ÇUKUROVA UNIVERSITY
INSTITUTE OF NATURAL AND APPLIED SCIENCES

MSc THESIS

Feyza YEL

EVALUATION OF WASTE TYRE GRANULES AS AN FILLER MATERIAL IN POLYMER MATRIX COMPOSITE MATERIALS

DEPARTMENT OF AUTOMOTIVE ENGINEERING

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ABSTRACT

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As the consumption of substances increases with the increasing population, waste materials also increase. The use of these waste materials in the produced composite materials has started to become widespread because it can reduce the amount of waste. In today's world, vehicle tires are one of these wastes. In this study, waste rubber rubber granules were used as filling material for the production of polymer matrix composite samples. One of the most preferred matrix materials, polyester resin, and recycled waste tire rubber granules were combined in different proportions by open molding method. The samples were experimentally characterized by tensile, impact and hardness tests. The morphological features of the samples were examined by scanning electron microscope images. The study revealed that the addition of untreated waste tire rubber granules to polyester resin reduces the tensile and yield strength values, while increasing the elongation and ductility of the material. Morphological investigations have shown that the interfacial adhesion of waste rubber granules and polyester resin is poor, and therefore, waste rubber granules must be properly processed to be used as filling material. The study showed that waste tire rubber granule/polyester composites can be used in non-structural applications with benefits such as using less fossil-sourced raw materials and contributing to the reduction of environmental pollution.

Key Words: Composite, Waste Tire Granules, Polyester, Mechanical Properties

ÖZ

YÜKSEK LİSANS TEZİ

POLİMER MATRİSLİ KOMPOZİT MALZEMELERDE ATIK LASTİK GRANÜLLERİNİN DOLGU MALZEMESİ OLARAK DEĞERLENDİRİLMESİ

Feyza YEL

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Artan nüfus ile madde tüketimi artarken atık maddeler de artmaktadır. Üretilen kompozit malzemelerde bu atık maddelerin kullanılması atık miktarını azaltabildiği için yaygınlaşmaya başlamıştır. Günümüz dünyasında araç lastikleri bu atıkların başında gelmektedir. Bu çalışmada, polimer matrisli kompozit numunelerin üretimi için dolgu malzemesi olarak atık lastik kauçuk granülleri kullanılmıştır. En çok tercih edilen matris malzemelerinden biri olan polyester reçine ile geri dönüştürülmüş atık lastik kauçuk granülleri açık kalıplama yöntemi ile farklı oranlarda birleştirilmiştir. Numuneler, çekme, Charpy ve sertlik testleri yapılarak deneysel olarak karakterize edilmiştir. Numunelerin morfolojik özellikleri taramalı elektron mikroskobu görüntüleri ile incelenmiştir. Çalışma, polyester reçineye işlenmemiş atık lastik kauçuk granüllerinin eklenmesinin çekme ve akma dayanımı değerlerini düşürürken, malzemenin uzama ve sünekliğinin artmasına neden olduğunu ortaya koymuştur. Morfolojik incelemeler, atık kauçuk granüllerin ve polyester reçinenin ara yüzey yapışmasının zayıf olduğunu ve bu nedenle atık kauçuk granüllerin dolgu malzemesi olarak kullanılmak üzere uygun şekilde işlenmesi gerektiğini göstermiştir. Çalışma, atık lastik kauçuk granül/polyester kompozitlerinin, daha az fosil kaynaklı hammadde kullanılması ve çevre kirliliğinin azalmasına katkıda bulunması gibi faydaları ile yapısal olmayan uygulamalarda kullanılabileceğini göstermiştir.

Anahtar Kelimeler: Kompozit, Atık Lastik Granülleri, Polyester, Mekanik özellikler

EXTENDED ABSTRACT

Rapid population growth and growing industries in the world increase consumption while increasing productivity. As a result, the amount of waste increases rapidly and disrupts the ecological balance. Most of these wastes that threaten the future of the world are polymeric wastes. Polymeric wastes are formed as a result of the consumption of polymer materials that we use in many areas of our daily life, which are basically divided into two classes as natural and synthetic according to their sources. While wastes made from natural polymers degrade easily in nature, the same is not true for synthetic polymers. Intensive production and use of these materials, which do not disappear in nature in a short time, create a large-scale waste problem.

To prevent waste from increasing, it must be produced first; then the generated waste is expected to be recycled. Waste disposal is a last resort since polymeric wastes contain toxic and soluble components and the products produced during disposal adversely affect the biodiversity in the region.

A significant amount of polymeric waste is generated when vehicle tires become unusable. Evaluating these tires in composite materials is one of the research topics that researchers focus on. As a result of the research, it has been evaluated that waste tire rubber can be used in different areas. Rubber exhibits a very high elastic behavior due to its very low Young's modulus and higher yield stress as a result of vulcanization. It has a thermoset cross-linked structure that makes the rubber durable and highly resistant to environmental factors. Rubber also has some valuable properties, such as damping or reducing vibration, improved wet grip, relative resistance to gas and permeability to water, and reducing swelling in hydrocarbon oils. Due to these important properties, it has been evaluated that it can be used in composite materials.

New materials obtained by physically mixing at least 2 different materials are called composite materials. Composite materials are not alloys. There is no

exchange of atoms between the components. That is, they do not affect each other chemically. If the components are dissolved in each other, these materials are called alloys. Composite materials usually have a main component called 'matrix' and consist of another component called 'reinforcing element'. There are different production methods to produce composite materials such as hand lay-up, injection molding, press molding, reinforced thermoplastic sheet molding, resin transfer molding, extrusion, injection molding, vacuum molding, autoclave, and vacuum infusion. Composite materials have some advantages and disadvantages compared to other engineering materials. Its main advantage is that its designs can be easily adapted to the area of use and desired features. In addition to these features, they also have advantages such as low density, durability, and high strength. On the other hand, they also have disadvantages such as production cost and being anisotropic.

The main purpose of this research thesis is to investigate the usability and advantages of composite products produced using recycled rubber granules (RRG) from waste tires in various applications and in the automotive industry. The use of recycled rubber granules as a reinforcement element aims to produce more environmentally friendly composite materials. In the composite structures produced in this study, two different polymer resins, the matrix material of which are polyester and vinyl ester, were preferred and the products were produced by the open molding method. To fabricate the samples, a homogeneous mixture was obtained by first mixing the accelerator (0.2% by weight) and the resin. Then the recycled waste tire granules were added to the mixture and mixed mechanically for 10 minutes. The hardening chemical (2% by weight) was added and mixed for an additional 10 minutes. After obtaining a homogeneous mixture, the mixture was poured into an open mold at room temperature. The samples were cured at room temperature for 24 hours and then the samples were removed from the molds. The mechanical structures of the produced composite structures were examined by applying the Charpy impact test, hardness test and tensile test, and their

morphological structures by SEM analysis, and whether their use in the automotive industry is more suitable for our ecosystem. According to the results of the research, adding waste rubber granules to the structure weakens the structure and increases the ductility of the material. In addition, it was observed in SEM analyses that the combination of untreated waste tire rubber granules and polyester matrix creates poor interfacial adhesion that needs to be improved with appropriate treatment. Finally, it was stated that the use of waste tire granules for the production of composite materials could be a promising alternative solution for sustainable and environmentally friendly production by recycling fossil-sourced raw materials and reducing the amount of use.

GENİŞLETİLMİŞ ÖZET

Dünyada hızlı nüfus artışı ve büyüyen sanayiler tüketimi artırırken verimliliği de artırmaktadır. Sonuç olarak, atık miktarı hızla artmakta ve ekolojik dengeyi bozmaktadır. Dünyanın geleceğini tehdit eden bu atıkların çoğu polimerik atıklardır. Polimerik atıklar, temel olarak kaynaklarına göre doğal ve sentetik olmak üzere iki sınıfa ayrılan, günlük hayatımızın birçok alanında kullandığımız polimer malzemelerin tüketilmesi sonucu oluşmaktadır. Doğal polimerlerden yapılan atıklar doğada kolayca bozunurken, sentetik polimerler için aynı şey geçerli değildir. Doğada kısa sürede kaybolmayan bu malzemelerin yoğun üretimi ve kullanımı büyük ölçekli bir atık sorunu yaratmaktadır.

Atıkların artmasını önlemek için önce üretilmesi; daha sonra üretilen atığın geri dönüştürülmesi beklenir. Polimerik atıkların toksik ve çözünür bileşenler içermesi ve bertaraf sırasında üretilen ürünlerin bölgedeki biyolojik çeşitliliği olumsuz etkilemesi nedeniyle atık bertarafı son çaredir.

