JANUARY 2017 M.Sc. in Mechanical Engineering KHAMIS MUSDIF THEIR ALBU-KHALEEFAH


UNIVERSITY OF GAZİANTEP

GRADUATE SCHOOL OF

NATURAL & APPLIED SCIENCES


# A RESEARCH ON EFFECT OF PISTACHIO SHELL ON THE MECHANICAL PROPERTIES OF COMPOSITE MATERIAL

M.Sc. THESIS

IN

MECHANICAL ENGINEERING


BY

KHAMIS MUSDIF THEIR ALBU-KHALEEFAH


JANUARY 2017

# A research on Effect of Pistachio Shell on The Mechanical Properties of Composite Material

**M.Sc. Thesis**

**in**

**Mechanical Engineering**

**University of Gaziantep**

**Supervisor**

**Assoc. Prof. Dr. Ahmet ERKLİĞ**

**by**

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**January 2017**

REPUBLIC OF TURKEY
UNIVERSITY OF GAZİANTEP
GRADUATE SCHOOL OF NATURAL & APPLIED SCIENCES
MECHANICAL ENGINEERING

Name of the thesis: A research on Effect of Pistachio Shell on The Mechanical Properties of Composite Material

Name of the student: Khamis Musdif Their ALBU-KHALEEFAH

Exam date: 26.01.2017

Approval of the Graduate School of Natural and Applied Sciences.

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**Khamis Musdif Their ALBU-KHALEEFAH**

## ABSTRACT

**A RESEARCH ON EFFECT OF PISTACHIO SHELL ON THE MECHANICAL PROPERTIES OF COMPOSITE MATERIAL**

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**M.Sc. in Mechanical Engineering**

**Supervisor: Assoc. Prof. Dr. Ahmet ERKLİĞ**

**January 2017, 65 pages**

In the current study, the effect of micro-scale content of natural particle (pistachio shell) on the mechanical properties of polyester matrix composites is experimentally investigated. The particle contents of pistachio shell are 0, 5, 10, 15, 20 and 25 wt%. The tensile, flexural, and charpy impact tests were carried out on the moulded composite specimens according to ASTM and ISO standards. Good dispersion of pistachio shell particles in polymer matrix was observed using SEM micrographs. It is observed that the mechanical properties of polyester composite is increased with the increasing of pistachio shell particle content. The highest tensile strength, flexural strength, and impact strength was obtained at pistachio shell particle content of 10, 5, and 5 wt%, respectively.

**Keywords:** Pistachio shell, polymer composites, mechanical properties.

# ÖZET

**Fıstık Kabuğu Katkısının Kompozit Malzeme Mekanik Özelliğine Etkisi Üzerine Araştırma**

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**Ocak 2017, 65 sayfa**

Bu çalışmada, doğal parçacık olan mikro ölçekteki fıstık kabuğunun polyester kompozitlerin mekanik özelliklerine etkisi deneysel olarak araştırılmıştır. Antepfıstığı kabuğu parçacıkları ağırlıkça % 0, 5, 10, 15, 20 ve 25 kullanılmıştır. ASTM ve ISO standartlarına göre oluşturulan kalıplar ile kompozit numuneler üretilmiştir. Üretilen numunelere gerilme, eğilme ve çentik darbe testleri yapılmıştır. Antepfıstığı kabuğu parçacıklarının polimer matris içindeki dağılımı SEM fotoğrafları kullanılarak gözlenmiştir. Antepfıstığı kabuğu parçacık içeriğinin ağırlıkça artmasıyla polyester kompozitin mekanik özellikleride arttırılmıştır. En yüksek gerilme mukavemeti, bükülme mukavemeti ve darbe mukavemeti sırasıyla, fıstık kabuğu tanecik içeriği ağırlıkça % 10, 5 ve 5'de elde edildi.



**Anahtar Kelimeler:** Antepfıstığı kabuğu, polimer kompozitler, mekanik özellikler

*This thesis is dedicated to my family, sons and daughters for their endless love, support and encouragement*

# ACKNOWLEDGEMENT

First and foremost, I would like to thank the GOD for his guidance and rewards toward me and my family, then, I'd like to express my deepest respect and most sincere gratitude to my supervisor, Assoc. Prof. Dr. Ahmet ERKLIG, for his guidance and encouragement at all stages of this mutual work. I will always be grateful for the valuable advice and insight supervision and encouragement to me during this research.

I would like also to express my gratefulness to my best near friend, a Ph.D. student Mr. Muhamad ALSAADI for his valuable advices, encouragement during my academic life

Finally, I would like to express my deepest regards and respects to a M.SC students Mr. Majeed AL-FAHDAWI and Mr. Ahmed AL-FAHDAWI for their encouragement and support throughout my thesis study.

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# CHAPTER 1

# INTRODUCTION

## 1.1 Outline of the Study

This thesis is organized into six chapters. In the first chapter of the thesis, a small background about composite materials will be given and it will be the introduction of the thesis. A literature review is given in chapter two. Chapter three will be concern about the definition of composites and their advantages and disadvantages, application areas of composites, classification of composites, manufacturing techniques of composite materials, and types of tests those are applied to composite materials. Each of these will be also detailed. In chapter four, discussed parameters will be a polyester resin, hardener (Methyl Ethyl Ketone Peroxide-MEKP), pistachio shell powder, stirring of materials, mechanical tests (tensile test, three-point bending test, impact test). Chapter five contains the experimental results and discussions. Chapter six contains conclusion.

## 1.2 Background

Technological progress depends on development in understanding and application of materials. The design of an advanced aerospace engine or superconducting device is of no use if required materials to bear the service load and environment isn't available. The field may be the ultimate limitation on enhancement depends on material development. Composite materials during this regard represent a large leap that mankind has created towards the improvement of materials.

The word "Composite" means that "consisting of two or more different parts". Therefore, composite mentions two or additional completely different constituent materials that are mechanically or metallurgically bonded together separated by a distinct interface.

Composites are created of individual materials known as constituent materials. The reinforcing phase material i.e. reinforcement may be in the form of fibers, particles, and flakes. At least one portion of every kind is required.

Once the reinforcement of polymer composites is in the kind of particles, with all their dimensions roughly equal i.e. the composite behaves basically as an anisotropic material that properties are freelance of direction. Once the dimension of the reinforcement particles is unequal, the composite could behave as an isotropic material on condition that the particles are at random oriented, like within the at random oriented tiny fiber bolstered composites. In different cases, the producing method (e.g., molding of a brief fiber composite) could induce orientation during a most popular direction and thus induce some anisotropy. In continuous-fiber-reinforced composites, like unidirectional or cross-ply composites, anisotropy could also be fascinating. Moreover, the first advantage of the composites is the capability to regulate anisotropy by plan and manufacture counting on need. The concentration circulation of the particles denotes to their spatial associations to every different. Particles could by uniformly spread in an every composite and placed at regular spacing in order that no two particles trace one another. On the opposite hand, it's additionally potential to imagine a dispersion of particles, therefore, organized that they form a network such an internal path connects all particles [1].

Environmental awareness in these days makes the researchers universal on the educations of natural fiber reinforced polymer composite and price effective choice to synthetically fiber reinforced composites. The availability of natural fibers and simple manufacturing have tempted researchers to do domestically obtainable cheap fibers and to review their feasibility of reinforcement purposes, and also to whatever ambit they achieve the desired specifications of well-reinforced polymer composite for various applications. With low cost and high specific mechanical properties, natural fiber represents a decent reconditions and biodegradable various to the foremost common artificial reinforcement, i.e. glass fiber.

The word “natural fiber” covers a wide vary of vegetable, animal, and inorganic fibers. However, in the composite production, it's sometimes remarked wood fiber and agro-based bast, seed, leaf, and stem fibers. In spite of the attention and ecological demand of natural fibers, they are restricted to non-bearing applications because of their lower strength compared with artificial fiber armored polymer composite.

The natural fibers as Jute, Sisal, bagasse, and Kenaf have several advantages over glass fiber or carbon fiber as renewable, environmentally friendly, little-cost, light-weight, and superior specific mechanical performance. High-performance synthetic filler materials like thermoplastic polymers have been employed over the previously limited periods [2] to serve as filler materials in epoxy resin composites and they were exhibited larger thermal and toughness stability over the years. However, the past little years have seen a development in research efforts towards finding environment-friendly solutions that might lead to the production of additional natural filler materials. Natural filler materials can serve as effective alternatives to artificial filler materials for purposes of reinforcement of polymeric composites. They can be treated naturally to acquire strength and rigidity properties the same as their artificial counterparts.

A number of the disadvantages of natural filler materials is wet sensitivity which reduces effectiveness per hydrophobic polymers, biological decay, non-uniform filler forms and sizes, vulnerability to natural ecological attacks, and lack of hardiness below higher temperatures. However, natural filler materials can be post-processed to decrease the amount of these disadvantages, namely, degradation under wet and alternative environment effects [3-4].

Literature survey reveals varied makes an attempt created to develop polyester composites changed with varied fillers, so as to develop the presentation of this matrix.

Recently conductive polymer composites obtained by filling polymer matrixes with varied pistachio shell powder were additionally reported.

These fillers are value-added to polymers to attain fascinating and to develop the merchandise service abilities. It is, therefore, essential to developing a viable different supply of fillers from reconditions resources like agrarian leftover and bamboo stem that are in nature and wealthy in organic materials. The pistachio shell (PS) particles have extraordinary strength and modulus properties along with the else advantage of high polymer content. The high polymer content makes the composites created with this filler additional weather resistant and thus many appropriate for application as construction materials.

