metal matrix. In discontinuous reinforced composites, material properties vary in accordance with the volume ratio of the reinforcement phase. In continuously doped metal matrix composites (MMC), the modulus of elasticity, yield and tensile strength increase compared to the unadulterated matrix. This increase in mechanical properties is achieved by the fact that the fiber properties determine the properties of the composite, and the metal matrix transmits the load to the fibers in continuous fiber reinforced MMCs. In discontinuous doped MMCs, the metal matrix, which has a structure as if hardened with dispersion, is determinant in the properties of the composite.

Metal matrix composites maintain their mechanical properties such as high modulus of elasticity, high yield, and tensile strength under high temperature conditions, and have unique characteristics such as relatively low density, low thermal expansion coefficient, high wear resistance and resistance to environmental effects. The high temperature resistance of metal matrix composites is achieved by adding SiC, tungsten fibers or whiskers to the matrix phase [21].

In the strength of discontinuous reinforced composite materials, the type of the metal matrix, the type, shape, size, amount and uniform distribution of the reinforcement

material, the production method and process parameters of the composite, the phases formed within the microstructure, the compatibility of the matrix and the additive phase, and in addition to the heat treatment of the composite. many factors play a role. Subjecting the matrix phase to precipitation hardening provides extra strength to the composite material. In addition to the particles in the composite material, the presence of the precipitate phases increases the strength of the composite material. Particles should be homogeneously distributed in the composite material. The non-homogeneous distribution and agglomeration of the particles in the matrix phase causes the strength of the composite material to decrease. In case of agglomeration of the particles, a certain bond cannot be formed between the matrix phase and the particles, and the agglomerated areas behave as a void in the matrix. Therefore, composite material deforms at lower loads. Fine particles increase the strength of the composite material more than coarse particles. Particle shape also affects the strength of the composite material. Sharp angular particles play a stress-increasing role in the matrix phase. The commercial manufacture of particle-reinforced light metals, with their promise for cheap cost, high modulus and strength, excellent wear resistance, and ease of fabrication, is just beginning [22].

The density of the materials used as reinforcement is generally higher than the density of the matrix alloy. The addition of reinforcement particles to the matrix alloy increases the density of the metal matrix composite. Since the increase in strength after reinforcement is higher than the density, the specific strength of metal matrix composites is higher than the unadulterated matrix. For mechanical properties, hardness is described with respect to measurement methods, models, and experiments concerning particle reinforced MMCs [23].

The most important disadvantage of metal matrix composites is that they have very low ductility. The addition of ceramic particles to the metal matrix significantly reduces the ductility of the metal matrix. (The low ductility of metal matrix composites makes it difficult to apply processes such as extrusion, forging, rolling and machining to the composite material [22].

2.3. TRIBOLOGICAL PROPERTIES OF COMPOSITE MATERIALS

As the material science advances and improvements have been made, many new technologies such as metal matrix composites are developed to satisfy unique demands. The combined characteristics of metal and ceramics are composites of metal matrix. Silicon Carbide wear behavior demonstrating that the composites have slightly lower weight loss than the matrix alloy, which is reinforced by particles produced by liquid metallurgy technology. Casting with excellent machinability, strength, resistance to wear and corrosion has recently been patented by Oishi in cupro alloy [24].

While the load is less than 60 N and when the load exceeds 60 N, the frictional coefficient of the ceramic is smaller than the matrix alloy. These findings are shown in the Figure 2.1. The coefficient of friction in composites is observed in this figure to rise steadily and the matrix alloy to decrease quickly as load increases [25]. Recently, Shioda et al. have invented outstanding wear and slide alloy [24].

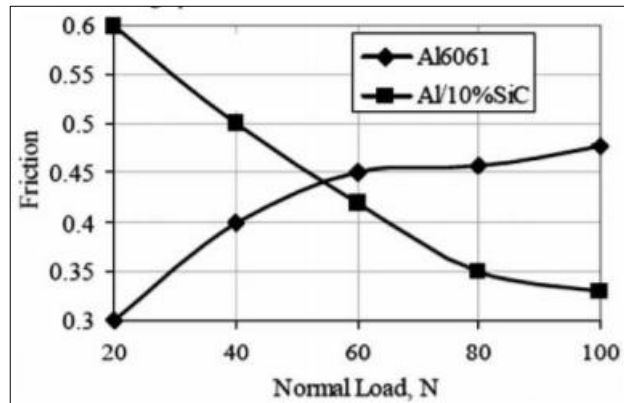


Figure 2.1. Coefficient of friction versus applied load for Al and Al/SiC [25].

2.4. THERMAL PROPERTIES OF COMPOSITE MATERIALS

Composites are anisotropically thermal conductive in nature. For accurate design, the knowledge of composite thermal conductivity is needed. Resin thermal conductivity data helps to reduce pressures associated with composite decreases during healing and maladjustment of thermal expansion coefficients. Awareness of the effects of different

parameters which affect thermal conductivity is important before experiments are conducted to determine the thermal conductance of different composites. The main benefit of using plastic filler material is to reduce the composite's total cost. To increase composites efficiency by increasing the crack resistance, minimizing declines, affecting mechanical power, strengthening the fire resistance and so forth, the filler material often acts as the main component. Calcium carbonate, kaolin (clay), aluminum trihydrates, etc. are the major filler materials used in composite production [26].

The main application of additive materials is to amend and customize the composite's material properties. It improves the processability or longevity of the composite by incorporating additives into the resin scheme. Additives tend to improve the output or a given property, as well as to increase product overall costs. Different additives often used to improve resin thermal and electrical conductivity include graphite powder, cut carbon fiber, carbon nanotubes, etc. Although the thermal conductivity of a composite polymer is driven by the conductivity of fibers and resins, which compose the polymer, resins typically separate, and fibers dominate the conductivity. The conductivity of the composite is affected by the compactness of fibers per field. The packaging of fibers in a composite relies on the production process. Manual hand-laying, compression molding, resin transfer molding, pultrusion, etc. are all different composite manual techniques [27].

PART 3

POLYMER MATRIX COMPOSITES

3.1. MATRIX MATERIALS

Matrix component of polymer matrix composites; It is examined in two main groups as thermoplastic matrix and thermoset matrix. The bond structure of these matrices is shown in Figure 3.1 [28].

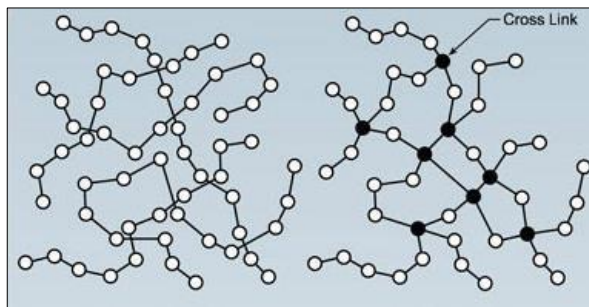


Figure 3.1. Thermoplastic structure (left), Thermoset structure (right) [28].

3.1.1. Thermoplastic Polymers

Thermoplastic materials are molecular structures consisting of long chains connected to each other by weak Van Der Waals bonds [29]. Thermoplastics: When heated, they soften and become fluid. Thermoplastics are recyclable materials, thanks to these properties that only cause changes in their physical structures without any change in their chemical structures [29]. Thermoplastic materials: It has found use in various sectors such as building-construction, aviation, automotive and space industry. The use of thermoplastic materials among the plastic materials used today is 80% [30].