Araç lastiklerinin kullanılamaz hale gelmesiyle önemli miktarda polimerik atık oluşmaktadır. Bu lastikleri kompozit malzemelerde değerlendirmek araştırmacıların odaklandığı araştırma konularından biridir. Araştırma sonucunda atık lastik kauçuğunun farklı alanlarda kullanılabileceği değerlendirilmiştir. Kauçuk, çok düşük Young modülü ve vulkanizasyon sonucunda daha yüksek akma gerilimi nedeniyle çok yüksek bir elastik davranış sergilemektedir. Kauçuğu dayanıklı ve çevresel etkenlere karşı oldukça dirençli hale getiren termoset çapraz bağlı bir yapıya sahiptir. Kauçuk ayrıca, titreşimi sönümleme veya azaltma, geliştirilmiş ıslak kavrama, gaza karşı nispeten direnç ve suya karşı geçirgenlik ve hidrokarbon yağlarında şişmeyi azaltma gibi bazı değerli özelliklere de sahiptir. Bu önemli özelliklerinden dolayı kompozit malzemelerde kullanılabileceği değerlendirilmiştir.

En az 2 farklı malzemenin fiziksel olarak karıştırılmasıyla elde edilen yeni malzemelere kompozit malzemeler denir. Kompozit malzemeler alaşım değildir.

Bileşenler arasında atom değişimi mevcut değildir. Yani kimyasal olarak birbirlerini etkilemezler. Bileşenler birbiri içinde çözülürse bu malzemelere alaşım denir. Kompozit malzemeler genellikle 'matris' adı verilen bir ana bileşene sahiptir ve 'takviye elemanı' adı verilen başka bir bileşenden oluşur. Kompozit malzemeler I üretmek için el yatırması, püskürtme, pres kalıplama, güçlendirilmiş termoplastik levha kalıplama, reçine transfer kalıplama, ekstrüzyon, enjeksiyon kalıplama, vakum kalıplama, otoklav, vakum infüzyonu gibi farklı üretim yöntemleri bulunmaktadır. Kompozit malzemelerin diğer mühendislik malzemelerine göre bazı avantaj ve dezavantajları vardır. Başlıca avantajı, tasarımlarının kullanım alanına ve istenilen özelliklere göre kolaylıkla uyarlanabilmesidir. Bu özelliklerinin yanı sıra düşük yoğunluk, dayanıklılık ve yüksek mukavemet gibi avantajlara da sahiptirler. Öte yandan üretim maliyeti ve anizotropik olmaları gibi dezavantajları da vardır.

Bu araştırma tezinin temel amacı, atık lastiklerden geri dönüştürülmüş kauçuk granüller (RRG) kullanılarak üretilen kompozit ürünlerin çeşitli uygulamalarda ve otomotiv endüstrisinde kullanılabilirliğini ve avantajlarını araştırmaktır. Takviye elemanı olarak geri dönüştürülmüş kauçuk granüllerin kullanılması daha çevre dostu kompozit malzemeler üretmeyi amaçlamaktadır. Bu çalışma ile üretilen kompozit yapılarda matris malzemesi polyester ve vinil ester olan iki farklı polimer reçine tercih edilmiş ve ürünler açık kalıplama yöntemi ile üretilmiştir. Numuneleri imal etmek için önce hızlandırıcı (ağırlıkça %0,2) ve reçine karıştırılarak homojen bir karışım elde edildi. Daha sonra geri dönüştürülmüş atık lastik granülleri karışıma ilave edildi ve 10 dakika mekanik olarak karıştırıldı. Sertleştirici kimyasal (ağırlıkça %2) ilave edildi ve 10 dakika daha karıştırıldı. Homojen karışım elde edildikten sonra karışım oda sıcaklığında açık kalıba dökülmüştür. Numuneler 24 saat oda sıcaklığında kürlenmiş ve ardından numuneler kalıplardan çıkarılmıştır. Üretilen kompozit yapıların mekanik yapıları Charpy darbe test, sertlik testi ve çekme testi, morfolojik yapıları SEM analizi uygulanarak otomotiv endüstrisinde kullanımlarının ekosistemimiz için daha uygun olup olmadığı incelenmiştir. Araştırma sonuçlarına göre yapıya atık lastik granüllerinin eklenmesi yapıyı zayıflatırken, malzemenin sünekliğini de arttırmaktadır. Bunun yanı sıra SEM analizlerinde işlenmemiş atık lastik kauçuk granülleri ve polyester matris kombinasyonunun, uygun işlemle iyileştirilmesi gereken zayıf ara yüzey yapışması oluşturduğu gözlemlenmiştir. Son olarak atık lastik granüllerinin kompozit malzeme üretimi için kullanılmasının, fosil kaynaklı hammaddelerin geri dönüştürülmesi ve kullanım miktarının azaltılması ile sürdürülebilir ve çevre dostu üretim için umut verici bir alternatif çözüm olabileceği belirtilmiştir.

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Thus, I finished my academic life, which lasted for 4 years, on April 30, 2022.

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ABBREVIATIONS

F1	:	Formula 1
MMAs	:	Metal Matrix Composites
CMCs	:	Ceramics Matrix Composites
PMCs	:	Polymer Matrix Composites
RRG	:	Recycled Rubber Granules
NA	:	Natural Aggregate
TA	:	Tire Aggregate
BI	:	Brittleness Index
EQI	:	Elastic Quality Index
ASTM	:	American Society for Testing and Materials
PVC	:	Polyvinyl chloride
MPa	:	Megapascal
HV	:	Vickers Hardness
SEM	:	Scanning Electron Microscopy

XIX

1. INTRODUCTION

In the world, rapid population growth and growing industries increase productivity while increasing consumption. As a result, the amount of waste increases rapidly and disrupts the ecological balance (Phale, 2005). Most of these wastes, which threaten the future of the world, are polymeric wastes (Pacheco-Torgal et al., 2012).

Polymeric wastes are formed as a result of the consumption of polymer materials that we use in many parts of our daily lives, which are basically divided into two classes as natural and synthetic according to their source. While wastes made of natural polymers are easily degradable in nature, the same is not true for synthetic polymers (Ministry of National Education, 2014). Intensive production and use of these materials, which do not disappear in a short time in nature, creates a large-scale waste problem.

Polymer materials are used in many areas from automotive to food. It can be said that it is heavily involved in tire production in the automotive field. Around 1.4 billion units (approximately 17 million tons) of waste tires are produced every year all over the world (Onay and Koca, 2015) and a large amount of these are stored in the open or thrown into landfills, causing major environmental problems and fire hazards(Kebritchi et al., 2013).

In order to prevent the increase of waste, it should not be produced first; then the generated waste is expected to be recycled. Disposal of waste is the last resort because tires contain toxic and soluble components and the products produced during disposal negatively affect the biodiversity in the area (Day et al., 1993).

Alternative uses of tire rubber, which can create value-added products, are among the subjects that researchers have focused on in recent years (Fernández et al., 2012; Laresgoiti et al., 2004; Murillo et al., 2006). As a result of the research, it has been evaluated that waste tire rubber can be used in different areas. In fact, it has begun to be thought that tire rubber, like other polymers, will provide significant benefits to humanity. Rubber exhibits a very high elastic behavior due to its very low Young's modulus and higher yield stress as a result of vulcanization. It has a thermoset cross-linked structure that makes the rubber durable and highly resistant to environmental factors. Rubber also has some valuable properties, such as damping or reducing vibration (Bin Samsuri, 2010; Imbernon and Norvez, 2016), improved wet grip, relatively resistance to gas and permeable to water, and reducing swelling in hydrocarbon oils (Ashour et al., 2015; Riyajan et al., 2012). Due to these important properties, it has been evaluated that it can be used in composite materials.

New materials obtained by physically mixing at least 2 different materials with each other are called composite materials. Composite materials are not alloys. Exchange of atoms between components is not available. That is, they do not chemically affect each other. If the components dissolve in each other, these materials are called alloys. Composite materials generally have a main component called 'matrix' and It consists of another component called 'reinforcing element (Mazumdar, 2001).

In ancient times, composite materials such as wicker bricks were used extensively as building materials. However, its relative importance declined in the early 20th century due to the use of metals and polymers (Kaw, 2006). However, with the significant development of technology, the relative importance of composite materials has greatly increased (Ashby, 1992).

The emergence of composite materials is not new. Composites have been used by mankind for a very long time. However, new developments in engineering technologies have brought along new needs that monolithic materials alone cannot provide. Composites are generally man-made materials, but there are also examples of composite materials in nature. For example, a tree trunk or bones are good examples of composites in nature (Yıldızhan, 2020).