### 1.3 Importance of the Study

Pistachio shells (PS), among the agricultural residues, are one of the high waste quantities in Turkey. Tons of pistachio shells are either burned after harvest or left on the field every year. As presented in Table 1.1, according to statistics of Food and Agricultural Organization of United Nations in 2012, Turkey is one of the largest pistachio producers in the world [5]. Due to specific weather conditions and landing requirements, cultivation trees of pistachio are widespread in the southern east region of Turkey, such as Gaziantep, Şanliurfa, and Siirt.

**Table 1.1** Pistachio production in the world [5].

| Country Production | (tons) |
|---|---|
| **Iran** | 446,647 |
| **USA** | 213,000 |
| **Turkey** | 128,000 |
| **Syria** | 57,300 |
| **China** | 48,700 |
| **Greece** | 9,000 |
| **Afghanistan** | 3,000 |
| **Tunis** | 2,600 |
| **Italy** | 1,600 |
| **Kyrgyzstan** | 800 |
| **Others** | 1,532 |
| **Total** | 912,179 |

However, particular attention has not been paid as an agricultural residue that can be used as raw materials in polymer composites industries. Therefore, the usage of pistachio shells as filler in polymer composites is very important from the economic and environmental viewpoint. Many useful usage of pistachio shell can be applied such as medium for orchids, fire starter, animal feed, additive for wood items, etc. [6]. Moreover, Pistachio shell particles have high strength and modulus properties. Fabrication of polymer composites with this filler is more suitable for many engineering applications. Unsaturated polyester is widely used in industry like the car industry, aeronautics, or building materials industry due to easily of fabricating and having good mechanical properties, in addition, its low cost compared with epoxy and other polymers. A variety of applications has been extensively used polyester resins as a polymer matrix for polymer composites, ranging from water tanks and automobile to building materials [7, 8].

To the best of our knowledge, in literature, several researchers have studied the mechanical properties of polymer composites filled with synthetical and agricultural remains but the study of flexural and impact of PS filled polyester composites is not considered yet.

In the current research, the opportunity of using pistachio shell for industry of polymer composites was considered. PS particles were used as a source of waste filler material for polyester resins in order to evaluate tensile, flexural and Charpy impact properties. The current study also includes preparation of composite specimens with various filler contents of 5, 10, 15, 20 and 25 wt% using hand lay-up method. The micro-PS particles distribution within the polyester matrix were observed using SEM.

# CHAPTER 2

# LITERATURE REVIEW

A brief literature review related to composite, effects of different fillers, tensile, flexural, impact strength of the composite material is presented in this chapter. The literature review has been grouped in four sections: studies on organic particle filled composites are reviewed in section 2.1. Studies on inorganic Particle Filled Composites are given in section 2.2. Studies on General Particle Filled Composites are given in section 2.3. Conclusion on literature review is given in section 2.4.

## 2.1 Studies on organic Particle Filled Composites

Zabihzadeh et al. [9] investigated the effects of filler loading on the flexural and physical properties of the natural-filled thermoplastic polymer composite panels. Test results showed that the elasticity modulus and flexural strength were basically affected by the filler content, and the flexural strength was appeared to decrease with an increase in filler content, while the flexural modulus rising in consideration.

Monteiro et al. [10] studied to produce a composite material of little price composites by using the sugar cane bagasse waste as reinforcement for polymeric resins. It was observed that composites with homogeneous microstructures could be manufactured. In another side, it can be achieved mechanical properties similar to wooden agglomerates.

Jiménez et al. [11] illustrated a composite material by unsaturated polyester resin and olive brush seed (OBS). OBS was cured with sodium hydroxide and maleic anhydride after that it was utilized in a percentage of 35 wt% to produce a composite material.

Abessalam [12] investigated the powdered olive pits as a novel filler material/epoxy resin. The influence of the untreated and treated powder loading (weight fraction) on the void content and the mechanical properties of the composites were checked. The results appeared important improvements in mechanical properties for composites reinforced with treated olive pits than composites reinforced with untreated olive pits.

Koutsomitopoulou et al**.** [13] studied the effect of olive pits powder reinforced polylactic acid (PLA)-matrix. This study is focused on recycling potential of leftover materials, for example, olive pits, i.e. the hard phase derived from an olive oil mill, mixed with thermoplastic polymers which were utilized for the production of new materials applied in industrialized containers and formworks. Consequently, the powder was submitted in a bio-based and biodegradable matrix (PLA) at many percentages.

Venkatesh [14] studied fabrication and testing of coconut shell powder (CSP) reinforced epoxy composites. The density of the composite gradually decreases with the increase of weight percentage in fiber content. The rigidity rate of the composite increases with increasing the fiber content. Maximum CSP reinforced composite is 28.87 MPa. It was upper when compared to another natural fiber composites produced from acacia, kenaf, coir, water hyacinth banana, EFB, ramie, curaua, and jute. But it has less strength compared to the polymer, sisal, and bamboo as a result of its fiber strength which exhibits naturally.

Alaa [15] studied investigation of tensile and impact of composite materials reinforced with natural materials. The value of percentage elongation, impact strength and fracture toughness decreases with increasing weight fraction and grain size of the powder. Mathematical model results display that the weight fraction of powder has a greater effect than grain size on properties.

Oladele [16] studied the particles of bagasse fiber were sieved into 75 μm and added to the polyester in vary weight fraction ratios of 5%, 10%, 15% and 20% in order to manufacture the particulate polymer composites. It was observed that the reinforcement was able to enhance the mechanical properties (tensile and hardness) of the industrialized composites with an edge point rate of 10 wt% reinforcement. There is an improvement in the mechanical properties of the reinforced polyester composites up to particle content of 10 wt% bagasse fiber loading. Bagasse particulate fiber in the range of 5-10 wt% resulted the highest values which appear that little fiber weight content is good for better augmentation of properties. The curative rate for the polyester material is developed by the adding of bagasse fiber so leading to increased production rate.

Oladele et al. [17] studied the effect of corn cob particulate on the mechanical and biodegradability properties of reinforced polyester composites. The use of corn cob particulate (treated and untreated) phases, may enhance the mechanical properties of unsaturated polyester. Likewise, their usage in both fine are; 150 μm particle size and coarse; 300 μm particle size form. The flexural properties were greatly enhanced by 150 μm particle sizes while both particles sizes used improved the tensile properties. The flexural properties are very susceptible to particle sizes greater than the tensile properties. The curing time results showed that the presence of a tiny amount of the corn cob in treated condition will aid fast curing and hence, a high proportion of production. This was established by the performance of the composites developed with 2 wt% from treated corn cob of 150 μm and 300 μm particle sizes. The biodegradability results showed that coarse particle sizes enhanced the biodegradability affinity of the developed composites more than the fine particles and the addition of a large amount of the corn cobs in both treated and untreated conditions but with the treated conditions more promising. Little weight fraction offered the best enhancement for the developed composites in most of the mechanical properties examined while great weight fraction improved biodegradation affinity.

Ameh et al. [18] studied the particle size and loading of date palm seed particulate/polyester. The least particles size (0.5mm) provided the optimum tensile strength and elastic modulus with 15 wt% and 10 wt% loading respectively. The elongation of the composite specimen decreases with increase of particle loading and impact strength increase with smaller particle sizes. The hardness of the composite increases as particles loading increase while the maximum hardness is obtained with particle loading 25 wt% and particle size of 2mm and flexural strength decrease as particles loading increase. Lower particle sizes have inferior percent water absorption and the density of the composite is concluded to be intercepted on the density graph.

Mohammed [19] studied some mechanical properties of unsaturated polyester filled with the seed shells of sunflower and water-melon. The results appeared that the flexural strength, elasticity modulus, compressive strength and rigidity improved with the rising of the shells powder percent. Whereas the impact strength was reduced with increasing of powder content.

Ahmed [20] studied on properties of unsaturated polyester resin reinforced with rice husk. The results showed that the composite materials of rice husk gained superior mechanical properties compared with the composite intended from unsaturated polyester resin without filler.

Yang et al. [21] studied the effect of rice-husk flour on some the physical, mechanical and morphological properties of polypropylene composite materials. The results showed that the tensile strengths of the composites somewhat reduced as the filler loading increased. The modulus of tensile was enhanced with rising filler loading. The addition of filler causes dropped in the strength of the notched and unnotched Izod impact.

Sumangala et al. [22] studied characterization of particulate epoxy composites for mechanical behavior. Experimental results on these composites show that the specific modulus of these lightweight composite is higher. The modulus of composite flexural can be efficiently tailored by changing the weight fraction. The flexural strength is mainly affected by the resin content of the composite. It is obtained that the flexural strength decreases when the weight fraction rises. The flexural strength of the composite is achieved to be upper with 40% weight fraction of walnut shell powder.

Raghad et al. [23] studied the influence of addition (beans shell powder)/ polyester composites. The result acquired was compared with the in forced polyester plate at 10% filler loading, it is found a 79% enhancement in tensile properties and 66% increase in impact strength.

Shehu et al. [24] studied the effect of palm kernel shell (PKS) with particle sizes of (75μm, 150μm, and 300μm) on the mechanical and physical properties of polyester/ PKS composites. The particle loadings were 10, 20, 30 and 40 wt% of PKS particles. It was shown that the densities of the composites increased for all the sieve sizes under consideration with an increase in weight percent of PKS particles. The composites produced with the 300μm sieve size PKS showed the best tensile strength values as compared to those of other sieve sizes. The impact energy values for all the sieve sizes were better than that of the unreinforced polymer.