3.1.1.1. Acrylonitrile Butadiene Styrene (ABS)

Acrylonitrile butadiene styrene: It is the general name given to the engineering polymer formed by the polymerization of acrylonitrile, butadiene and styrene monomers. Its density is 1.05 g/cm³. As can be seen in Figure 3.2, it has an important place among engineering polymers thanks to its strength and toughness values due to its dual-phase amorphous structure.

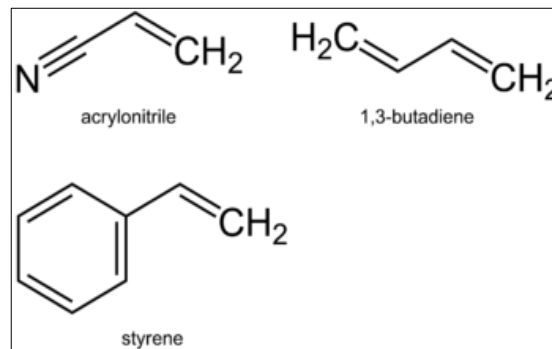


Figure 3.2. ABS structure [31].

ABS, which started to be used in the mid-1940s, has found many different areas of use because it has high rigidity and toughness properties as well as stress cracking resistance under heat, environment and chemical effects [31]. It can also be encountered in applications where the cost is tried to be reduced by mixing with nylon or different materials. The most common examples are items frequently used in daily life such as TV sets, telephones, hairbrushes, suitcases, hard hats, toys, sports equipment [29,31].

The properties of ABS polymer may vary depending on molecular or morphological parameters. Parameters such as matrix composition and molecular weight, volume fraction of continuous phase, particle size can be effective in determining ABS properties.

The optimum temperature range at which ABS polymer can work as tough and durable is between -30°C and +60°C. Compared to other thermoplastic materials, the polymer material that shows the best operating properties below zero degrees is ABS [32].

3.1.1.2. Polyoxymethylene (POM)

Polyoxymethylene; It is an important thermoplastic group that shows high abrasion and mechanical properties, good fracture resistance and moldability properties among the engineering polymers formed by the combination of -CH₂O- mers. It can be used in fields such as electricity, automotive, construction, biomedical applications [32]. The POM structure is shown in Figure 3.3.

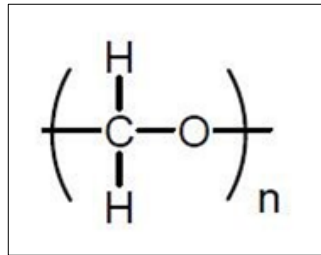


Figure 3.3. POM structure.

The POM structure is a highly crystalline structure, it is widely used in gears and bearings because it has very good friction properties. Besides its good mechanical properties, it has some disadvantages. Since the methylene-oxygen bonds in its structure are weak and easily breakable, depolymerization can be seen in formaldehyde. To prevent this, the structure can be modified with comonomers such as dioxane or dioxolane. In this case, when compared to the POM homopolymer initially, it can be said that the POM copolymer has better thermal stability and long-lasting mechanical strength [32].

POM: It is often used as a gear and bearing material as mentioned above, as well as for making nipples, plumbing connections, electrical connections and switches. Since its density is higher than other polymers, its use in the automotive industry is limited. In some applications, it may not be preferred because the production method is not easy and the cost may be high [32].

3.1.1.3. Polyamides (Nylon)

Polyamides or, as trade name, nylons; It is the name given to the engineering polymers developed in the 1930s, formed by the polymerization of amide (-CONH-) monomers. Nylon is a thermoplastic with a wide area of use, it can be used in materials such as food containers, cables, nylon fabrics, carpets, ropes. It can be examined commercially in two different categories, nylon 6 and nylon 6.6 [33].

3.1.1.4. Polycarbonates (PC)

Polycarbonates: It is an amorphous thermoplastic with a high Young's modulus and excellent mechanical and optical properties. It is a frequently preferred material group in recent years in military, ballistic applications, sports equipment construction, applications requiring high impact resistance [34]. Polycarbonates: carbonate groups (-OCOO-) are formed by bonding. The PC structure is shown in Figure 3.4.

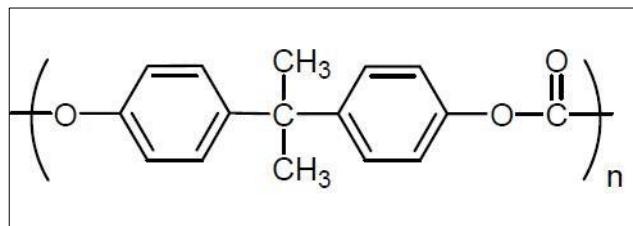


Figure 3.4. PC structure [35].

The most known polycarbonates: They are formed by the polycondensation of phosgene in bisphenol A and methylene chloride. The resulting hydrogen chloride can be removed using sodium hydroxide or a different material [28]. Since this polymer group is transparent, it has good light transmittance and can be used instead of glasses in some cases [35].

3.1.1.5. Polyethylene Terephthalate (PET)

Polyethylene terephthalate is a thermoplastic material that has found a wide area of use in the world, especially in the food and beverage industry, due to its low cost and

high-performance properties. The slow crystallization of PET and its high melting temperature at 290 °C compared to other polymers are among the advantages that increase the use of this material for the packaging industry [36].

PET, which has high mechanical properties and fracture toughness values, is a thermoplastic polymer that exhibits good chemical resistance up to 150°C to 175°C. Therefore, it can be used in many different applications such as packaging of beverages, pharmaceutical applications, photographic applications, X-ray films, electrical-electronic applications. To improve features in some applications; It can be used in combination with silicon, graphite or Teflon [36]. The PET structure is shown in Figure 3.5.

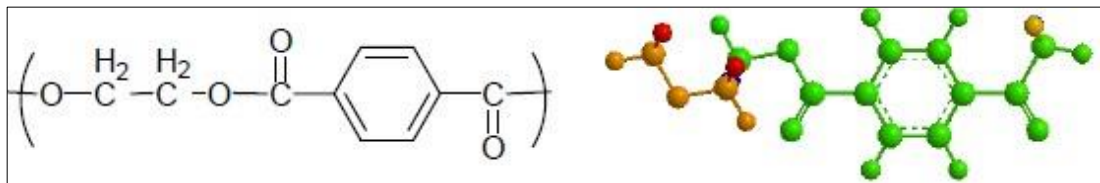


Figure 3.5. PET structure [35].

3.1.1.6. Polyethylene (PE)

PE is produced from the monomer ethylene, derived from petrochemical sources, over 80 million tons each year and has the chemical formula $(-\text{C}_2\text{H}_4)_n$ [37]. The physical and mechanical properties of PE are highly dependent on variables such as degree and type of branching (Figure 3.6).