One of the most important features of the composite material is that it can be adapted to the design criteria according to the place where it will be used. With the developments in the field of engineering, the desired properties from materials began to become more qualified, while composite materials began to come to the fore because they can have these properties. When the main materials that bring the composite materials together are considered separately, they show very different properties from the product. In other words, it can be said that it combines the strong features of its raw materials.

Composite materials have some advantages and disadvantages compared to other engineering materials. Its main advantage is that its designs can be easily adapted according to the area of use and desired features. In addition to these features, they also have advantages such as low density, durability, and high strength. On the other hand, they have disadvantages such as production cost and being anisotropic (Ashby, 1992; Kaw, 2006; Mazumdar, 2001). In summary, the advantages and disadvantages of composite materials can be listed as follows:

Advantages;

- There is no corrosion problem. Choosing the matrix and material is of great importance in this,
- It has been saved in weight,
- Their fatigue resistance is quite high,
- Some of the composites have very high yield point (yield stress) values,
- Design flexibility,
- It is long-lasting,
- Low density,
- Durability,
- Electrical Properties; with the selection of suitable materials, composite products with very superior electrical properties can be obtained,

- High specific strength (strength/density) and modulus (modulus/density),
- Corrosion and chemical resistance.

Disadvantages;

- It has different mechanical properties in different directions. Tensile, compression, shear, and bending strength values are different for the same composite materials,
- Manufacturing difficulty; production difficulty for some composites compared to metals,
- Production method influences material quality, quality standardization is a problem,
- Recycling is generally absent,
- Low fracture elongation.

Nowadays, it is clearly seen that the use of composites has increased. The areas where these materials, whose usage rate varies according to the sectors they are in, are mostly used as automotive, aviation, construction, and energy. According to the composite Turkey magazine published by the Turkish composite industrialists' association, the market share of composite materials in the world market has reached 84 billion and a production volume of 11.1 billion tons. For Turkey, the composite industry reached a production volume of 1.3 billion Euros and 242000 tons. Composite materials, which have great advantages over traditional materials, are expected to continue to increase (Turkish Composites Manufacturers Association, 2019). The market share percentages of composite usage in the world, Europe, and Turkey are as shown in Figure 1.1.

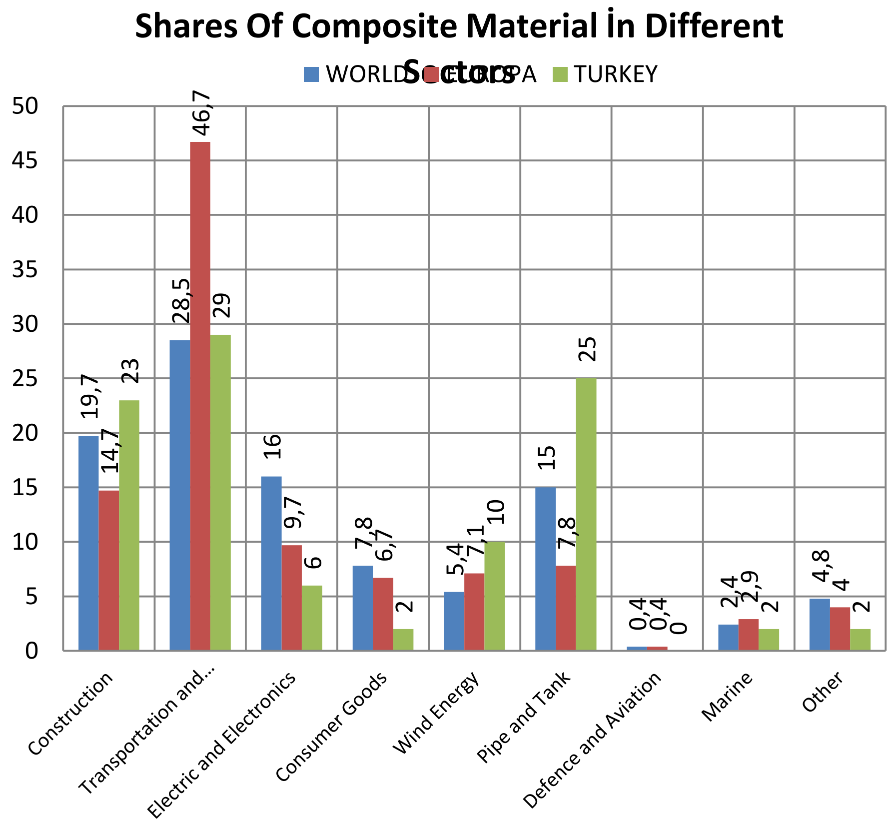


Figure 0.1. Shares of composite material in different sectors (Turkish Composites Manufacturers Association, 2019)

As stated above, composite materials have a wide range of uses, from sports to aviation, from automotive to energy. It has started to be preferred by manufacturers because it can be adapted to the design more easily than traditional materials. In addition, the fact that it has strong features such as lightness in unit volume, energy absorption ability, and high strength, which traditional materials cannot show, has been the most important factor in their use in sectors such as automotive, defence, aerospace and energy.

Formula 1(F1) vehicles can be given as examples for the use of composite materials in the automotive field. Regulations for the construction of Formula 1 vehicles are very specific and strictly enforced. The total weight of the vehicle should not exceed 605 kilograms. Design engineers are striving to reduce vehicle weight by switching from aluminum, a lightweight metal material, to composite materials. Hardness can be doubled with much lower weights using composite materials. It is very important for the vehicle to be light so that it can accelerate or get rid of it with little damage in case of an accident. In addition, the number of parts required in the production of F1 cars to be produced with complex parts and composite materials can be reduced. Composite materials are used in many parts of the vehicles, including the engine cover, nose cap, front and rear wings, spoiler, chassis, electrical distribution panels, and fasteners such as bolts

Examples of usage applications of composite material in other sectors are as follows;

Aerospace; Structural components, frames, wing skins, fuselage (Mangalgırı, 1999), spars, rudders, rotor blades, fins, elevons.

Construction; Bridge decks, profiles, columns, prefabs.

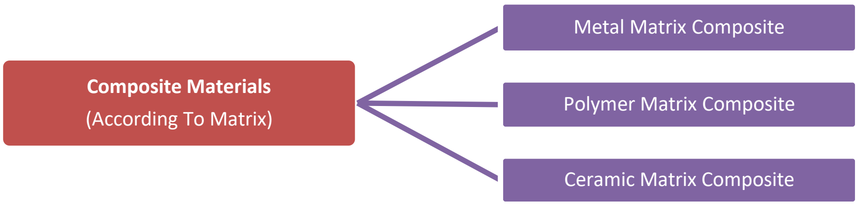
Defence and military; Aircraft bodies, helmet, vest.

Marine; Boat, lifeboats.

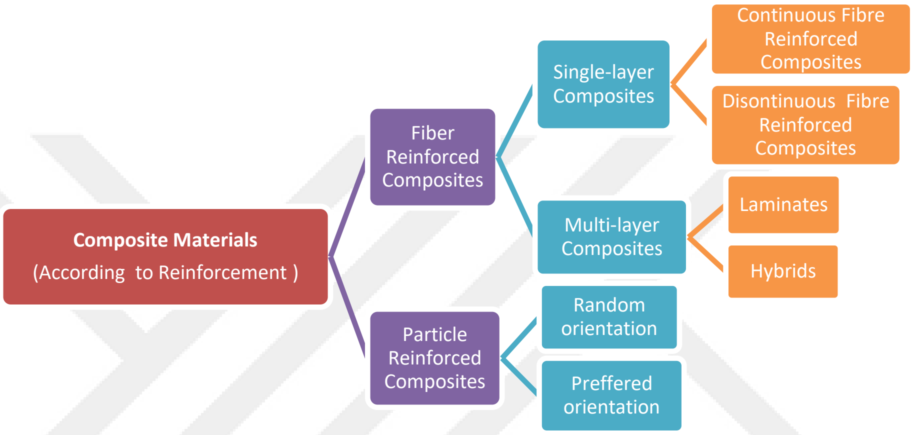
Rail; Bogie, train interior parts.

Energy; Isolators, antennas, fuses, cables, carriers.

Composite materials can be classified according to the matrix material, reinforcement material, or reinforcement type. In Figure 1.2, a general classification of composite materials is shown.



(a)



(b)

Figure 0.2. General classification of composite materials a) according to matrix classification of composite material b) according to reinforcement classification of composite material (Kinloch et al., 2015; Yıldızhan, 2020)

In composite materials, any conventional material such as ceramic, metal, or polymer can be used as matrix material. Composite materials are classified according to their matrix materials as metal matrix composites, ceramic matrix composites, and polymer matrix composites. Metal matrix composites (MMA) are generally produced by reinforcing a low-density metal component such as magnesium and aluminum with a reinforcing element. Because MMCs are extremely costly, they are used only when they need to meet important specifications. MMCs are used in internal combustion engines, robots, high-speed machinery, and aerospace (Kumar et al., 2018).