Ibrahim et al. [25] studied the mechanical and thermal properties of composites from unsaturated polyester filled with oil palm ash. The results showed that the tensile and flexural modulus of UP/OPA composites were enhanced with the particle content.

Adeosun et al. [26] studied the thermo-mechanical properties of unsaturated polyester reinforced with coconut and snail shells. The results appeared that the coconut shell enhance the tensile strength of the polyester composite. The coconut shell reinforced polyester shows less thermal stability than the snail shell reinforced polyester.

Sahari et al. [27] studied the mechanical properties of oil palm shell (OPS) composites with different OPS volume proportions of (0%, 10%, 20%, 30%) by unsaturated polyester (UPE) as a matrix. It was observed that the tensile strength and tensile modulus of the UPE/OPS composites was increased as the OPS loading increased.

Romisuhani et al. [28] investigated the action of acrylic acid (AA) as a connected agent on low-density polyethylene (LDPE)/palm kernel shell (PKS) bio-composites on tensile properties and morphology. The spread of palm kernel was improved by (AA). (LDPE) was filled by (PKS) at different ratio of weight fraction (0%, 10%, 20%, 30%, 40%). The results appeared that the tensile strength and break elongation were decreased with increasing filler content.

Ebrahim et al. [29] studied the influence of nano-clay particles and pistachio shell flour on the mechanical properties of wood-plastic composites. The results revealed that using 5% Nano clay increased bending strength and bending and tensile modules compared to 1 and 3% Nano clay treatment. Additionally, results proved that using 1 and 3% nano clay treatment increased the tensile strength and the impact resistance compared to the 5% treatment. They also show that using 50% pistachio shell flour (PSF) treatment increases bending strength along with tensile and bending modules compared to 25 and 35% treatments in wood-plastic nanocomposites (WPNC). Results finally proved that usage of 25% and 35% (PSF) increases the sheared impact resistance compared to 50% treatment in (WPNC).

Baştürk et al. [30] investigated the waste fillers of acorn and pine cone filled polymer composites with three changed proportions (10%, 20%, and 30%) and manufactured with casting method. It was observed that the pine cone based composites showed slightly better results compared to the acorn reinforced samples. The composites independent of filler type appeared higher tensile and rigidity properties with increasing the particle content.

Singh et al. [31] studied the mechanical properties and absorption behavior of coconut shell powder-epoxy composites. The results showed that the extremity tensile strength is acquired for the composite prepared with volume fraction of 20% coconut shell powder (CSP) volume fraction. An increase of filler volume the tensile strength goes on decreasing. Thus, the proportion of decrease in tensile strength between (20%- 30%), while (30% - 40%) CSP filled epoxy composite is approximately constant. A composite prepared with 30% CSP filled while; the flexural strength is the minimum for the composite intended with 40% CSP filled. The increase of filler volume flexural strength increases between (20% - 30%), while, the flexural strength decreases on increasing filler volume from (30% - 40%). Thus, the proportion of decrease in flexural strength from (30% - 40%) is bigger than the proportion of increase between (20% - 30%) CSP filled composite.

### 2.2 Studies on inorganic Particle Filled Composites

Güngör [32] studied the mechanical properties of high-density polyethylene (HDPE) consists of Fe polymer composites. As compared to the mechanical properties of unfilled HDPE, Fe-filled polymer composites appeared lower yield and tensile strength, elongation, and Izod impact strength, whereas the rigidity and the elasticity modulus of the composites, were upper than those of HDPE. It was observed that Fe addition with volume fraction of 5% increased the elasticity modulus of HDPE about 31 % otherwise it decreased elongation and Izod impact strength of HDPE about 90 % and 40 %, respectively.

Zhang et al. [33] studied the influence of the size and content for the filler ($Al_2O_3$)/high-density polyethylene composites. Thermal accessibility and tensile strength of the composites rise with the decrease of the particle size. The dependence of impact strength on the particle size is more complex. The SEM micrographs of the crack surface found that $Al_2O_3$ with small particle size is usually more efficient for the augmentation of the impact strength, whereas the 100 µm particles prone to aggregation due to their high surface energy deteriorate the impact strength. Composites filled with $Al_2O_3$ of 0.5 µm at the content of 25% volume show the best synthetic properties.

Setsuda et al. [34] studied the effects of fly ash in composites fabricated by injection molding. The results reveal that the content of fly ash is extremely important and instrumental to the shrinkage ratio and bending strength. The shrinkage ratio, bending strength and a flexural modulus of LLDPE composites consist of raw fly ash were appeared to increase. The shrinkage ratio and flexural modulus of PP composites including ground fly ash were also appeared to increase.

Oladele [35] studied the development of bone ash and bone particulate reinforced polyester composites for biomedical applications. It was observed that cow bone may be used as reinforcement in polyester to improve composites for biomedical applications having assembled structural conditions necessary. Bone ash and bone particle reinforced tensile and flexural composite samples were developed from pre-determined proportions of 2, 4, 6, and 8 %. The results showed that at 8% offered the best results in both: bone ash and bone particulate reinforced composite testers.

Aruniit et al. [36] studied the influence of filler content on the mechanical and physical properties of the particulate composite**.** Test slabs were fabricated with various filler contents using vacuum supported extruder. It is shown that with a higher percentage of alumina trihydrate, the flexural strength decreases but the rigidity and flexural modulus increases together with density.

Oladele et al. [37] studied the usage of cow bone-reinforced polyester composites with changed size of particles (75, 106 and 300 μm). Both tensile and flexural strength were highly improved by 8 wt% for 75 μm whereas toughness was highly improved by 6 and 8 wt% for 300 μm. It means that small particles lead to improve strength whereas large particles lead to improve toughness.

Oladele [38] studied the physical and mechanical properties of steel-making slag/polyester composites. The results appeared that the composites produced have certainly acquired increased in the properties compared to the unfilled polyester material. The ideal results were achieved from the use slag particles with particle size of 106 μm and weight fraction of 2%.

Aruniit et al. [39] studied the effect of hollow glass microspheres on the mechanical and physical properties and cost of particle reinforced polymer composites. The results indicated that the composite material filled with 6 wt% hollow glass microspheres appeared 3% reduce in the tensile strength and 26% reduce in the surface rigidity compared to the composition without the filler. The weight reduced by 13% compared with the initial composition. The net value of the composite material was increased 7% as a result of adding of hollow glass microspheres.

Srivastava et al. [40] studied the mechanical properties of epoxy resin stuffed with fly ash particles. It has been reported that the fracture surface energies of epoxy and polyester resin and their resistance to crack are relatively low.

Selvin et al. [41] studied the mechanical properties of titanium dioxide-filled polystyrene micro composites. If the particulate filler is added to these resins (epoxy and polyester), the particles prevent crack expansion. As the volume fraction of filler is varied, the fracture energy increases up to critical volume fraction and then decrease again.

Osman et al. [42] studied the effect of overmuch filler coating on the tensile properties of little density polypropylene (LDPE)/calcium carbonate composites. It was observed that by merging filler particles into the matrix of fiber reinforced composites, mutual effects may be acquired in the form of higher modulus and decrease material costs, yet accompanied with decreased strength and impact toughness.

Hassan et al. [43] studied development of polyester/eggshell particulate composites. The results appeared that bending and tensile strengths of the composite increase with augmentation weight fraction of the eggshell particles. Rigidity values achieved when reinforcing polyester with eggshell as particles increase the weight of the particles increased in the polyester matrix. The best reinforcement influence on carbonized eggshell can be attributed to the best interfacial bond between carbonized particles and polyester matrix as revealed from the SEM studies.

Asuke et al. [44] studied effects of the bone particle (BP) on the properties and microstructure of polypropylene (PP)/bone ash particulate composites. It was noticed that successful fabrication of PP and BP composite by compounding and compression molding. The hardness values obtained from PP reinforced with BP increased with an increase in the weight fraction of BP. The developed composites have better properties at the ranges of weight fraction (5%–15%) BP additions, BP addition should not be exceeding 15 % in order to have better properties.

### 2.3 Studies on General Particle Filled Composites

Dhawan et al. [45] studied the influence of natural fillers such as (coconut coir, rice husk, and wheat husk) on mechanical properties of glass fiber reinforced plastics (GFRP) composites. Epoxy and polyester resins have been used to study the interaction of matrix on the properties of GFRP. It noticed that the natural fillers offered good results in polyester-based composites. The addition of coconut coir appears superior results in polyester and epoxy composites. Coconut coir fillers should be utilized instead of wheat husk and rice husk in general to enhance the properties of the developed glass fiber reinforced composite laminates.

Ramesh et al. [46] studied the enhancement of tensile and flexural strength with wood dust and glass fiber filled epoxy hybrid composites**.** The prepared composite was tested to study the mechanical properties for example, flexural and tensile strength of wood dust particles filled glass fiber/epoxy composites. The results indicated that merger of glass fiber in pine wood dust filled epoxy resin enhanced strength both in tensile and flexural modes of unfilled epoxy.

Pazarlıoğlu et al. [47] studied the mechanical properties of biaxial glass fiber and pistachio shell reinforced polyester composites. All tensile and impact test results showed that the mechanical properties of biaxial glass non-crimp fabric reinforced composite laminates with 0° directions are higher than 90° directions. Tensile and impact strengths increase with decreasing of particle size for pistachio but decrease with increasing of pistachio shell rate.