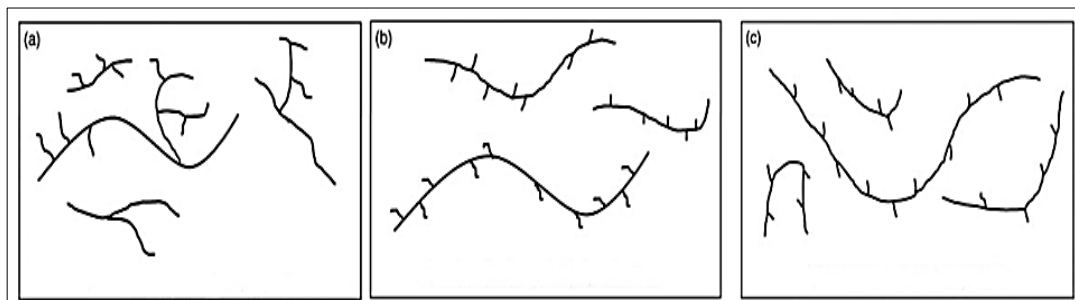


Figure 3.6. Structure of polymer chains, a) LDPE, b) LLDPE, c) HDPE [38].

Low density polyethylene (LDPE) is a branched thermoplastic, with the main molecular chain having relatively long branches. LDPE has lower tensile strength than high-density polyethylene (HDPE) due to the irregular packing of the polymer chains. Generally, LDPE is used for food packaging materials, plastic bags, film wraps, etc. used in plastic film applications. Medium density polyethylene (MDPE) has properties that fall between the properties of LDPE and HDPE. It has less branching than LDPE and more than HDPE. MDPE has lower stiffness and stiffness than HDPE, but these properties are not lower than LDPE. MDPE is typically found in gas pipes and fittings, sacks, bags, packaging film, plastic closures, etc. used in applications. HDPE consists of long chains and high crystallinity. HDPE is more rigid than LDPE due to its higher crystallinity content. It is used in packaging products such as HDPE, margarine, milk, detergent skewers, water pipes, etc. used in applications [38].

3.1.2. Thermoset Materials

Unlike thermoplastic polymer materials that melt with heat, thermoset materials; It is a group of materials that harden when compared to heat and therefore cannot be recycled. Epoxy, phenolic, unsaturated polyester, polyurethane, dicyanate, polyimide, acrylate and many other resin groups are in the thermoset material group [39].

Unlike thermoplastics, the chemical reactions that take place are related to their usage conditions. With the activation of heat or light, the curing mechanism starts in the thermoset material, and after this stage, cross-links are formed while an increase in viscosity is observed. Thermosets before curing; It exists as a mixture of small reactive molecules such as monomers and the curing mechanism is initiated by the addition of various catalysts. Many thermoset materials are used together with various additives or fiber supports to reduce costs or to gain various properties such as flame retardancy [39].

Thermosets: Thanks to its features, it provides the opportunity to be used in many areas. It can be used as an adhesive in the aerospace industry to bond primary and secondary structural elements, as conductive polymers in electronic applications, in

dental materials, and as an adhesive in sports equipment such as tennis rackets and bicycles [39].

Figure 3.7 shows the changes in the structure of thermosets from the gelation stage to the post-curing stage.

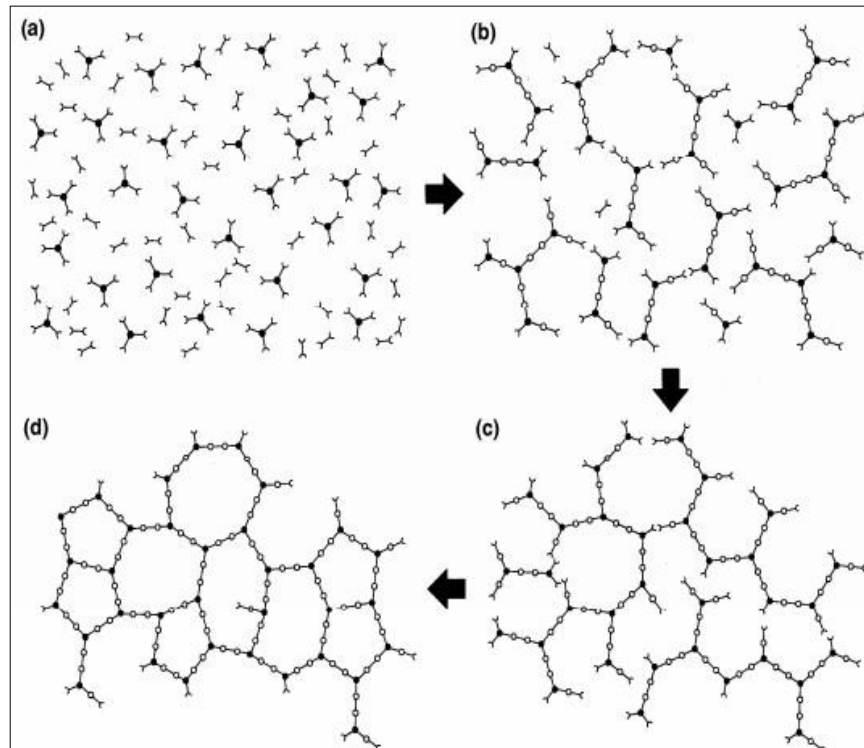


Figure 3.7. a) Reactive viscous structure, b) Beginning of gelation stage, c) End of curing process, d) Thermoset structure [39].

3.1.2.1. Unsaturated Polyester Resins

Unsaturated polyester resins: They are macromolecules formed by condensation polymerization of diol (1,3-propylene glycol), saturated acid (such as phthalic anhydrous acid) and unsaturated acid (such as maleic acid/anhydride). Unsaturated polyester resins are the general name given to polyester groups containing acid components in ester bonds [34,39]

Monomer can be added to provide crosslinking with double bonds found in unsaturated polyester resins [39]. Styrene can be added to reduce viscosity values, and chloromalic

acid can be added to provide flame retardant properties. Apart from styrene, active vinyl monomers such as vinyl toluene, diallyl phthalate or methyl methacrylate can be used to obtain various properties from polymers [34,39].

Catalysts such as methyl ethyl ketone peroxide or benzoin peroxide are used to initiate the curing mechanism of unsaturated polyester resins, and activator chemicals such as cobalt naphthalate and cobalt octanoate are used to activate the chemical reaction [39].

3.1.2.2. Phenolic Resins

Phenolic resins; They are macromolecules formed by condensation polymerization as a result of the reaction of an aldehyde group (usually formaldehyde) with phenol under alkaline ambient conditions [34,39]. Phenolic resin matrix composites are structures with very good flame resistance, good operating performance at high temperatures, long service life and chemical resistance [34].

Phenolic resins can be used easily in applications exposed to high temperatures. It is used in the nozzles of the rockets and the nose of the space rocket. Phenolic resins show good dielectric properties because they have dimensional and thermal stability [39].

3.1.2.3. Vinyl Ester Resins

Vinyl ester resins; They are epoxy-based resins formed by unsaturated polyester resins to which vinyl monomers ($\text{CH}_2=\text{CH}$) are attached, built on the excellent physical properties of epoxy systems [34,40]. They can be used in similar applications with polyester resins and generally in applications requiring higher performance [40].

3.1.2.4. Bisphenol A-diglycidyl ether type (BADGE)

This type of resins; They are formed because of the reaction of BADGE epoxy system with methacrylic acid. The resulting resin is diluted in styrene to produce a solids ratio

of at least 50%. BADGE type vinyl ester resins; They can be used in many applications requiring high performance such as chemical tanks or pipes [40].