Generally, the problem of ceramic materials, which are used as a matrix for their high resistance properties against high temperatures, is that they have a very fragile structure. Therefore, the ceramic matrix is strengthened with reinforcement, and higher fracture toughness is obtained. Ceramics Matrix Composites (CMCs) are used in medical, military, aerospace, and automotive applications (Naslain, 2005).

Polymer matrix composites (PMCs) are produced using a polymer as the matrix material. The most widely used matrix in composite materials is the polymer matrix. The polymer used can be natural or synthetic. This type of composite, which can be quite light and high-strength, is used in racing cars, sports equipment, aerospace, and the military as a spall-liner.

The reinforcement materials are generally long-length fibers of small diameter or particles of fibre small diameter and a short length. Small particles are also used as reinforcements in some applications. Composite materials are generally classified as fiber or particle composite materials according to the type of reinforcement. Figure 1.2 shows a general classification of composite materials by the type of reinforcement. As a fiber, the most common reinforcements are synthetic carbon, aramid or glass, and jute, cotton, flax, bamboo, etc. such as natural fibers. In addition, plastic granules or polystyrene particles are reinforcement materials used in other types of composite materials. The type of reinforcement is a very critical parameter as it directly changes the mechanical properties of the final product and therefore it must be carefully selected according to the design criteria.

The main purpose of this research thesis is to investigate the usability and advantages of composite products produced using recycled rubber granules (RRG) from waste tires in various applications and in the automotive industry. The fact that recycled rubber granules will be used as reinforcement element aims to produce more environmentally friendly composite materials. In addition, the introduction of a new particle-reinforced material aiming at minimum cost will be ensured. In the composite structures produced with this study, two different polymer resins, the matrix material of which are polyester and vinyl ester, were preferred, and the products were produced by open molding method. By analyzing the mechanical, thermal properties, and microscopic-macroscopic structure of the produced composite structures, it has been examined whether their use in the automotive industry is more suitable for our ecosystem. Thus, such studies will increase the share of environmentally friendly, cost-effective, renewable, and less harmful materials in the automotive industry. It is thought that the results to be obtained in the light of these studies will give an idea about the future studies on waste rubber granule reinforced polymer composite materials.

2. LITERATURE SURVEY

Developing technologies, industrial requirements, and an increasing amount of waste lead researchers to evaluate waste materials in composite materials, which are the combination of at least two different materials. Since the variety of raw materials (different reinforcements, matrices, fillers, additives, coupling agents, etc.), different production methods, as well as production parameters (reinforcement type, weight ratio, etc.), creates a lot of variabilities, there are many missing points in composite material studies exist. Due to the flexible adaptation opportunity in composite materials, a wide variety of composite materials with different properties are produced, where wastes such as rubber granules are also used in the production. This causes many deficiencies in composite material research. This shortcoming has led researchers to focus on this issue.

Generally, researchers used concrete (cement) or polymer (epoxy, polyester, vinyl ester, etc.) as matrix material, while using waste tire rubber and granules as reinforcement or filling material. Bravo et. al. (2012) evaluated the performance of concrete containing rubber aggregate made from used tires in terms of durability. Shrinkage, carbonation and chloride penetration resistance, water absorption by immersion and capillarity tests of the produced concrete composite structure were performed. In this study, concrete mixes were produced in which 5%, 10% and 15% of the volume of natural aggregate (NA) were replaced by aggregate derived from used tires (TA). According to results of tests; compressive strength is highly affected by the replacement of NA with TA, with a reduction of approximately 50% for a replacement ratio of 15%, water absorption by immersion increases with the replacement ratio of NA by TA, carbonation resistance is significantly affected by the incorporation of TA, finally, chloride penetration resistance decreases as the NA/TA replacement ratio increases.

Li et al. (2004) made comparisons by adding waste tire products in the form of particles and fibers into the concrete mix. In addition, a hybrid fiber was formed by adding polypropylene fiber to the fibers produced from waste tires and they observed the change in strength. Compressive strength, compressive elasticity modulus, Poisson ratio, and split tensile strength tests were performed on the products. Fiber reinforcements have shown superior properties to particle reinforcements, but the difference is not great. In addition, the hybrid reinforced sample gave better results than the waste tire fiber-reinforced sample by about 25% in terms of compressive strength, about 25% in terms of split tensile strength, and approximately 12% in terms of elastic modulus.

Ho et. al. (2012) produced a new concrete composite by replacing 40% by volume of natural sand in concrete with rubber aggregates produced from waste tires. The effect of rubber aggregate on Brittleness Index (BI) and damage formation was investigated by performing three-point bending tests on the notched beam. In addition, the Acoustic Emission technique was applied to determine the damage mechanism in the concrete. In addition, the Elastic Quality Index (EQI) was calculated at different temperatures. According to the results of the tests; As the amount of rubber in the concrete increases, the Brittleness Index decreases and is close to zero for a concrete composite containing 40% rubber aggregate. The Elastic Quality Index of the rubberized composite decreases with increasing temperature. EQI values for concrete composite containing up to 40% rubber aggregate remain within the limits for pavement design on the ground. According to these results, it has been evaluated that waste tire aggregates can be used in concrete.

Shen et. al. (2013) prepared polymer-rubber aggregate modified porous concrete as pavement course material and investigated the properties of these samples such as strength, stress-strain, abrasion resistance, impact resistance and microstructure. He created 3 types of samples; The first sample is without rubber aggregate, the second is with coarse aggregate, and the third is with fine aggregate.

There are seven replacement values varying from 0% to 30%, i.e. 0%, 5%, 10%, 14%, 18%, 22% and 30% in both testing serials of the second and third. Shen et al. observed that the optimum replacement ratios of coarse aggregate and fine aggregate samples were 14% and 18%, respectively, and obtained maximum flexural strength and compressive strength at these ratios, and the flexural strength of composite samples with rubber aggregates increased significantly compared to samples with mineral aggregates. In addition to these results, by replacing mineral aggregate with rubber aggregate, the elastic modulus of polymer modified porous concrete decreases, wear and impact resistance is significantly improved, polymer-rubber aggregate modified porous concrete shows ductility.

Aslani et. al. (2018) produced self-compacting rubberized concrete by replacing natural aggregates in self-compacting concrete with aggregates produced from waste tires. 3 types of samples were formed with aggregates of 2 mm, 5 mm and 10 mm diameters. In the study, three granule rubber sizes were changed at 10%, 20%, 30% and 40% by volume. In total, they produced 12 kinds of samples. The 7 and 28 day compressive and tensile strengths, hardened densities and 28 days compressive stress-strain behavior of the samples were tested. According to the test results; All rubber aggregates replaced by 10% achieved a compressive strength higher than 30 MPa, and it was observed that the sample replaced by 10% with 5 mm aggregates reached a maximum compressive strength of 40.68 MPa. When evaluated in terms of rubber aggregate size, the samples formed from aggregates with a diameter of 5 mm provided the highest strength, while the samples produced with aggregates of 10 mm gave the worst results.

Jena et. al. (2020) produced a polymer matrix composite with an epoxy matrix by granulating waste tires that are normally burned and harm the environment when burned. They investigated the acoustic properties of these composite products, which they produced using the hand lay up method, using the impedance tube method using LABVIEW. Using stainless steel, PVC and glass to prepare impedance tubes according to ASTM standards, Jena et al. performed the tests between 500 Hz and 5000 Hz. The sound observation coefficients measured between these values were and averaged, and the Noise Reduction Coefficient, which indicates the absorption of sound energy, was calculated in this way. According to the results, it was evaluated that waste tire granules could be used as acoustic material. It has also been mentioned that it will have a great effect on reducing the waste tire rate.

In another study, Jena et. al. (2019) produced composites from waste tire granules by hand lay-up method. Using epoxy as matrix material, Jena et al. investigated the mechanical and physical properties of composite samples produced with different granule ratios (10%, 20%, 30%, 40%, 50%) by volume. In the research, it was observed that the inclusion of waste tire granules in epoxy increased the hardness, tensile strength, and water absorption properties of the polymer matrix composite, but it was observed that the modulus of elasticity, hardness and tensile strength decreased as the proportions of the granules increased. It has been concluded that the use of waste tires in composites by granulating is mechanically and physically positive, as well as it can play a big role in reducing the amount of waste.