### 2.4 Conclusion on literature review

As a conclusion on the summarized literature, the effect of fillers on the composite material can be clearly seen. Weight fraction or volume fraction of additive materials affect almost all properties of the composite material. Type of additives may affect with different weight fraction/volume fraction of pistachio shell (additive) or different filler type. Also, the mathematical model results show that the weight fraction of particle has a higher effect than grain size on the physical and mechanical properties of composite materials**.**

The success of combining natural particulate fillers with polymer matrices results in the improvement of mechanical properties of the composites compared with the matrix materials. These fillers can be acquired from renewable sources and easily recyclable. Despite their little strength, they can produce composites with superior specific strengths in addition to cost reduction of the composites production.

# CHAPTER 3

# COMPOSITE MATERIALS

## 3.1 Definition of Composites

Composite materials are defined as materials containing of two or more physically distinct phases, appropriately organized or distributed. The continual section is adverted to as the matrix, whereas the distributed section is named the fillers. Three things verify the specifications of a composite: a form of matrix, type of filler and the intermediary between them [48, 49]. Since the previous twenty years, natural plant fibers are receiving significant attention because of the replacement for artificial fiber reinforcement like glass in plastics [50, 51]. There are economic and ecological reasons for substitution a fraction of the plastics wood, however, the wood might additionally task as reinforced of the plastics. The elastic modulus of wood fibers is about forty times upper than that of polyethylene and the strength nearly twenty times upper [52]. The fascinating within the usage of wood as filler or reinforced in thermoplastics is as a result of several benefits, like little density, great stiffness, strength, and small cost [53-56].

The ecological sensibility of people nowadays is paying the manufacturer to choose natural materials as alternatives for non-renewable materials. Wood has been utilized as a constructing and engineering material meanwhile early times and displays the benefits of not simply being esthetically pleasing however additionally recyclable, renewable and perishable [57]. Wood fiber polymer composites (WFPC) are ordinarily created by combining wood fiber with a polymer, or by addition wood fiber as filler in a polymer matrix, and compacting with great pressure and suitable temperature [58].

There are four recurring themes which will form the upcoming of the composite engineering: systems keys, inexpensive manufacturing processes, various markets, and new skills.

Composites usually use thermoset resins that started as fluid polymers and modified to solids throughout the casting method.

This process, define as cross-linking, so that the composite materials have chemical resistance, higher mechanical properties, and larger structural sturdiness than thermoplastics. The electric utility trade will facilitate the composite productiveness grow in the twenty-first century by serving to makers of composite materials to develop producing processes that are extra economical, productive, and efficient [59].

### 3.2 Polymers

Polymers also identified as large molecules in which atoms are coupled together by covalent bonds along the molecules. The polymer chain consists of a huge diversity of tiny molecules adverted to as monomers or continual units which are linked together with chemicals. Polymers are prepared by a method called polymerization wherever monomers (structural units) react along chemically to make linear or split chains or three-dimensional polymer network. These extended molecules are primarily based predominately upon carbon chains, that include atoms having robust bonds among themselves, however comparatively weak bonds to adjacent molecules [60- 63].

The most vital in these polymers are acrylic, cellulose acetate, nylon polymeric amide, and polycarbonate. Epoxy and polyester are wide utilized as matrix materials in reinforced composites. Their physical and mechanical properties are presented in Table 3.1.

Principally application areas of polymeric matrix composites are marine applications, automotive and different transportation industries and sports equipment because of their erosion resistance and light weight [64].

**Table 3.1** The physical and mechanical properties of polymeric matrix materials [64]

| Property | Epoxy | Polyester | Vinylester |
|---|---|---|---|
| Density ($g/cm^3$) | 1.1-1.4 | 1.2-1.5 | 1.044 |
| Modulus of elasticity (GPa) | 3-6 | 2.0-4.5 | 3.2 |
| Poisson's ratio | 0.38-0.4 | 0.37-0.39 | --- |
| Tensile strength (MPa) | 35-100 | 40-90 | 80 |
| Compression strength (MPa) | 100-200 | 90-250 | --- |
| Thermal conductivity (W/m°C) | 0.1 | 0.2 | --- |
| Thermal expansion (°C) | 50-300 | 50-110 | --- |
| Heat distortion temperature (°C) | 50-300 | 50-110 | --- |
| Solidifies tensile (%) | 1-2 | 4-8 | --- |
| Water absorption (%) | 0.1-0.4 | 0.1-0.3 | 0.150 |

### 3.3 The Classification of Polymers

Important classifications of polymers are shown in Table 3.2.

**Table 3.2** Shows the basis of polymers classification [65]

| Basis of classification | Polymer type |
|---|---|
| Origin | **Natural, semisynthetic, synthetic** |
| Thermal response | **Thermoplastic, thermosetting** |
| Mode of formation | **Addition, condensation** |
| Line structure | **Linear, branched, cross – linked** |
| Application and physical properties | **Rubbers, plastics, fibers** |
| Tacticity | **Isotactic, syndiotactic, atactic** |
| Crystallinity | **Non – crystalline (amorphous), semi – crystalline, crystalline** |

### 3.3.1 Thermoplastic Polymer

This group of polymers consists of linear or branched molecules, for example, polyethylene, acrylic, and polystyrene are known as thermoplastics. These materials may be heated and formed in addition they can be reheated and returned to the liquid state. Figure 3.1 shows types of polymers schematically [66, 67, 68, 69].

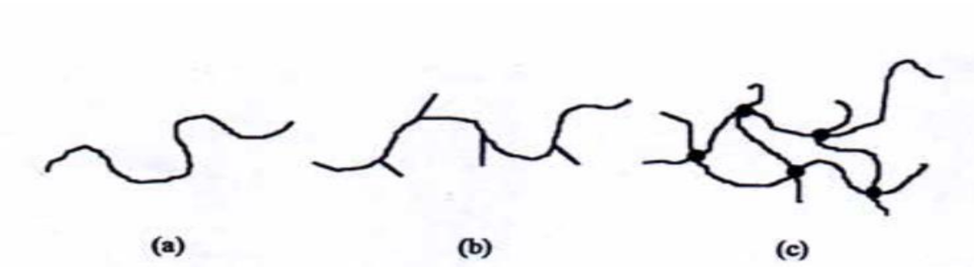


**Figure 3.1** Schematic representations of (a) a linear polymer, (b) a split polymer, and (c) a network polymer [66]

### 3.3.2 Elastomers Polymers

These polymers are network polymers that are gently cross-coupled. They are reversibly stretchable to high extensions. When unexpanded they need fairly tightly arbitrarily rolled molecules which are stretched once the polymer is stretched. The cross- links inhibit the molecules from flowing past one another once a material is expanded. On cooling, rubbers got crystallize (slightly). On heating, they can't melt within the typical sense, (i.e.); they can't flow, attributable to the cross- links [66].

### 3.3.3 Thermosetting Polymers

These polymers are network polymers that are mightily cross- joined to present a dense three–dimensional network. They are ordinarily rigid, cannot melt on heating and that they decompose if the temperature is high enough. The name arises because it's necessary to heat the first polymer of this sort for cross-linking or treating, to take place. The term is currently utilized to adjective this sort of material even when heat isn't needed for the cross-linking to take place [66, 68, 69].

### 3.4 Unsaturated Polyesters

Unsaturated Polyesters resin (UPR) is a lined polymer with low molecule weight. The polyester includes double bonds and carboxylic teams.

UPR is achieved by the process of condensation polymerization for three basic components: saturated carboxylic acids, unsaturated carboxylic acids, and glycols with rending out water, and increasing in molecular weight by a method known as esterification [70, 71, and 72]. Some monomer like styrene can boost the polyester to urge cross-linking during polymerization method process [73].

The unsaturated polyesters that contain styrene are also polymerized at room temperature without the attendance of a catalyst if given enough time, thus inhibitors are accessorial to forestall such reaction till ready use. Cure begins by adding an initiator (a catalyst) [72].

### 3.5 Composite Materials

Composite is mostly outlined as any physical mixture of two or more dissimilar materials used to manufacture a replacement material which is that can't be obtained by every part separately. Composites include one or more disconnected phases subsumed during a constant phase. The disconnected phase is sometimes tougher and violent than the continual phase, it is termed the reinforcement, whereas the continual phase is definite the matrix. The boundaries amidst the two phases are adverted to as interface [74, 75].

### 3.6 Classification of Composite Materials

The composite materials advanced so far have been invented to enhance the properties, like strength and toughness, additionally the electrical properties. The strengthening mechanism depends on the sort of reinforcement materials and on the reinforcement geometry [74]. The composite materials are distributed in line with the sort of matrix into polymers, metals, and ceramic-matrix composites [76]. Composite materials are categorized into fibrous composites, particulate composites and laminated composites [77]. The classification pattern of the composites product is summarized in the following diagram of Figure 3.2.

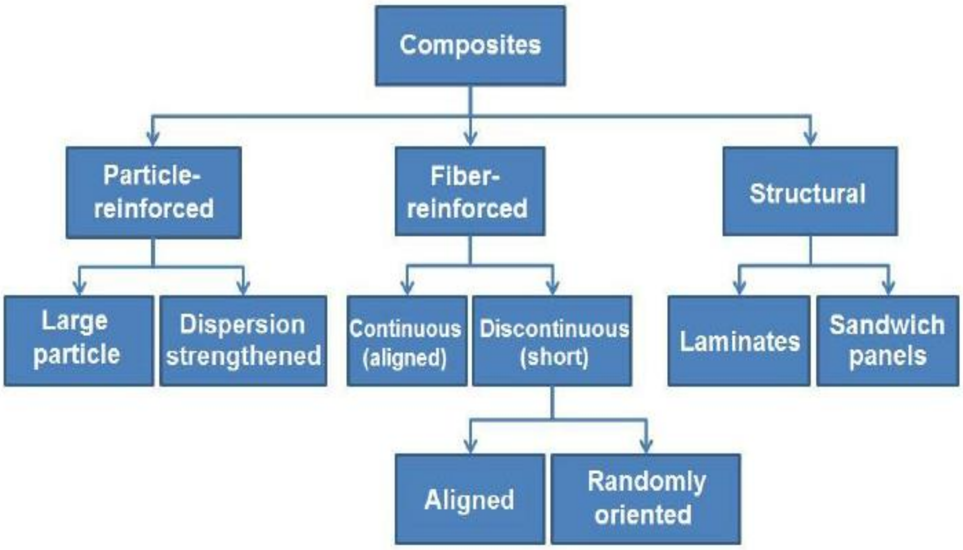


**Figure 3.2** Classification of composite materials [78]

**3.6.1 Fibrous Composites**

In these composites, the reinforcing materials are in the variety of fibers like glass fibers, boron fibers, graphite fibers, carbon fibers, silica fibers, tungsten fibers, kevler-49 fibers, beryllium fibers, whisker fibers and different fibers [79].