3.1.2.5. Phenol Novolac Epoxy Type (EPN)

This type of resin is a reaction product of EPN and methacrylic acid, and the solids content diluted in styrene ranges from 30% to 36% [40]. Vinyl ester resins show more active behaviour in chemical reactions than polyesters, thanks to their molecular structures and the cross-links they contain. Due to their structure, these resins; it is also more resistant to water and chemicals [40].

3.1.2.6. Epoxy Resins

Epoxy resins; They are resins that started to be used in the early 1950s and have a wide range of use in industrial applications today [40]. Epoxy resins; They can be defined as low molecular weight oligomers containing two or more epoxy groups in each molecule [39]. Epoxies; It is a thermoset resin group whose properties such as toughness and hardness are activated by the addition of a curing agent in the plastics industry [40]. The epoxy chain and the oligomers form crosslinks thanks to the catalysts, and this gives the structure various properties such as resistance to solvents and chemicals, hardness, good adhesion properties [39].

3.2. PRODUCTION METHODS OF POLYMER MATRIX COMPOSITES

There are many production methods to meet different needs in the production of composites. Each of these methods has several advantages. For this reason, it is necessary to apply the appropriate production method to the material in order to achieve the desired quality in the final product by giving the desired shape to the composite.

These production methods are divided into two for metal and polymer matrix composites according to the matrix type of the composite material.

- Production methods of metal matrix composites
- Production methods of polymer matrix composites

Production Methods of Metal Matrix Composites:

1. Hot pressing
2. Powder metallurgy
3. Liquid metal impregnation
4. Electrolysis method
5. Steam precipitation
6. Rolling [41].

Production Methods of Polymer Matrix Composites:

Polymer matrix composites are of two types, thermoset and thermoplastic resinous. Accordingly, the production methods vary. Production methods of thermoset resin composite materials:

1. Hand deposit method
2. Spray method
3. Resin transfer moulding method
4. Helical winding method
5. Vacuum bagging method
6. Pultrusion method [42].

PART 4

REINFORCEMENT TYPES

4.1. FIBER REINFORCED COMPOSITES

Fiber reinforced composites, as the name suggests, are composites in which fiber is used as a reinforcement material. Although the compressive strength is good in composite materials that do not use fiber as a reinforcement element, many mechanical properties such as tensile, bending and notch impact strength are low. To improve these mechanical properties, fiber is used as a reinforcement element. In fiber-reinforced composite materials, the matrix material holds the fiber-reinforced element together and gives it rigidity. The volumetric ratios of the fiber and matrix material in the composite material change the mechanical properties of the composite material. As the fiber ratio in the composite material increases to a certain extent, the mechanical properties of the material increase and if the fiber ratio is further increased, the matrix material in the composite material will not be able to bind the fibers together, and on the contrary, it has a reducing effect.

Another feature that determines the strength of the composite material is the diameter of the fibers. The thinner the fiber diameter is, the better the resin wets the fiber, and a good bond strength is formed between the resin and fiber, which increases the strength of the composite material produced by providing a more homogeneous load distribution. An advantage of using fiber reinforcement is that even if the fiber bundle is damaged such as breaking, breaking, scratching due to the external load, this damage is not transmitted to other fiber bundles. If the same load occurs in a bulk glass material rather than a fiber, the crack may progress and cause damage to the entire material. The direction in which the fiber bundles are placed in the composite material is of great importance in the strength of the composite to be produced. Because if the load is applied to the material in the direction of the fiber, the strength of the material will be

high, while a load that will be applied in the perpendicular direction to the fiber will cause the strength of the composite to be low. For this reason, when the fiber material to be used is placed in two perpendicular directions or when different woven fiber fabrics are used, it has been observed that the strength increases in different directions. In addition, the fatigue strength and corrosion resistance of fiber-reinforced composite materials are very good. In terms of these features, they are preferred in many areas such as automotive, maritime, space, aviation, chemical industry, construction industry [38].

4.2. PARTICULATE REINFORCED COMPOSITES

Particle-reinforced composite materials are formed by combining the matrix material and the particulate reinforcing element in the size of macro or micro particles without dissolving in each other. Particle reinforcement is preferred in composite materials to improve the properties such as abrasion performance, electrical conductivity, hardness, and high temperature resistance of the produced composite material rather than improving the mechanical properties.

There are some negative situations in the production of particle-reinforced composites, these are that the matrix material and the particles cannot provide a homogeneous mixture and the particles cannot be fully wetted by the matrix material. In general, particle reinforcement creates a lowering effect on the mechanical properties of the composite material.

Therefore, they are preferred in the production of products used for decorative purposes. If the load applied to the composite material is compared by which of the matrix and reinforcement element is carried more; In fiber-reinforced composites, if the fibers are placed in bundles, up to 90% of the load is covered by the fiber bundles, while in particle-reinforced composites, the load is carried equally by the matrix and the particles [2].

4.3. LAYERED COMPOSITES

For layered composite materials, a composite layer is formed by combining the matrix material with reinforcing elements such as fibers, rods and braids. A layered composite structure is formed by combining multiple phases of these composite layers. Layered composites have properties such as high strength, high heat and corrosion resistance, and lightness. Layered composites; It is a type of composite that provides many different properties to the composite material by improving the existing properties of the materials by combining a matrix material that can create a good bond strength between the layers and at least two of the same or different material groups in the form of fiber, plate or coating [43].

By producing composite materials as layers; It is ensured that the mechanical (such as tensile, bending, compression, notch impact, abrasion performance) of the material, physical (such as thermal and electrical conductivity, lightness) and chemical properties (such as corrosion resistance) are improved [44]. For example, two different metal sheets can be brought together to give the material the desired thermal or electrical conductivity. By combining different oriented fiber layers, many desired mechanical properties can be created in a single material. By coating a metal with another metal material, it can be provided to have many different properties such as corrosion or abrasion resistance. Sandwich structures used in aerospace can be given as an example to layered composites [2].

4.4. HYBRID COMPOSITES

Composites produced by using at least two fiber types in the same composite material are called hybrid composites. Hybrid composites are composite materials prepared by using at least two or more fiber types. Here, too, the good physical, mechanical and chemical properties of certain types of fibers used are increased with the superior properties of other types of fibers, enabling the new product to obtain properties that we cannot obtain from a single fiber [44]. At the same time, a new generation of more economical composites can be developed with hybrid composites [2].

PART 5

VANADIUM CARBIDE AND COPPER

5.1. GENERAL INFORMATION OF VANADIUM CARBIDE

Several potentially valuable uses have been researched for some time in non-stoichiometric vanadium carbide. The material has a unique combination of characteristics, including high melting temperatures, high durability, a high heat strength and highly efficient thermal and electrical conductivity. More recently, the usage of hard transition metal carbide as functional coatings was also becoming more and more important [45], which had similar qualities to their bulk counterparts and were urgent to research new materials that might be used into VCs. WC was deemed particularly important from such materials.

The grid symmetry in stoichiometric VC is simple cubic with a Fm3m space group structure, although the material is susceptible to C vacancies. For the non-stoichiometric phases many structures were suggested and such carbides can include a large C vacancy in the ordered octahedral sublattice [46]. Different configurations have been suggested. A cubic phase seems to be generated from VC_{0.86} to VC_{0.88} and a double spacer of the cubic unit cell has been proposed to have in the space group of P4332 or P4132 the disordered carbide spacing [43]. An further ordered phase, appropriate for phase VC_{0.75}, may have a trigonal symmetry (P31 space) or monoclinic symmetry (C₂, or C_{2/m}), with stoichiometries less than VC 0.86 [47]. The energy behind the development of these vacancies in these various ordered phases was not examined from the point of view of an atomic study.