He et al. (2021) et al. obtained a new thermoplastic composite product by using polyurethane and waste surface rubber rubbers. By adding 3.44% by weight of waste surface rubber to the polyurethane, the tensile strength was compared with pure polyurethane, resulting in an improvement of 166.7%. It was stated that the dynamic heat index, which indicates that the material has low dynamic heat, decreased from 1.62 in pure polyurethane to 0.97, which is relatively much lower. In other words, it was evaluated that the synthesized thermoplastic composite had higher strength and lower heat generation.

Elenien et. al. (2021) created a new composite by composing ground rubber granules with polyester resin. Shaping the granule and polyester resin mixture with a mold, Elenien et al. treated rubber granules using microwave energy. With this applied process, they aimed to increase the adhesion between the granules and the resin. In this research, mechanical and morphological tests of the samples produced by various processes were carried out. The effect of the treatment applied to the rubber particles was investigated. According to the test results, the offset yield strengths of the samples with rubber particles treated with microwave energy were higher. It was also observed that the interfacial adhesion between the treated slips and the resin improved.

Hittini et. al. (2021) investigated the usability of devulcanized rubber tires as fillers for thermoplastic thermal insulators. Devulcanized rubbers were pulverized and mixed with polystyrene at different rates (0-50% by weight). The mixture was then transferred to the hot press to finalize the sample. Afterwards, physical, thermal and mechanical tests of the produced samples were carried out. Composites with a devulcanized rubber tire content of less than 40% by weight; It has been observed that they have thermal conductivity ranging from 0.0502 to 0.07084 W/(m.K), density between 462.8 and 482.32 kg/m3, compressive strength between 11.66 and 7.47 MPa and flexural strength between 40.4 and 19.26 MPa. It has been stated that the new devulcanized rubber tire polystyrene composite can be used as an alternative insulation material due to its superior properties compared to traditional insulation materials.

3.MATERIAL AND METHOD

3.1. Materials

3.1.1. Waste Tire Granules

Waste tire rubber granules, which are in polymer structure, are produced by breaking up tires that are no longer used. Rubber granules were obtained from a local supplier in this study. The diameters of the granules vary between 2-5 mm and the density of waste rubber granules is 527 kg/m^3. The granules are included in the production as supplied from the supplier, without any changes. The ready state of the granules before they are combined with polyester is as seen in Figure 3.1.

Figure 0.1. Waste tire rubber granules

3.1.2. Matrix Resins

Vinyl ester and polyester were tried as matrix materials. The vinyl ester interacted poorly with the rubber granules. Therefore, while polyester and vinyl ester were being produced as a reference; they were compared by producing polyester-rubber granule composites. Polyester and vinyl ester resins used as matrix are polymer products. Some properties of the resins used are shown in Table 3.1 and Table 3.2. These resin types are solidified using accelerator and curing additives. Some properties of the additives used are shown in Table 3.3.

Table 0.1. Properties of polyester resin

Viscosity (cps)	350-450	Liquid Resin
Monomer content (%)	38-42	
Density (gr/cm^3)	1.1	
Tensile Strength (MPa)	65	Cured Resin
Elongation at Break (%)	2	

Table 0.2. Properties of vinyl ester

	Molding	**Laminate**
Heat distortion temperature (°C)	137	-
Flexural strength (MPa)	130-140	132
Bending coefficient (MPa)	3400-4000	7400
Viscosity (mPas)	180-220	
Tensile strength (MPa)	69-79	93
Barcol Hardness	42	50

Table 0.3. Properties of additives

	Curing Agent	**Accelerator**
Chemical	Methyl Ethyl Ketone Peroxide	Cobalt (II) 2-Ethyl Hexanoate
Brand	AKPEROX A60	AKCOBALT KXC6
% Used	2% of resin (by weight)	%0.2 of resin (by weight)

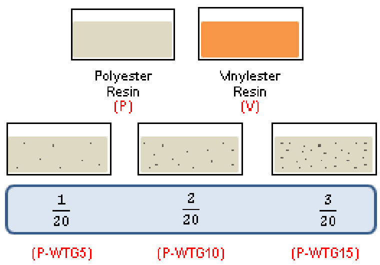
3.2. Methods

3.2.1. Production of Samples

Within the scope of the research, it was aimed to produce and examine the samples by determining 3 different percentages of content in order to see the effect of the waste tire rubber granules. It was decided to combine the polyester determined as the matrix material and the granules determined as the reinforcement material in the ratios of 100:0, 100:5, 100:10, 100:15 by weight. The percentage combinations and production designations of the samples are as shown in Table 3.4.

Table 0.4. Material ratios of the experimental samples

Sample Designation	Resin	Resin:Waste Tire Granule
V	Vinyl Ester	100:0
P	Polyester	100:0
P-WTG5		100:5
P-WTG10		100:10
P-WTG15		100:15



Afterward, a kind of mold release wax was applied to the surface and left for about 30 minutes. It enables the removal of produced composite material from the mold easier. Figure 3.2 showed the wax material and application of it.

Figure 0.2. Mold release wax application to the surface

The samples were produced with the open molding method. To the production of samples, firstly accelerator (0.2% wt.) and polyester resin were mixed and stirred to obtain a homogenous mixture. Then, the recycled waste tire granules were added to the mixture and mixed mechanically for 10 minutes. The hardener chemical (2% wt.) was added and 10 more minutes stirring was applied. After the homogeneous mixture is obtained, the mixture was poured into the open mold at room temperature. The samples were cured at room temperature for 24 hours and afterward, the samples were removed from the molds. The sample production procedure was applied for each sample and the schematic of the production processes are shown in Figure 3.3.

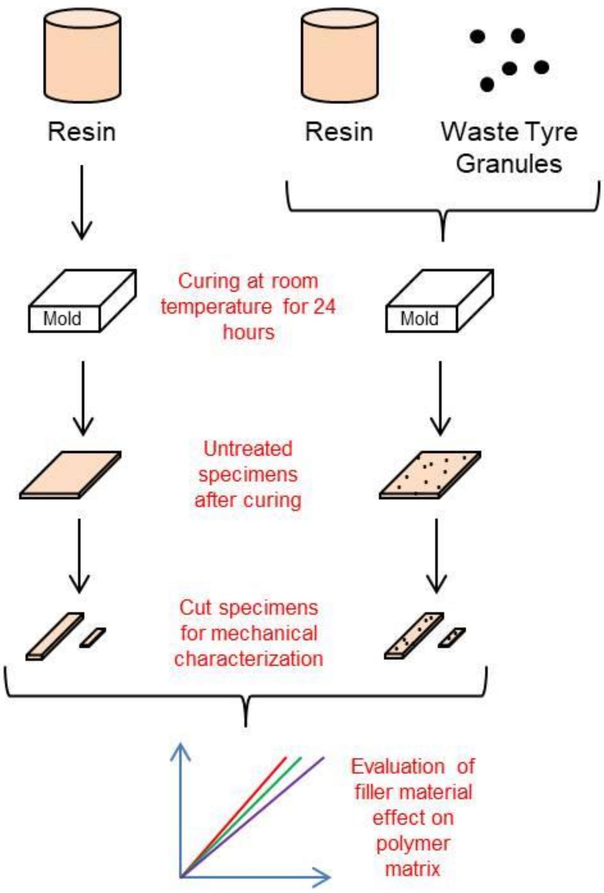


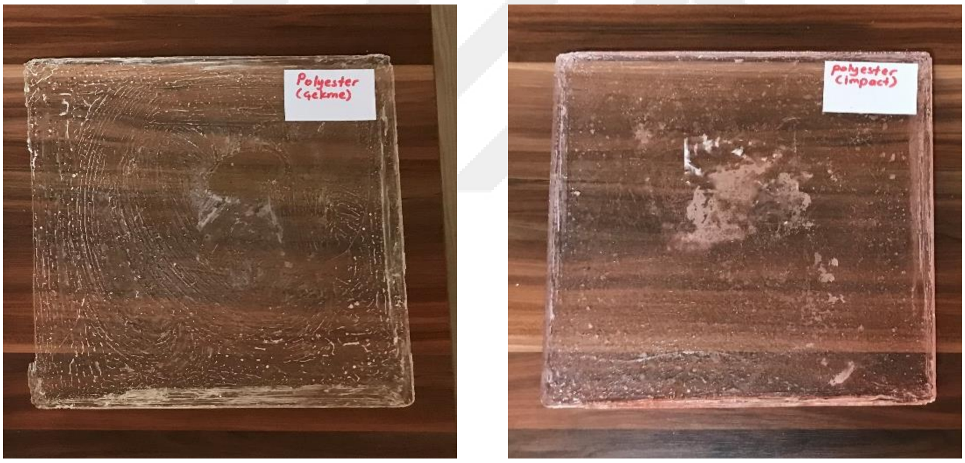
Figure 0.3. Production processes of samples

3.2.2. Characterization of Products

The production of the samples was formed according to the tests to be made. The samples were produced in accordance with the impact test and tensile test standards. For this reason, the samples were produced in two different thicknesses in molds without changing the resin-granule ratios. The state of the granular samples produced for impact test and tensile test before the cutting process is in Figure 3.4, the state of the ungranulated samples before the cutting process is seen in Figure 3.5 and Figure 3.6.