Composites for large fibers are known as continuous-fiber-reinforced composites while these for tiny fibers are named as discontinuous-fiber-reinforced composites [80]. The contact between the matrix and fibers is incredibly efficient in rising the crack resistance of the matrix. The fibers have little cross-sectional dimensions in order that they are incorporated in matrix materials to make fibrous composites as shown in Figure 3.3. The matrix helps to fix the fibers jointly, transferal forces to the fibers and defend them versus ecological injury because of handling. The fibrous composites become the foremost vital category of composite materials as they are capable of verifying high strengths. Fibrous composites are often classified into single-layer and multilayer composites.

Single-layer composites may be produced from some separate layers with each layer having the similar properties. Multilayer composites contain many layers of fibrous composites. Every layer is a single – layer composite. Once the essential materials in every layer are identical, they are known as simple laminates, the hybrid laminates are involving of layers created of completely various materials [74].

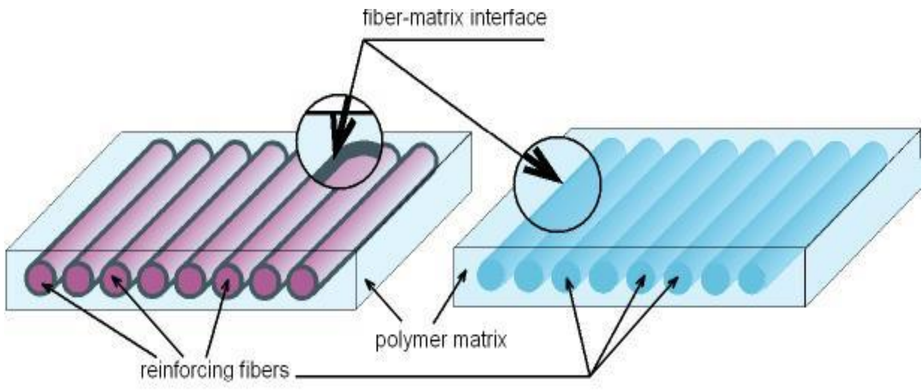


**Figure 3.3** Fiber and Self- reinforced Composite [81]

### 3.6.2 Particulate Composites

Particulate composite is a material consisting of one or additional constituents suspended in a very matrix of a different material (Figure 3.4). The particles and matrix material in these composites can be any mixture of mineral and unmineralized [82]. Particles like ceramic, metal and inorganic ones are utilized as reinforcement materials in metallic matrices. The particulate composite ensuing from ceramic as the non-metallic particles in metal matrix is termed cermet. Also, fillers are most generally used additives in the polymer composition. They are utilized in all plastics, natural and artificial rubber, and in the coating. The leading aim for their use is the need for cheaper materials or for an important improvement in some properties (rigidity, strength, toughness resistance to temperature, etc.).

Reinforcing fillers are further to boost sure mechanical properties like modulus or tensile strength, scale back the shrinkage and increase the hardness. Reinforcing fillers usually can upsurge the tensile, compressive, and shear strength, scale back shrinkage, increase the modulus, and expand the creep behavior [74, 82].

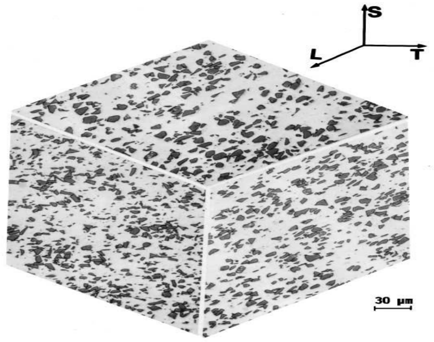


**Figure 3.4** Particulate Composites [83]

### 3.6.3 Laminated Composites

Laminated composites incorporate layers of a minimum of two totally different materials that are fused together (Figure 3.5). These composites are characterized by several properties like strength, toughness, little weight, erosion resistance and thermal isolation. There are abundant sorts of laminated composites like wried metals, laminated glass and laminated fibrous composites material [80, 82].

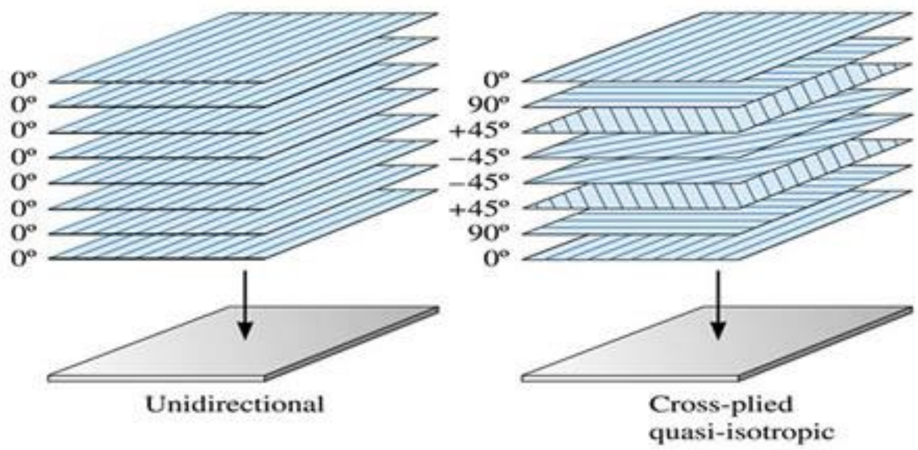


**Figure 3.5** Laminated Composite [84]

### 3.7 Wood

Wood is that the famous and more generally utilized in operational materials. It's documented usage in structural and ships spans over 5000 years ago.
In the 17th and 18th centuries, many of Europe were deforested fully by the exponential growing in exhaustion of wood [85].

Wood (timber) is that the most generally utilized in engineering construction material within the United States, with its annually created tonnage exceeding all alternative engineering materials, together with steel and concrete. Additionally to the employment of wood for timber and lumber for the development of homes, buildings, and bridges. Wood is additionally utilized to produce composite materials like plywood and particleboard. It is a naturally occurring composite material that includes principally of a combined array of cellulose cells supported by a polymeric material adverted to as lignin and alternative organic compounds [86].

### 3.8 Composition of Wood

The polysaccharides of wood are copulatively known as holo cellulose which means total cellulosic carbohydrates. The holo cellulose accounts for regarding 70-80 % of the extractive tree wood tissue, with polymer creating up the remainder [87]. Woods are distributed into softwoods that are important in creating paper as result of their longer fibers (about 3-4mm), and hardwoods with their shorter fiber length (1mm) [88].

The chemical variations are:

1. A lot of cellulose is current in hardwood than softwood.
2. Less lignin is present in hardwood than softwood.
3. A lot of hemicelluloses is present-day in hardwood than softwood.
4. Conversely, a lot of xylans is present in hardwood than softwood hemicelluloses.
5. The composition of hardwood and softwood extractive differ.

### 3.9 Cell-Wall Structure

The mainly components for the cell of wood cellulose with lignin. Cellulose crystalline molecules form up between (45 – 50) % of the solid material of wood. Cellulose is a lined polymer involving of glucose units with a grade of polymerization starting from 5000 to 10,000. The chemical bond inside and amidst the glucose units makes a straight and stiff molecule with spired tensile strength. Lateral relatedness bien the cellulose molecules is by hydrogen and permanent couple bonding. Hemicellulose makes up (20 - 25) % by weight of the solid material of wood cells and could be a branched amorphous molecule containing many varieties of sugar units.

The third main essential of wood cells is lignin, that constitutes regarding (20- 30) % by weight of the solid material.

Lignins are terribly complicated, cross-linked, three-dimensional polymeric materials shaped from phenolic units [86].

### 3.10 Natural Fiber Reinforced Polymer Composites

Above the older twenty years, natural fibers are receiving respectable interesting because of the replacement for fiber strengthening like glass/ plastics. The benefits of natural fibers are low price, little density, satisfactory qualitative strength, sensible thermal isolation, decreased dermal and respiratory irritation, natural resource and recycling potential while not moving the environmental harm, and perishable ability [89–93]. Several works dedicated to the properties of the natural fibers from micro to Nano scales are on the market.

On the past ten years ago, it was seen a quick and constant development of wood plastics manufacture. Among several accounts for the business success, a little value and reinforcing the capability of the wood fillers supply new chances to industry composite materials. Though the usage of wood-based fillers isn't as in style because of the usage of inorganic fillers, wood-derived fillers have many blessings over ancient fillers such as low density and ductility, throughout the process with no damage to the instrumentation. The most application fields of wood flour stuffed composites are the construction manufactures within which they are employed in building requirements as adorning, window components, roofline merchandise, door panels, etc. Natural fiber-reinforced polymers (Figure 3.6) have component attention in the automobile business for the subsequent causes. The requests within which natural fibers composites are currently applied as well as door inner panel, roof inner panel, seat back, and so on.