Moreover, the elastic characteristics of both stoichiometric and non-stoichiometric vanadium carbide have been studied. Brillouin extensive dispersal tests have recently been carried out for the Vanadium carbide range of VC 0.75 to VC 0.88 [48] and VC

0.85 to $P = 53$ GPa [49] for compression measurement. Regarding the bulk module and other elastic constants, such research seems to lead to a gradual decrease of the extent of the elastic constants as the concentration of carbon vacancies rises.

The VC vacancy of C reduces the material's elastic constants and, recently, the addition of the toilet could lead to cubic ternary V-W-C with a structure comparable to VC and maybe enhanced proprietary features [50].

5.2. USAGE OF VANADIUM CARBIDE IN COMPOSITES

High temperature strength, good resistance to corrosion, high chemical and thermal, and high hardness, stability are advantages of vanadium carbide, even when high temperature is present [51]; therefore its widespread use in industrial applications [52,53]. The mechanical properties and wear resistance of carbide composites are greatly enhanced by optimizing the composition ratios of carbon and vanadium elements [54]. The lifetime of the composites is thus increased. In 2006, J. G. Speer initially introduced the quenching and partitioning (Q&P) technique. The technique, which uses a carbon partition to stabilize austenite [55], can increase the plasticity and hardness. The Q&P procedure was implemented to alter the austenite ratio in the matrix to improve the mechanical properties of high-strength steels and their wearing behavior. The transformation-induced plasticity (TRIP) effect can lead to the creation of a hardening layer of the Q&P-treated specimen during wear tests [56]. The improved quenching and processing method and its TRIP impact improve the mechanical characteristics of the materials and their wear resistance. These innovative Vanadium-carbide-reinforced Fe-Matrix composites (VC-Fe-MCs) can therefore be used in certain industries as the substitute for high-chrome and high-manganese cast iron [57].

5.3. GENERAL INFORMATION OF COPPER

Copper, an essential element for living things, is in group I B of the periodic table. Copper can be found in free or complex form as a divalent cation in the tissues of organisms and can vary from 2 valences to 1 valence. Therefore, it is important for

biological events. Copper is used especially in the electrical industry, chemical industry, in the production of paints and wood preservatives, and in the production of algicides and fungicides. Although Copper is an essential element for life, it is one of the most toxic metals at high concentrations. Copper is an essential metal in trace amounts for all organisms and plays an important role in cell metabolism. It is present as a cofactor in the structure of various enzymes including biological processes such as growth, development and repair [58].

The antibacterial properties of copper have been known since ancient times. Copper compounds in different forms are used in many applications to provide hygiene. Ancient Egyptians used copper to clean drinking water or to prevent wounds from getting infected. The Romans, on the other hand, used copper in the treatment of diseases in many medical studies. While the Aztecs used copper in the treatment of sore throats, copper was used in the treatment of eye infections in Iran and India. In recent years, high-performance copper-containing materials have been developed and their application areas are expanding day by day [8].

5.4. USAGE OF COPPER IN COMPOSITES

The use of copper and its alloys in metal matrix composite materials is increasing day by day. made in areas such as electrical and electronic circuit elements designs have been coming to the fore lately. Reinforcement of the copper matrix with graphite particles materials with low thermal expansion coefficient and good conductivity are obtained. known to be possible. Copper-Molybdenum (Cu-Mo) alloys are well studied in the automotive and electrical industries [59-62]. Cu-Mo alloys have been recognized as a new type of composite in recent years due to their thermal conductivity and corrosion resistance [63]. Generally, copper can be used in polymer matrix composites to improve their thermal and antibacterial properties. However, addition of copper to polymer matrix cause to decrement of mechanical properties despite its positive effect on thermal properties.

PART 6

EXPERIMENTAL STUDIES

6.1. MATERIALS

Medium Density Polyethylene powders which have 3.1 MFI and 123 °C melting temperature (Total, Belgium) were used as matrix material. Cu powders (Purity: 99.99 %, Size: 325 mesh) and nano VC particles (Purity: 99.95%, Size: 550-750 nm) were purchase from Nanografi, Turkey.

6.2. FABRICATION OF COMPOSITES AND HYBRID SAMPLES

Composite sample production was completed in two steps. Firstly, powders were mixed in Turbula mixer for 2 hours according to composition given in Table 6.1. Secondly, mixed powders were fed to injection molding device and dog-bone shaped samples were produced (Figure 6.1).

Table 6.1. Compositions of composites.

Sample	PE (%)	Cu (%)	VC (%)
PE-15Cu	85	15	-
PE-15Cu-0.5VC	84.5	15	0.5
PE-15Cu-1VC	84	15	1
PE-15Cu-2VC	83	15	2



6.1. Produced dog-bone shaped samples.

6.3. CHARACTERIZATION

For DTA analysis, composites were heated up to 600 °C in a nitrogen atmosphere at the heating rate of 10 °C/min. The tensile test was conducted using a Shimadzu tensile testing machine at room temperature. The test speed was adjusted at 5 mm/min. As shown in Figure 6.2, flexural test was performed at 2 mm/min test speed using by 600 kN Zwick Roell Test Machine.

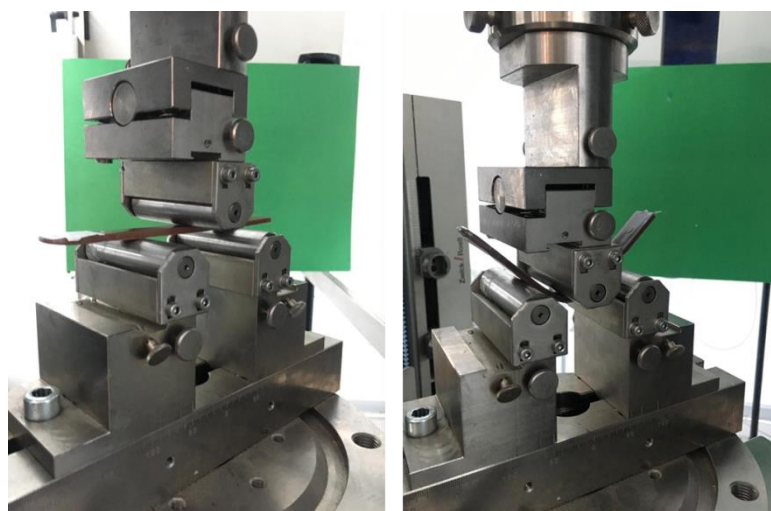


Figure 6.2. Image taken during the flexural test.

Charpy impact test was applied to unnotched samples which were shown in Figure 6.3 using a Zwick/Roell RKP 450 device.



6.3. Samples after impact test.

Zeiss Ultra plus Scanning Electron Microscope was used to investigate cross-sectional surfaces of samples after gold coating. The wear tests were applied under three different loads (30N, 40N, 50N) for a 100 m sliding distance, at 10 mm strokes and at room temperature (Figure 6.4).