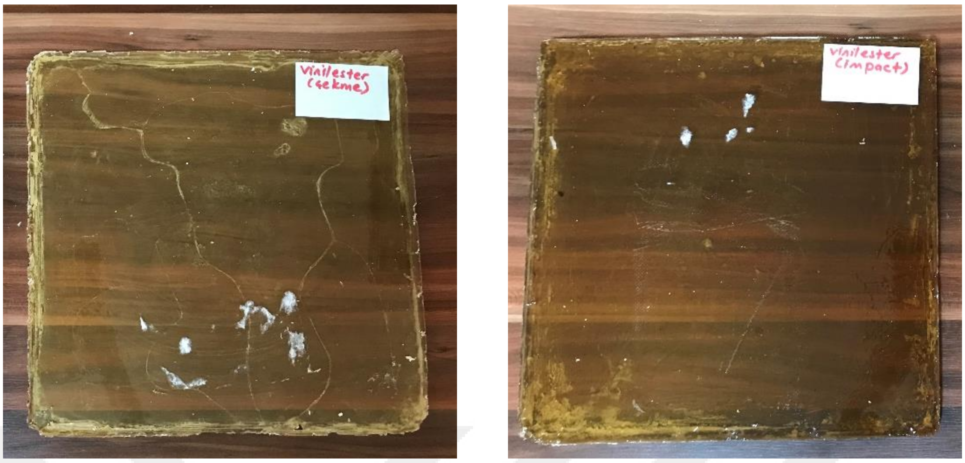
(a) (b)

Figure 0.4. The state of the granular samples produced for (a) tensile test and (b) impact test before the cutting process



(a) (b)

Figure 0.5. The state of the polyester (without granule) samples produced for (a) tensile test and (b) impact test before the cutting process



(a) (b)

Figure 0.6. The state of the vinyl ester (without granule) samples produced for (a) tensile test and (b) impact test before the cutting process

After the cutting process, the length, width and thickness values of tensile and impact test specimens were 250 mm x 25 mm x 2,5 mm and 125 mm x 12,5 mm x 10 mm, correspondingly, as shown in Figure 3.7. Tensile and impact test specimens were prepared according to the standards of tests.

(a) (b)

Figure 0.7. (a) Tensile (b) impact test samples

3.2.3. Experimental Tests

3.2.3.1. Tensile Test

Tensile test determines the elastic and plastic behaviors of the materials under static loading. The experimental procedure is conducted by applying an axial load to the specimen from both ends until the fracture of the material. The graph obtained from test (stress/strain) includes important parameters such as yield stress, modulus of elasticity, ultimate stress and fracture stress. It can also be observed from the fracture surface whether the material is ductile or brittle. Figure 3.8 demonstrated the testing procedure schematically.

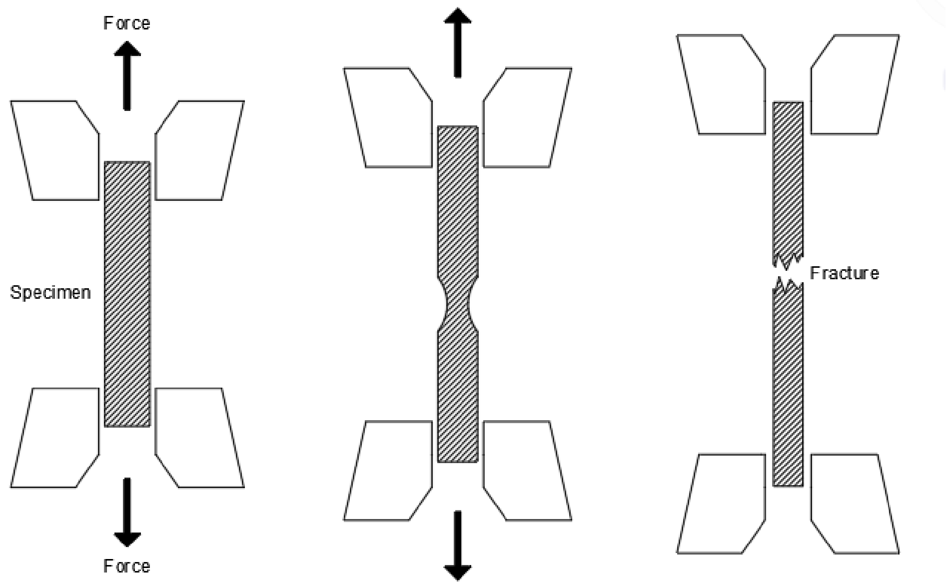


Figure 0.8. Tensile test procedure

Tensile tests were performed with ALSA universal tester (computer controlled, hydraulic, 60 tons load capacity, Figure 3.8). During the tests, the tensile speed was set to 2 mm/min. The samples were also prepared according to the relevant test standards.

Figure 0.9. Tensile test device

3.2.3.2. Charpy Impact Test

Impact test is performed in order to specify the impact resistance of material under dynamic impact loading conditions. The conservation of energy principle is utilized in testing. The test begins with raising a hammer (m kg) into a certain point (h_1) to gain potential energy. A specimen is positioned to be hit by a hammer. Then, hammer is released and strikes to the specimen. As waited, the hammer cannot reach to initial h_1 height after crash. It can only approach h_2 which is at lower level than h_1. The difference between the potential energy of hammer at level h_1 and potential energy of hammer at level h_2 can be evaluated as impact energy and it can easily been read from a calibrated scale. Figure 3.10 summarizes the impact test procedure.

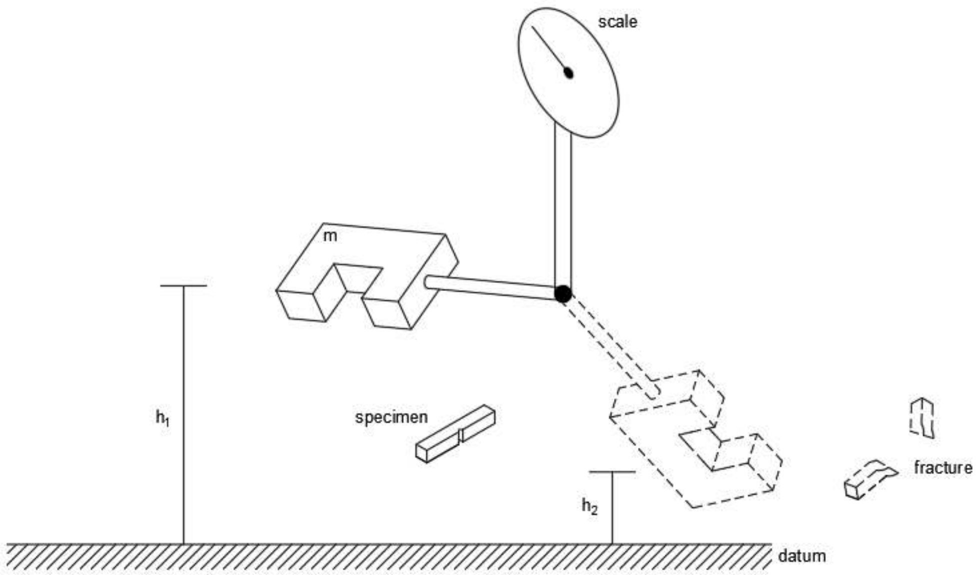


Figure 0.10. Impact test procedure

3.2.3.3. Hardness Test

Resistance of a material to plastic deformation can be described as the hardness of a material. A digital Vickers hardness tester (Jiashan - THV-1 MD) was used as the evaluation of hardness in this study. In the Vickers test method, an indentation was generated with a predefined load by a diamond indenter. Material is exposed to this load in a certain time. The indenter consists of a geometric configuration of a diamond square pyramid at an angle of 136° between opposite faces. Two diagonal indentation traces are left in the material surface after the removal of load as shown in Figure 3.10. Finally, Vickers hardness can be calculated with the following formulas.