1. They are ambience-friendly, which means that they are perishable, and in contrast to carbon and glass fibers, the consumption of the energy to provide them is incredibly tiny.
2. The density of natural fiber is within the vary of 1.25-1.5 g/cm3 comparable with 2.54 g/cm3 for E-glass fibers and 1.8-2.1 g/cm3 for carbon fibers.
3. The modulus–weight proportion of many natural fiber is larger than that of E-glass fibers, which implies that they can be terribly competitive with E- glass fibers in hardening-critical designs.
4. The natural fibers composites give upper voice damping than glass or carbon fiber composites; thus they are more appropriate for noise reduction, a progressively vital demand in internal automotive orders.

5. The natural fibers are abundant cheap than the other inorganic fibers.

There are many calibrations of natural fibers.

The natural fiber tensile strength is comparatively low. While the opposite limitations are lowland melting point and wet absorption. At higher temperatures than 2008°C, natural fiber begin to degrade, initial by the hemicellulose degradation so by the degradation of lignin [96, 97].

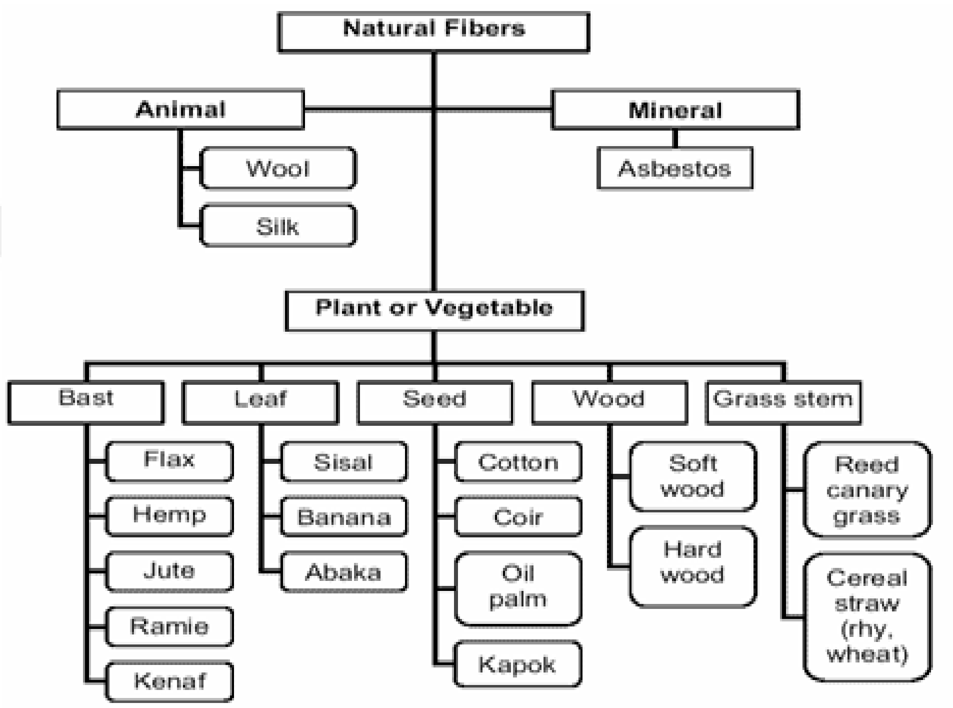


**Figure 3.6** Schematic illustration of natural fibers **[**98**]**

### 3.11 Manufacturing Techniques of Composite Materials

Composite materials can be produced by totally different producing techniques and be producing depends on geometry, desired quality, cost, and skill. These techniques might roughly divide into two groups as open and closed molding.

In open molding, the process is completed underneath the impact of the atmosphere. In closed molding, the method is completed with the help of molds or vacuum bags that block the effect of the atmosphere.

**Table 3.3** The data of composite manufacturing techniques in World, Europe, and Turkey [99]

| Manufacturing Techniques | World | European | Turkey |
|---|---|---|---|
| Hand Lay-Up | 21% | 18% | 25% |
| SMC | 10% | 14% | 3% |
| BMC | 9% | 5% | 3% |
| GMT/LMT | 2% | 7.5% | --- |
| Enjection | 29% | 25% | 10% |
| RTM | 3% | 8.5% | 5% |
| SRIM | 1% | 0.5% | --- |
| Pultrusion | 10% | 4% | 2% |
| Continuous Laminating | 7% | 6% | 7% |
| Filament Winding | 8% | 11.5% | 45% |

### 3.12 Types of Tests are being Applied Composite Materials

In order to see the strength properties of composites, mechanical tests are applied. By these tests, some data are obtained from made composites that carry or not the expected properties. By utilizing obtained data continuity of production is provided or by ever-changing of some parameters similar the applied production sort, quantity and kinds of materials were used, will expect mechanical properties are often manufactured. In general mechanical tests applied to composite materials are tensile, three-point bending, impact tests.

#### 3.12.1 Impact Test

The most important of those limitations is a reaction against regional impact which they are exposed to the foreign material under operating situation. To know the impact react of composite materials, several researches were accomplished. Test apparatuses used to measure the response to the impact load of a composite are Izod and Charpy pendulum impact test, drop weight test and gas gun test.

### 3.12.1.1 Charpy Impact Test

In the initial stages of the impact study for composite materials, designed primarily for metal charpy impact test system is used. Two basic causes for the employment of this mechanism is that it's comparatively easy and adjustable. Thus able to give data concerning energy absorption mechanisms for composites. The test sample is mostly a thick beam and sometimes a notch is opened within the medium of the beam. The sample is supported on a horizontal plane and exposed to oscillating loading by a hammer blow from the other of the notch (Figure 3.7). Throughout impact test absorbed energy is scan by the assistance of a scale that on the test device.

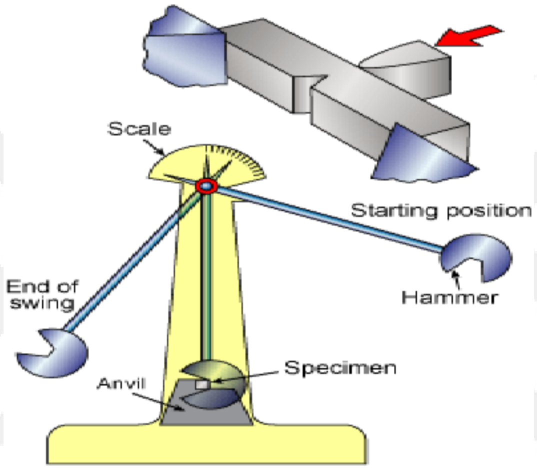


**Figure 3.7** Schematic illustration of charpy impact test [100]

### 3.12.1.2 Izod Impact Test

In the Izod impact test, test equipment and method steps are almost like mentioned within the Charpy test. Within the Izod test, the sample is connected from at one edge as a fixed beam at the vertical plane and is exposed to impact loads by a free pendulum from the unsupported finish. See Figure 3.8.

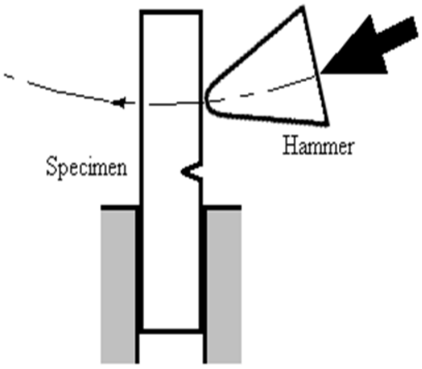


**Figure 3.8** Schematic illustration of izod impact test [101]

#### 3.12.1.3 Drop Weight Test

In the drop weight test, the load release from an explicit height to the sample that is fastened within the horizontal plane. Impact doesn't cause deformation of the sample absolutely. The speed of impact can be measured with the help of optical sensors placed simply on the sample or motion equations.

The advantage of this test according to Charpy and Izod test is applied to more complex material systems to the sample. Despite the employment of principally semi-spherical geometry tip purpose, sharp and flat sort tip purpose can also be used for impact.

#### 3.12.1.4 Gas Gun Test

In this testing mechanism, nitrogen or an identical gas is filled to the reservoir that is placed the end of the gun barrel. Where the gas is stated by a plastic diaphragm. The diaphragm explodes when the gas reaches the selected pressure and quickens the impact tip point that is within the tube and this allows to hit specimen that clamped to a various end. The hit speed of tip point may be measured by the assistance of optical sensors or with a straightforward wire breaking mechanism [102, 103].
It can be utilized for giant test items and so used to measure high-speed impact behavior of composite materials.

### 3.12.2 Tensile Test

The purpose of the tensile test is determining the elastic and plastic behavior of materials under constant load. For this, accordance with standards size circular or rectangular test pieces are being connected tensile test device and the variable and axial forces are applied. Tensile test device combined of two jaws which may move essentially up and down relative to each other (Figure 3.9). The test piece is connected to those jaws that provide force and movement. By one in every of the jaws move with a continuing speed, variable amounts of tensile force is applied to the test part and elongation similar to this force is recorded.