6.4. Image taken during the wear test.

The coefficients of friction of the samples recorded by software of the test device. The wear rate was calculated dividing W_v (worn volume) to l (sliding distance). W_v was determined using a Mitutoyo SJ-410 profilometer instrument [64].

PART 7

RESULTS AND DISCUSSIONS

7.1. THERMAL PROPERTIES

DTA analyzes of samples were presented in Figure 7.1. It can be observed that there is no significant change in melting temperatures of composites. While the melting point of the PE-15Cu is 122.7 °C, PE-15Cu-2VC is 123.5 °C. However, the temperature corresponding to the maximum decomposition rate is changed by presence of nano VC particles. This temperature is shifted from 476.7 °C to 480.6 °C with addition of 1 wt.% VC to PE/Cu matrix. This indicated that the thermal degradation of PE/15Cu was delayed with reinforcing VC [65]. In other words, addition of VC led improvement of thermal stability of PE/15Cu. Melting temperature and temperature at maximum decomposition rate of samples were given in Table 7.1.

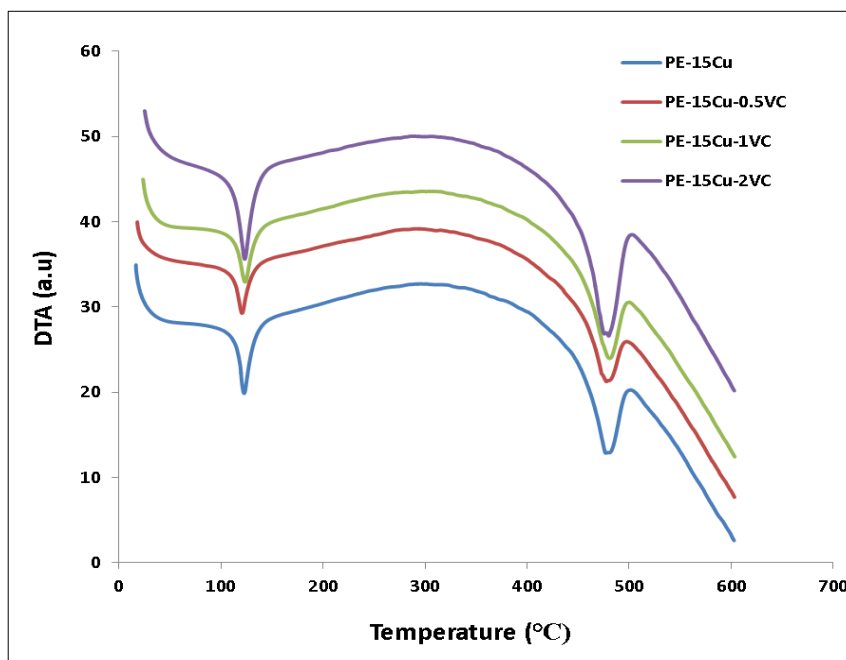


Figure 7.1. DTA curves of samples.

Table 7.1. Thermal properties of samples.

Sample	Melting Temperature (°C)	Temperature at maximum decomposition rate (°C)
PE-15Cu	122.7	467.7
PE-15Cu-0.5VC	122	478.6
PE-15Cu-1VC	122.7	480.6
PE-15Cu-2VC	123.5	479.7

7.2. MECHANICAL PROPERTIES

Stress-strain curves of samples were shown in Figure 7.2. It was observed that the all samples including vanadium carbide added did not show rupture up to elongation value of 500%. On the other hand, change in tensile strength of samples with vanadium carbide addition was not notable. Thus, it can be said that vanadium carbide content has no significant effect on tensile properties of copper/polyethylene samples. However, as shown in Figure 7.3, yield strength and flexural strength of PE/15Cu composite was improved nearly 6% and 17%, respectively. The increase in yield strength can be explained by the fact that nanoparticles can inhibit the movement of polymer chains [66].

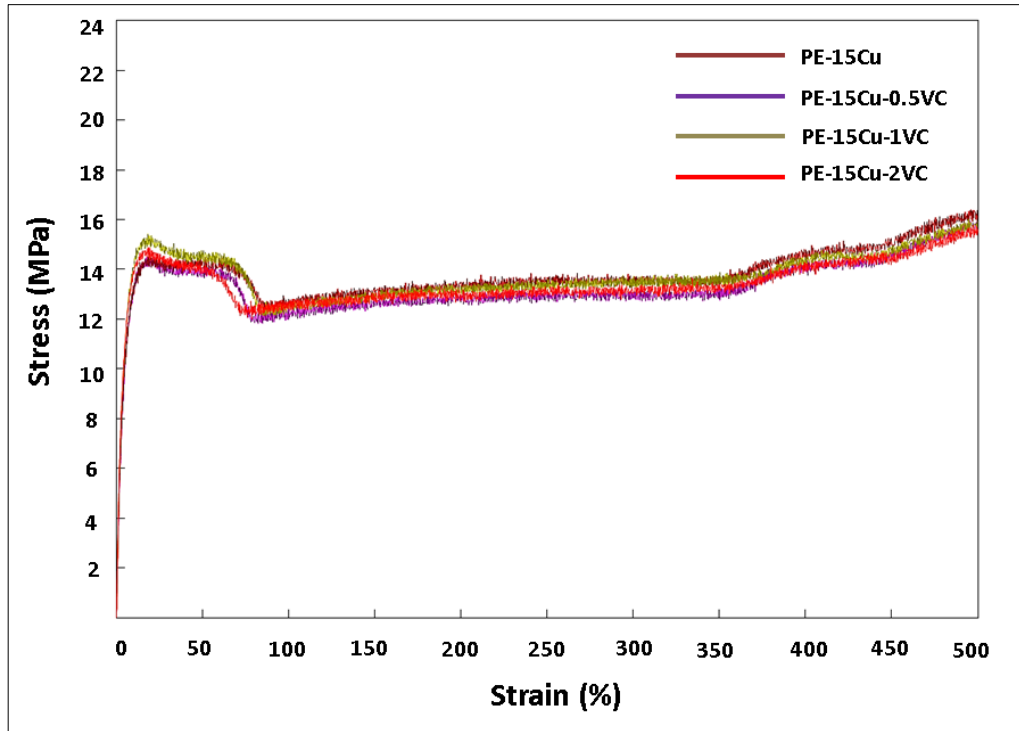


Figure 7.2. Tensile properties of composites.

On the other hand, it can be said that flexural strength of samples were also improved nearly 17% with addition of VC into PE/Cu composite. While the flexural strength of the PE-15Cu is 12.03 MPa, PE-15Cu-1VC is 15.41 MPa.