$$HV = 1{,}854\frac{F}{d^2} \quad (3.1)$$

$$d = \frac{d_1+d_2}{2} \quad (3.2)$$

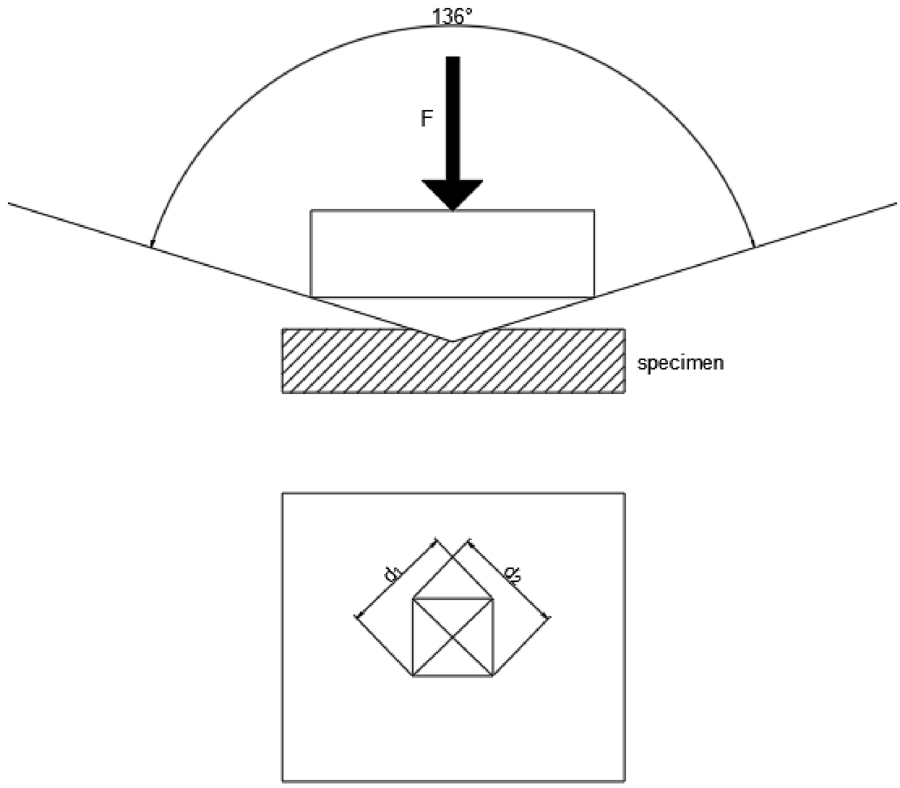
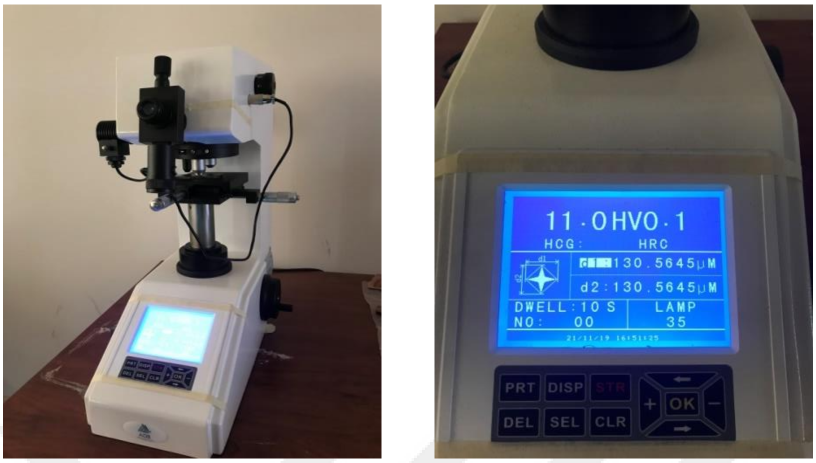


Figure 0.11. Vickers hardness test procedure

Figure 3.12 and Table 3.5 demonstrated the hardness test device and device specifications, respectively.



(a) (b)

Figure 0.12. (a) Vickers hardness tester (b) interface of tester

Table 0.5. Vickers hardness tester specifications

Brand / Model	Jiashan / THV-1 MD
Test Forces	0,01 kgf - 0,025 kgf - 0,05 kgf - 0,1 kgf, 0,2 kgf - 0,3 kgf - 0,5 kgf -1 kgf
Hardness Range	8HV - 2900HV
Microscope	100X for observation 400X for measurement
Dwell Time	0 - 60 s

3.2.3.4. Morphological Analyses (SEM Analyses)

Scanning electron microscopy (SEM) method is used to analyze the fracture structure of materials and in this way, regions of fracture in composites and reinforcement-resin interactions are examined. Produced samples were observed at 100V-30kV acceleration voltage using Scanning Electron Microscope (SEM) FEI Quanta 650 Field Emission device. The non-conductive surfaces in the composites were coated with gold with the help of the Q150R ES spray coating device before being subjected to SEM. This detailed analysis was carried out in Çukurova University Central Laboratory.

Figure 0.13. SEM anlaysis device

4. RESULTS AND DISCUSSIONS

4.1. Results of Tensile Test

The yield strength, tensile strength, elongation and tensile modulus of the samples were examined with the tensile tests performed on the samples. The obtained tensile test values are given in Table 4.1.

Table 0.1. Tensile test results

Specimen	Yield Strength (MPa)	Tensile Strength (MPa)	Elongation (%)	Tensile Modulus (MPa)
V	59.86	73	5.14	3120
P	51.46	62	1.95	3335
P-WTG5	45.20	56.51	2.15	3210
P-WTG10	43.96	53.62	2.28	3250
P-WTG15	41.82	51.96	2.35	3270

According to the test results, polyester was found to be more durable than vinyl ester. In addition, the results, when the change as a result of adding waste tire granules is observed; revealed that the addition of waste rubber to polyester resin reduces the tensile and yield strength of polyester resin, while the addition increases elongation. With the increase in the amount of waste rubber granules, the strength values decreased further. The tensile strength of polyester resin decreased by 8.85%, 13.51 and 16.19% for 100:5, 100:10 and 100:15 resin: waste rubber granule ratios, respectively.

The decrement of the strength may be due to poor interfacial interphase between the rubber granules and the polyester matrix (Adesina et al., 2020; Xu et al., 2020). The decrement of the strength values is a critical disadvantage for the application of this combination for a product. But also, it should be emphasized that using waste tire granule as filler material for composite material decreases the usage of polymeric resins which are mainly fossil sourced raw materials. The results show a decrement in strength values and this indicates that these combinations are not feasible for structural applications where good mechanical properties are required. In addition to this fact, this combination can be utilized in applications where low cost and environmental issues are the primary considerations.

4.2. Impact Tests

The values obtained as a result of the Impact Tests applied to the samples are shown in Table 4.1. According to the results of these tests, the highest hardness value was obtained from the P-WTG15 sample, while the lowest hardness value was observed in the P sample. When the effect of adding rubber granules to polyester resin on the hardness of the material is examined, it is striking that the addition of rubber granules increases the impact energy of the material. As can be seen in the table, the impact energy of the V sample was higher than the P sample, but with the rubber granules added to the polyester resin, the P-WTG15 sample had a higher impact strength than the V sample. The rubber granules added at a rate of 15% by weight increased the impact energy of the material by 21.47% compared to the reference sample.

Table 0.2 Impact test results

Specimen	Impact Energy(kJ/m^2)
V	12.21±0.52
P	11.41±0.72
P-WTG5	11.65±0.82
P-WTG10	12.21±0.25
P-WTG15	13.86±0.45

4.3. Results of Hardness Test

The values obtained as a result of the hardness test applied to the samples are shown in Figure 4.1. According to the results of these tests, the highest hardness value was obtained from the P sample, while the lowest hardness value was observed in the P-WTG15 sample. When the effect of adding rubber granules to polyester resin on the hardness of the material is examined, it is striking that the addition of rubber granules reduces the hardness of the material. As can be seen in the graph, the P sample is 15% harder than the P-GWT5 sample, 53% harder than the P-GWT10 sample, and 57.3% harder than the P-GWT15 sample, respectively. However, it is noteworthy that the P-WTG5 coded sample with 5% by weight rubber granules added is harder than the V coded sample produced only from vinyl ester. Finally, another important output of the test is that the P sample is 22% harder than the V sample.