**Figure 3.9** Typical tensile test machine [104]

### 3.12.3 Three-Point Bending Test

It may be outlined as the deformation occurring within the sample when force is applied in the midpoint of a test sample that is a circular or rectangular cross-section that placed freely on the two supports (Figure 3.10). If the sample is broken, applied load measured throughout the fracture. The section of the three-point bending test specimens may be a circular or rectangular section. In keeping with the overall number of supports that contact with specimen and load applications three-point bending test is split into completely different teams, like three-point bending test or four-point bending test. The most purpose of the three-point bending test is to see the flexibility to endure the cold state cracking of the material. Tensile and compressive stresses are happen at the cross section of the sample which subjected to bending [105].

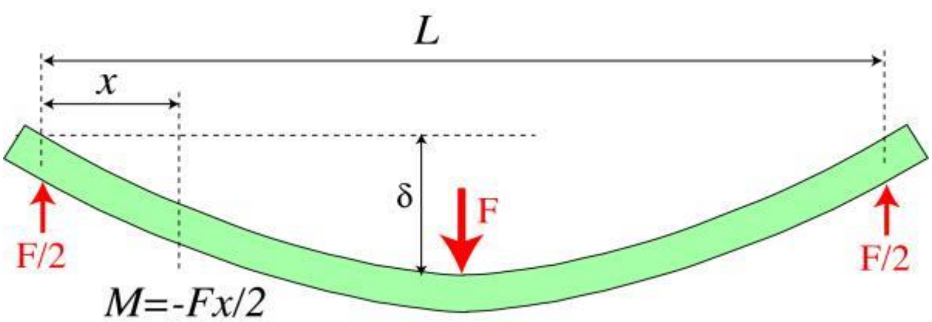


**Figure 3.10** A basic illustration of three-point bending test [106]

# CHAPTER 4

# EXPERIMENTAL METHOD

## 4.1 Polyester Resin

Polyester resins are the furthermost shared resin in the manufacturing of composite materials. These resins are collected in two groups as saturated and unsaturated polyesters. Saturated polyester resins are used in the injection molding process [107] and unsaturated polyesters resin (UPR) is utilized in the structure of fiber-reinforced materials.

Generally, a small quantity inhibitor should be added to slow down the movement of the gelation during the manufacture of polyester resin. For use in molding, several auxiliary products are added to the polyester resin. These products are usually the catalyst accelerator additives for instance pigments, fillers, supplements for chemical resistance. The polymerization proportion is very slow for polyester resins; therefore, the accelerator and catalyst must be added to polyester resins to obtain a faster polymerization process. The polyester resins show good properties in terms of chemical and mechanical resistance, low prices, and flexible usage of intended design [64]. Due to being inexpensive, polyester resins are preferable [99]. In this experimental study, the unsaturated polyester (Polipol 3401-TAB) with a density of 1.105 $gr/cm^3$ were obtained from Poliya chemical company, Turkey.

## 4.2 Hardener (Methyl Ethyl Ketone Peroxide - MEKP)

The famous common initiator for working which are made at room condition is (MEKP) as an organic peroxide. They provide the energy necessary to initiate the polymerization. Hardener (MEKP) is used as 2 % of polyester weight for all specimens. Some properties about MEKP are given as [108].

The physical condition : transparent liquid

Density (kg/$m^3$, 20 °C) : 1170

Flash point (°C) : 60

Viscosity (mpa.s, 20 °C): 25

### 4.3 Pistachio Shell Powder

Pistachio shell powder that is used as filler is provided from Gaziantep city, Turkey (Figure 4.1.a). Table 4.1 [109] is shown the composition of PS. One of the important critical points are the volume of particle on the mechanical properties of composite materials such as homogeneous distribution, adhesion and holding to resin/matrix [110, 111, 112, and 113]. The cleaned pistachio shells were grounded into powder form using a jaw crusher. The net powder was filtered to get particle size less than 5 microns (Figure 4.1.b). Short and small size additives should be preferred due to it provides great specific shallow area and uniform distribution in the composite [110, 112]. Pistachio shell powder is filtered in order to get homogeneous size distribution, good adhesion and provide a good uniformity which will be molded and tested. The weight fraction of powder was used between 0% and 25%.

**Table 4.1** Chemical compositions of pistachio shell [109]

| **Parameter** | **Value (wt %)** |
| --- | --- |
| Cellulose | 42 |
| Lignin | 13.5 |
| Cellulose lignin | 3.11 |
| Extractable | 0.18 |
| Ash content | 1.26 |

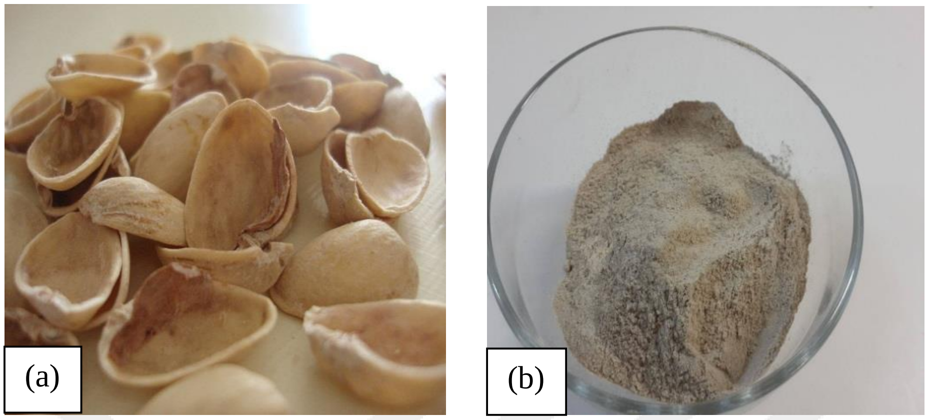


**Figure 4.1** (a) Pistachio shell (b) Pistachio shell powder

### 4.4 Stirring of Materials and Preparation of Specimens

For every specimen of samples, mixing and molding processes are performed simultaneously. All specimens were achieved using hand lay-up process by stirring the mixture of PS/polyester resin for 20 minutes at a constant speed of 1000 rev/min. Afterword, hardener with 2 wt% was added to the mixture for quick setting of composite specimens. The mixing process continued up to mixture resin became gel form. In this way, by hold on powder to the mixture, it is planned to prevent the agglomeration and sedimentation.

Then, the mixture was poured into the mold. These specimens were post cured at the room temperature for three weeks before mechanical testing. At least three specimens were prepared for every test and for each composite and average value of the resulted data was determined. The thickness of all specimens for tensile, three-point bending and charpy impact tests were 4 mm. Figure 4.2 showing the polyester before and after adding the pistachio shell powder; Figure 4.3 showing the beginning of mixture; Figure 4.4 showing the cast of the mixture (polyester- hardener and pistachio shell powder).

**Before** **After**

**Figure 4.2** polyester before and after adding the pistachio shell powder

**Figure 4.3** Beginning of mixture

**Figure 4.4** Cast of the mixture (polyester-hardener and pistachio shell powder)

### 4.5 Mechanical Test

#### 4.5.1 Tensile Test

Three samples are molded in order to apply tensile test accordance with standard ASTM D 638 (165 mm × 12.7 mm sizes for a gauge length of 50 mm) [114].

Samples are tested with a 300 KN capacity universal test machine Shimadzu AG-X series (Kyoto, Japan) (Figure 4.5) with crosshead speeds of 2 mm/min constant speed. The mechanical properties of the samples (tensile strength and strain, bending strength and strain, and flexural modulus) were determined from the test machine. Each test was performed for three different specimens. Results were interpreted and the average of three samples was taken. The specimens before tensile test are shown in Figure 4.6.


**Figure 4.5** Shimadzu AG-X series testing machine


**Figure 4.6** Tensile test specimens

### 4.5.2 Three-Point Bending Test

Three samples were molded in order to apply this test with standard ASTM D 790 (127 mm × 12.7 mm sizes with span to depth ratio of 16:1) [115].

Samples are tested with a 300 KN capacity universal test machine Shimadzu AG-X series (Kyoto, Japan) (Figure 4.7) with crosshead speeds of 1 mm/min constant speed. A big span to depth proportion increases the maximum stress without influence the inter-laminar shear stress and thus increases the propensity for linear failure. When the span is short, the failure will start and spread by interlaminar shear disappointment. The ultimate shear stress in a beam happens at the mid-plane. In the shear test, failure involves of a tear running along the mid-plane of the beam, therefore, crack plane is corresponding to the linear plane. Results are interpreted taking the average of three samples. The calculation of bending stress is made by using formulas 4.1, 4.2 and 4.3 [115]. Figure 4.8 showing the samples before three-point bending test.

$$\sigma_F = \frac{3P_{max}\,L}{2bh^2}\left[1 + 6\left(\frac{D}{L}\right)^2 - 4\,\left(\frac{h}{L}\right)\,\left(\frac{D}{L}\right)\right] \tag{4.1}$$

$$E_F = \frac{m\,L^3}{4bh^3} \tag{4.2}$$

$$\varepsilon_F = \frac{6Dh}{L^2} \tag{4.3}$$

Where L is the span, b is the width, h is the depth, m is the slope of the tangent to the initial straight-line portion of the load–deflection curve, D is the maximum deflection before fracture, and P is the loads at a point on the curve.