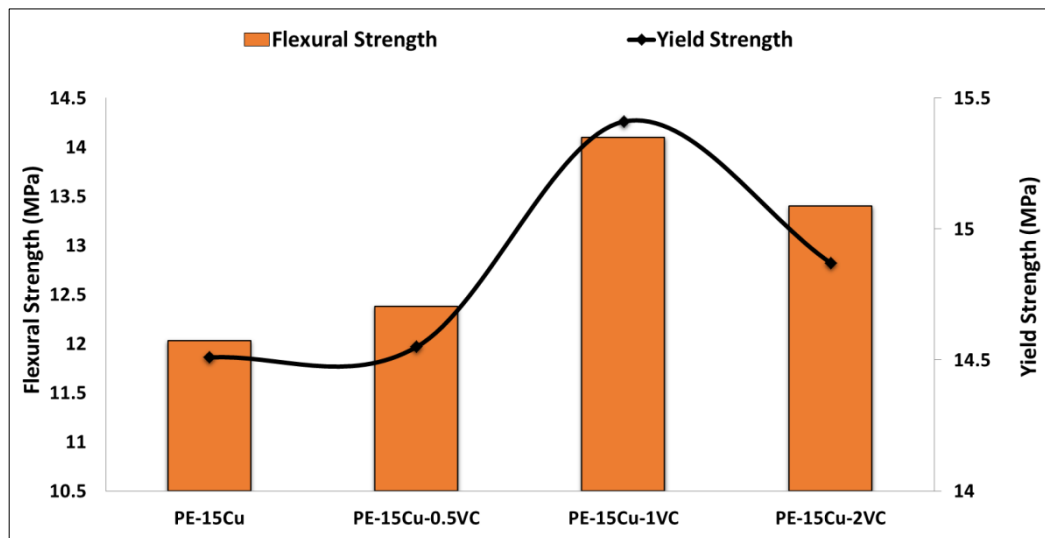


Figure 7.3. Flexural and yield strength of samples.

Figure 7.4 shows the impact energy values of samples. It can be said that the impact test results showed a similar trend with the flexural test results. The impact energy values of the samples increased up to 1% reinforcement ratio. Hence, the PE-15Cu-1VC sample had highest impact energy of 20.3. However, when the reinforcement ratio was increased from 1% to 2%, a decrease was observed in the impact energy value as well as the bending-yield strength. This can be explained by agglomeration of VC nano particles in PE matrix because agglomeration of VC nano particles may cause to formation of weak matrix-reinforcement bonding [67].

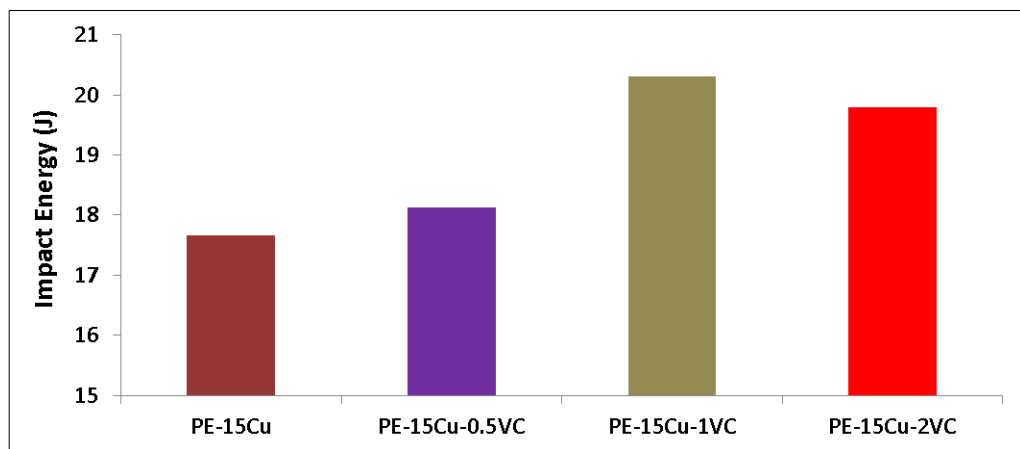


Figure 7.4. Impact energy of samples.

To confirm agglomeration of VC particles in matrix, SEM Mapping analyzes were conducted as shown in Figure 7.5. It can be seen that VC nanoparticles were homogeneously distributed in the matrix while the reinforcement ratio is 0.5 wt.% and 1 wt.%.

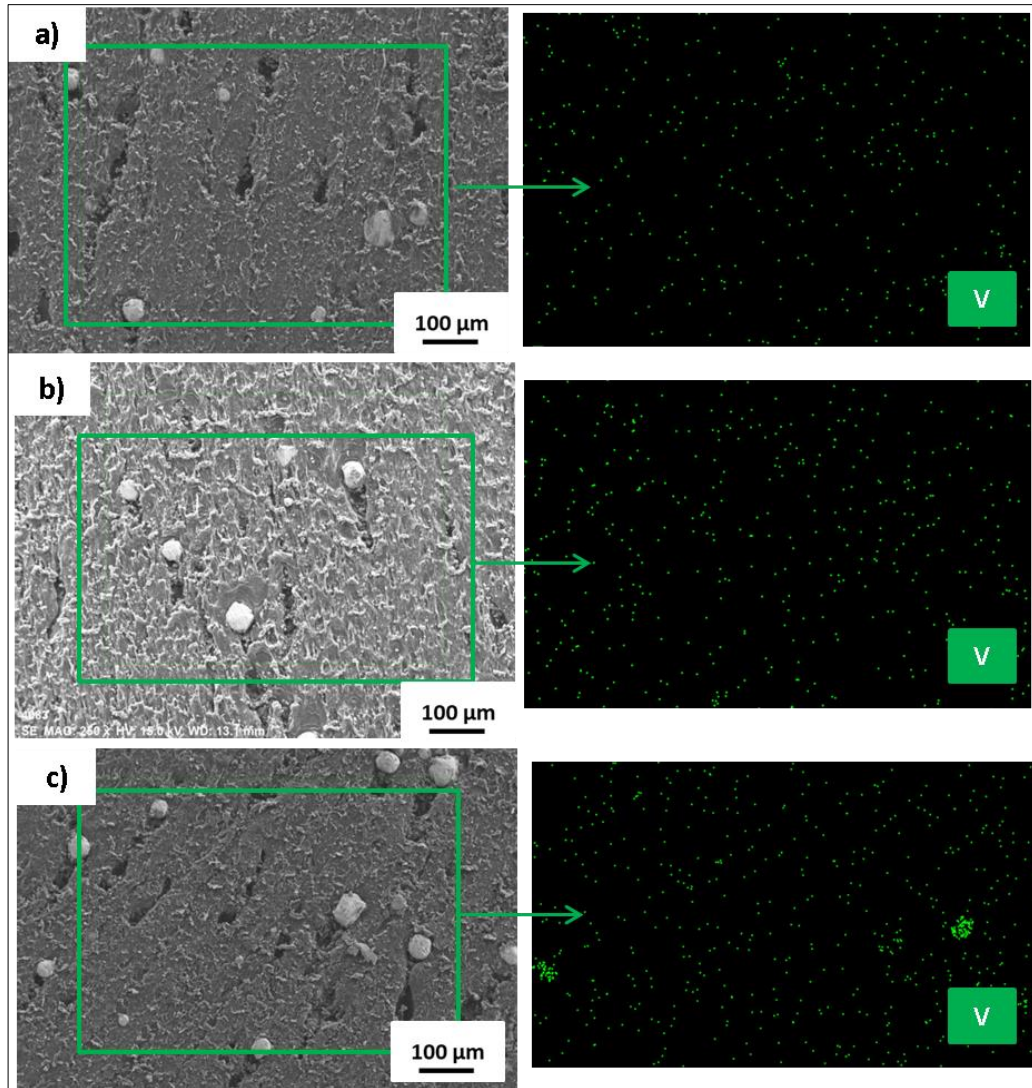


Figure 7.5. SEM images and SEM Mapping results of impact surfaces samples.