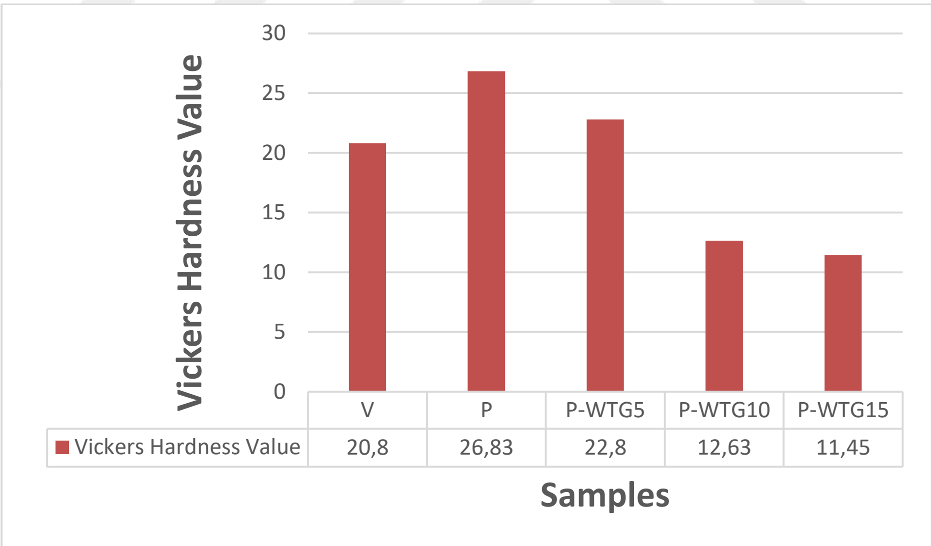
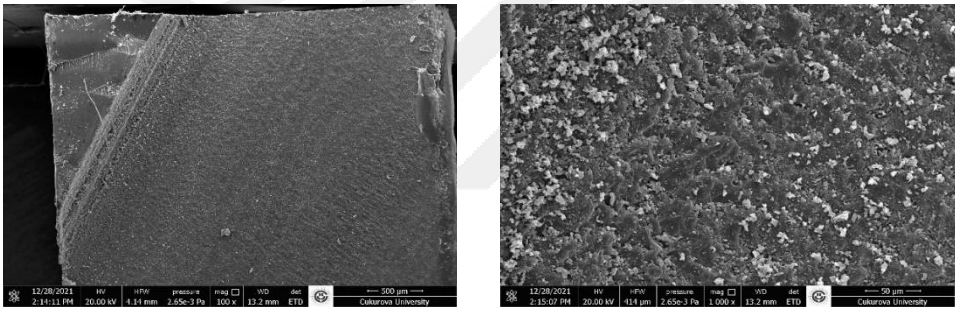


Figure 0.1. Hardness values of samples

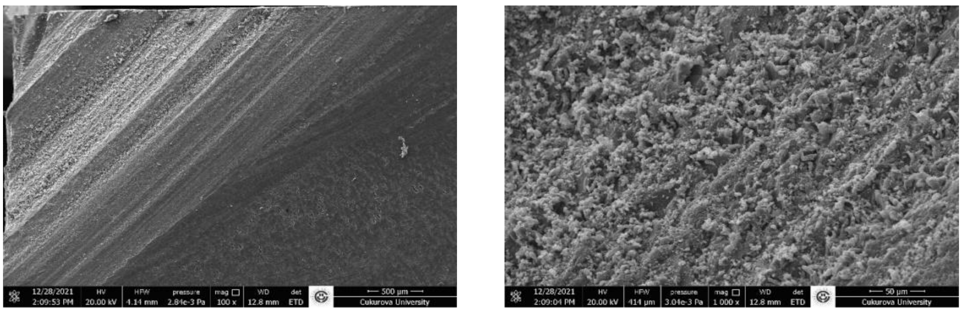
4.4. Results of Scanning Electron Microscope (SEM) Analyses

In order to observe the strength values of the samples, the fracture structures were examined after the tensile test was performed, and this examination was made by SEM analysis. Images obtained from SEM provide information about the microstructure, morphological properties, and fracture mechanism of the material. It can be seen in Figure 4.2 and Figure 4.3 that polyester and vinyl ester resins have a brittle structure and their breakage creates significantly sharp surfaces. Besides, in Figure 4.4, Figure 4.5, and Figure 4.6, it is observed that this sharp fracture structure decreases with the addition of rubber granules to polyester. These images explain the increase in the amount of elongation with the addition of rubber granules to the material.



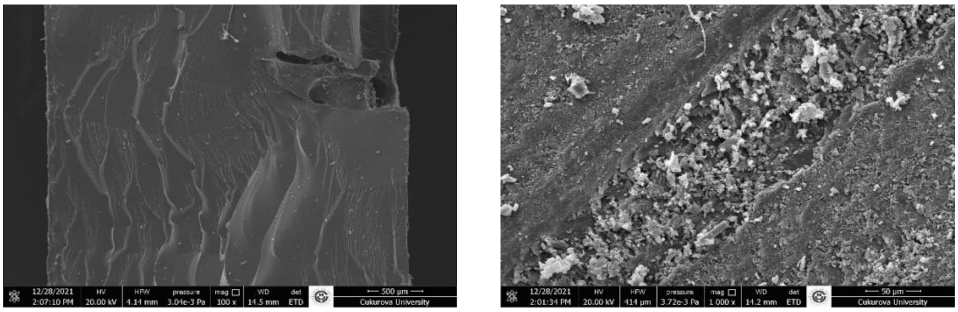
(a) (b)

Figure 0.2. SEM images of V (a) 100 X (b) 1000 X



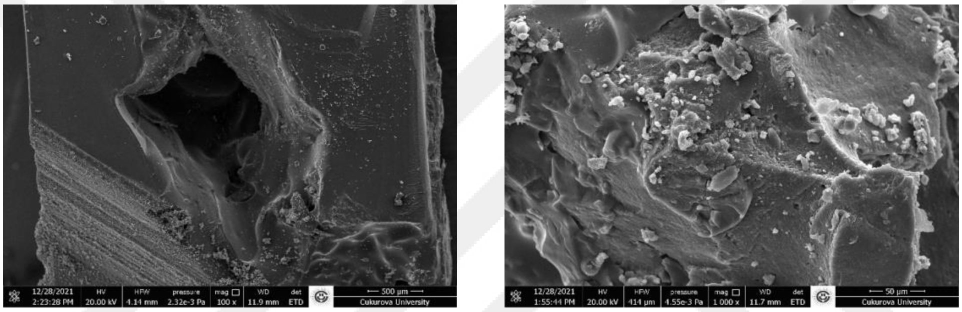
(a) (b)

Figure 0.3. SEM images of P (a) 100 X (b) 1000 X



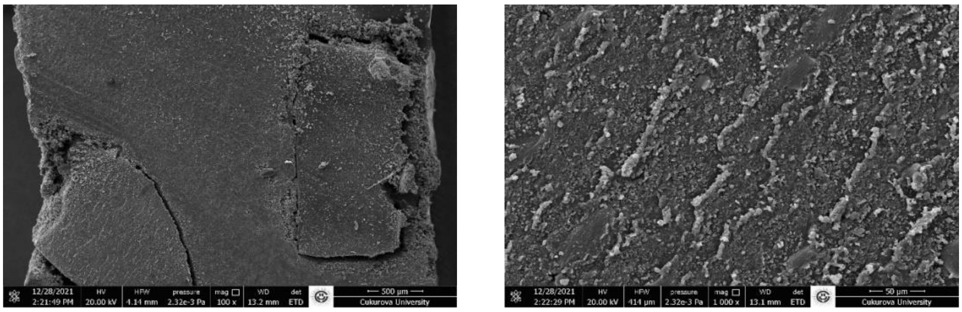
(a) (b)

Figure 0.4. SEM images of P-WTG5 (a) 100 X (b) 1000 X



(a) (b)

Figure 0.5. SEM images of P-WTG10 (a) 100 X (b) 1000 X



(a) (b)

Figure 0.6. SEM images of P-WTG15 (a) 100 X (b) 1000 X

5. CONCLUSIONS

In this study, waste rubber rubber granules were used as filling material to produce polymer matrix composite samples. Polyester resin was used as the matrix material and three different ratios of waste rubber rubber granules were used to produce the experimental samples. The samples were produced by open molding method. Tensile, impact and hardness tests were performed to determine the mechanical properties of the samples and SEM images were taken to examine the microstructures of the samples. The following statements were removed from the study;

- Since the addition of rubber granules to the polyester matrix reduces the strength of the structure, waste rubber rubber granules can be used as filling material, especially in non-structural applications.
- The addition of waste rubber granules to the structure weakens the structure, but also increases the ductility of the material.
- The combination of untreated waste tire rubber granules and polyester matrix creates poor interfacial adhesion that must be improved by proper treatment.
- In the research, it was observed that the waste tire granules increase the impact energy. For this reason, its use in absorbing its energy in collisions should be evaluated.
- The use of waste tire granules for composite material production is a promising alternative solution for sustainable and environmentally friendly production by recycling fossil-sourced raw materials and reducing the amount of use.

The use of waste tire granules in composite materials is important for recycling polymer wastes. It is necessary to pave the way for the use of these wastes in composite structures. It is obvious that a great deal of recycling will be achieved with more composite structures produced from these wastes.

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