However, partial agglomerations were detected in the composite containing 2 wt.% VC. Therefore, this finding supported the decrease in mechanical properties. Mechanical properties of samples were presented in Table 7.2.

Table 7.2. Mechanical properties of samples.

Sample	Yield Strength (MPa)	Flexural Strength (MPa)	Impact Energy (J)
PE-15Cu	14.51	12.03	17.66
PE-15Cu-0.5VC	14.55	12.38	18.12
PE-15Cu-1VC	15.41	14.1	20.3
PE-15Cu-2VC	14.87	13.4	19.8

7.3. TRIBOLOGICAL PROPERTIES

Figure 7.6 represents the wear rates of samples versus applied loads. It was observed that only 0.5 wt.% VC added composite showed better wear resistance compared to PE/15Cu composite. When the VC reinforcement ratio was increased, wear resistance of samples decreased. Thus, PE-15Cu-2VC sample had highest wear rate among all samples. This is because the poor matrix-reinforcement interface due to agglomeration also cause to decrease of wear resistance [68].

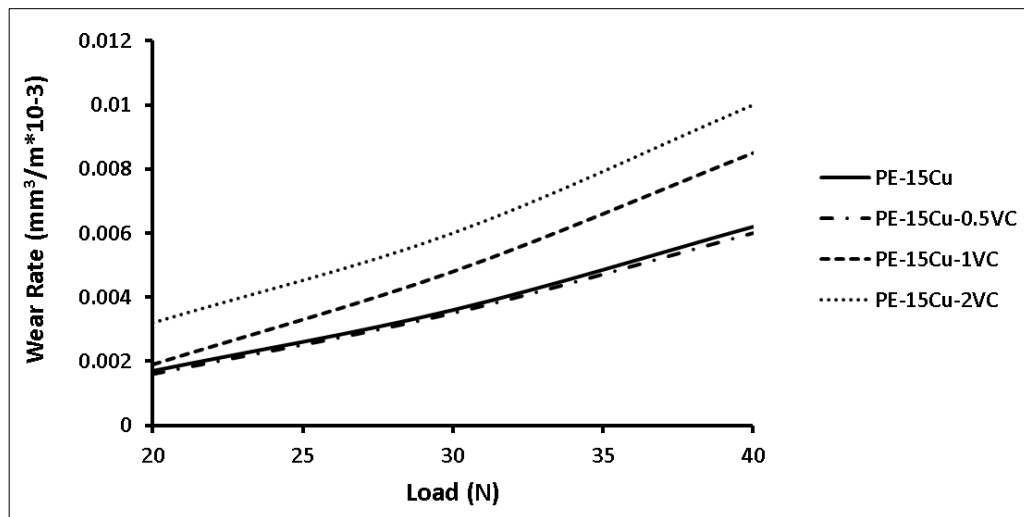


Figure 7.6. Wear rates of samples versus applied loads.

Agglomerated VC nanoparticles for PE-15Cu-2VC sample were presented in Figure 7.7. As shown in SEM-EDX results, VC nanoparticles were agglomerated in Area 3 in matrix.

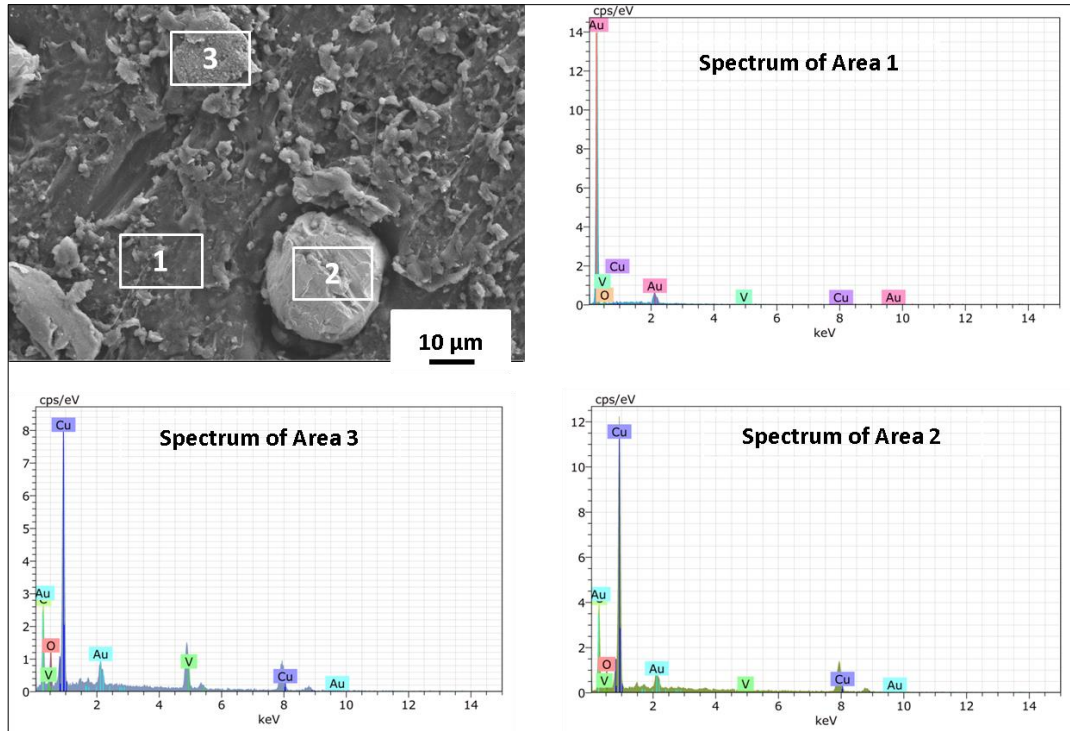


Figure 7.7. SEM-EDX results of PE-15Cu-2VC sample.

PART 8

CONCLUSIONS

In recent studies, to improve thermal and antibacterial properties of polyethylene, copper is added to matrix. However, addition of copper to polyethylene matrix cause to decrement of mechanical properties despite its positive effect on thermal properties. Thus, mechanical properties of copper/polyethylene composites are needed to be enhanced using a reinforcement material. In this study, vanadium carbide and copper reinforced polyethylene matrix hybrid composites have been successfully produced by injection molding. The effect of vanadium carbide content on thermal, mechanical and tribological properties of copper/polyethylene composites were investigated. The results of the characterization of produced samples can be summarized as below:

- 1) The addition of VC to PE/15Cu composite led enhancement of thermal stability of samples.
- 2) Also, yield strength, flexural strength and impact energy values of PE/15Cu composite were improved nearly 6%, 17% and 15% with addition of 1wt.% VC nanoparticles to matrix, respectively.
- 3) However, mechanical properties decreased when the reinforcement ratio was increased from 1 wt.% to 2 wt.% due to agglomeration of VC particles in matrix.
- 4) Agglomeration of VC particles in matrix was confirmed with SEM-Mapping analysis of cross section of samples.
- 5) The agglomeration of VC particles at higher amounts also caused to decrease in wear properties. Hence, while PE/15Cu-0.5VC had lowest wear rate, PE/15Cu-2VC showed worst wear resistance.

As a result, thermal, mechanical and tribological properties of PE/Cu composite were increased by reinforcing VC nanoparticles. In other words, PE/Cu/VC hybrid composite showed better mentioned properties compared to PE/Cu composite.

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RESUME

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