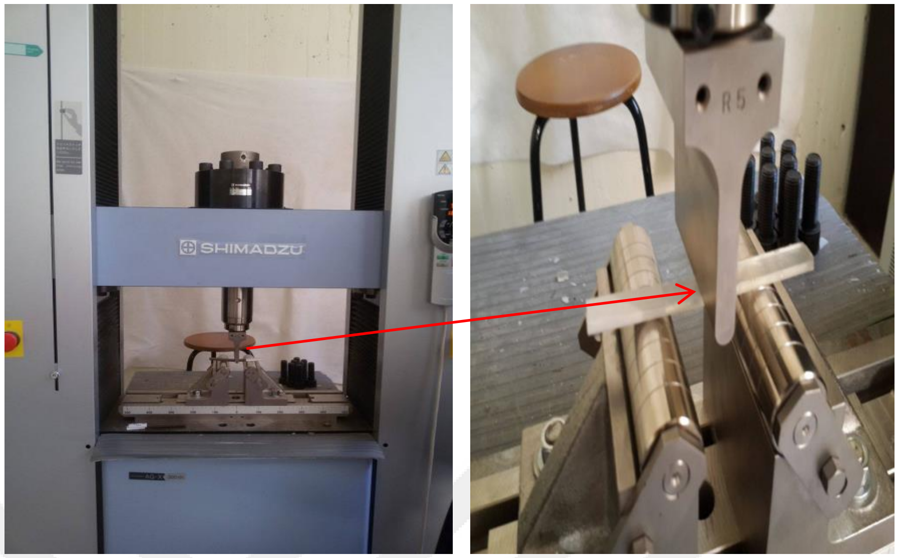


**Figure 4.7** Shimadzu AG-X series testing machine

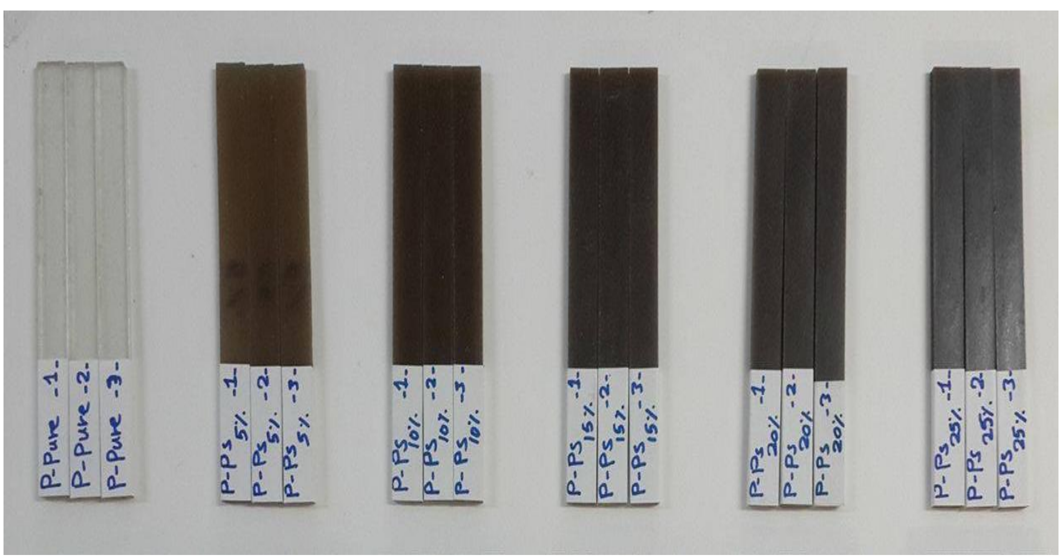


**Figure 4.8** Three-point bending test specimens

### 4.5.3 Charpy Impact Test

Charpy impact test machine having a 15 J hammer (Köger, Germany), as shown in Figure 4.9, was employed for the charpy impact test according to ISO 179/92 standard. The test arrangement consists of four main parts: pendulum, anvils, and dial of the test machine, in addition to the specimen. During charpy impact test, the absorbed impact energy is obtained by the assist of a scale which on the test machine. The un-notched specimens were prepared with a size of 55 mm × 10 mm in accordance with the ISO standard of 179/92. All experiments were achieved at room temperature with typical moisture. Charpy impact test represents the energy needed to break the sample. This energy is called as absorbed impact energy and denoted by W and measured by J. Impact specimens before test are shown in Figure 4.10. The absorbed impact energy and impact strength of tested specimens were determined according to the following equations:

$$W = W_1 - W_2 \quad (4.4)$$

$$K = W/(bh) \quad (4.5)$$

K is the impact strength ($KJ/m^2$), b is the width (m), h is the thickness (m), and $W_1$, $W_2$ is the potential energies before and after impact.

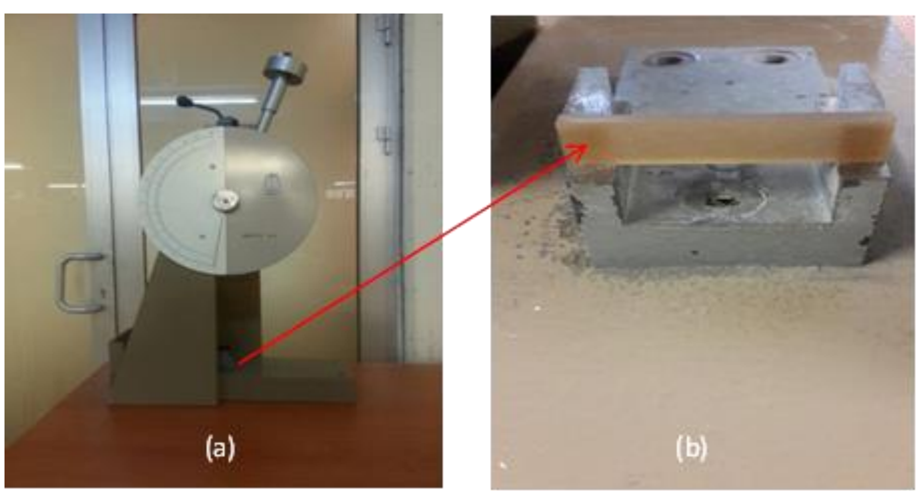


**Figure 4.9** Köger 3/70 testing machine: (a) the gradated dial/ impact tester, (b) the specimen propped on the anvil.

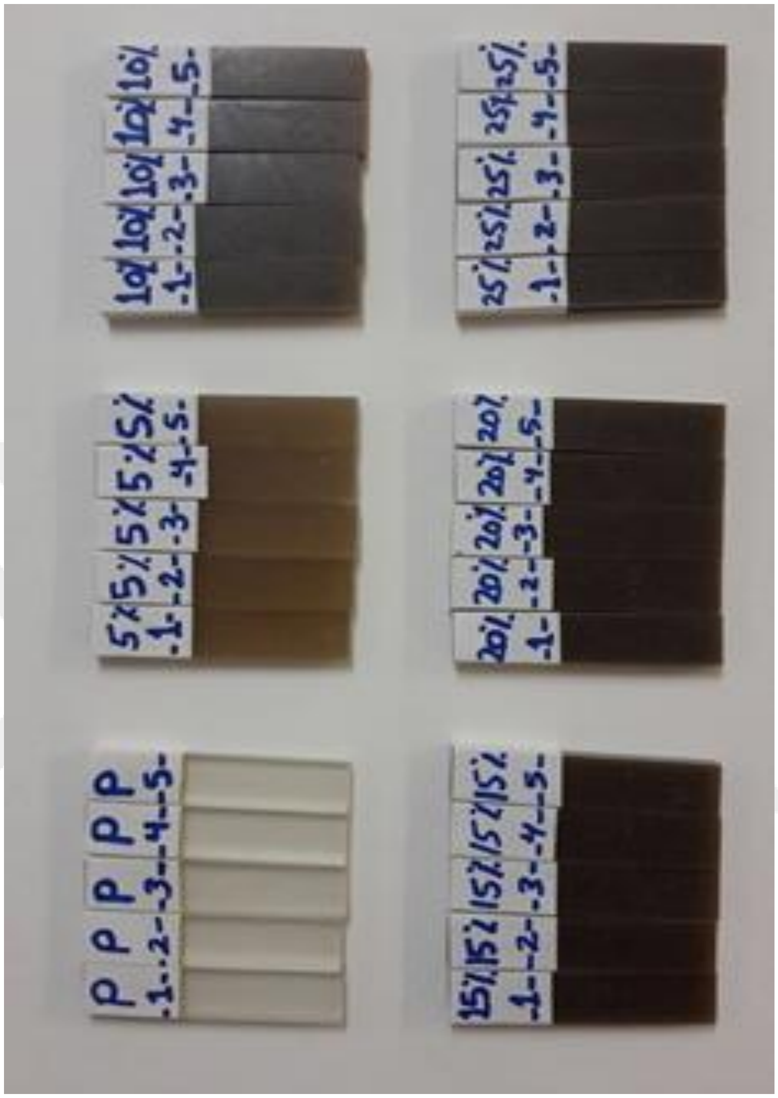

**Figure 4.10** Charpy impact test specimens

# CHAPTER 5

# EXPERIMENTAL RESULTS AND DISCUSSIONS

## 5.1 Scanning Electron Microscopy

The morphology of the polyester matrix composites filled with pistachio shell (PS) particles was observed using Scanning Electron Microscope (SEM), and their images for particle contents of 0, 5, 10, 15, 20 and 25 wt% are presented in Figure 5.1. The good distribution of pistachio particles was obtained for PS content of 5 and 10 wt%.

In addition, particle aggregation occurs at content above~10 wt% of pistachio particles, thus resulting increase in particle size leading to weakness in composite strengthening. As shown in Figure 5.1, the diameter of large particles increased from 5 µm at a content of 10 wt% until more than 30 µm at a content of 25 wt%. Therefore, the larger particle size may be worked as stress concentration points and eventually weakened the interfacial adhesion bond between PS particles and polyester matrix [116, 117]. The images were taken at a magnification of 5 KX.

## 5.2 Experimental Test Results

Tensile and three-point bending tests are performed for three different specimens and results are taking the average of three samples but charpy impact test is performed for five specimens and results are taking the average of five samples.

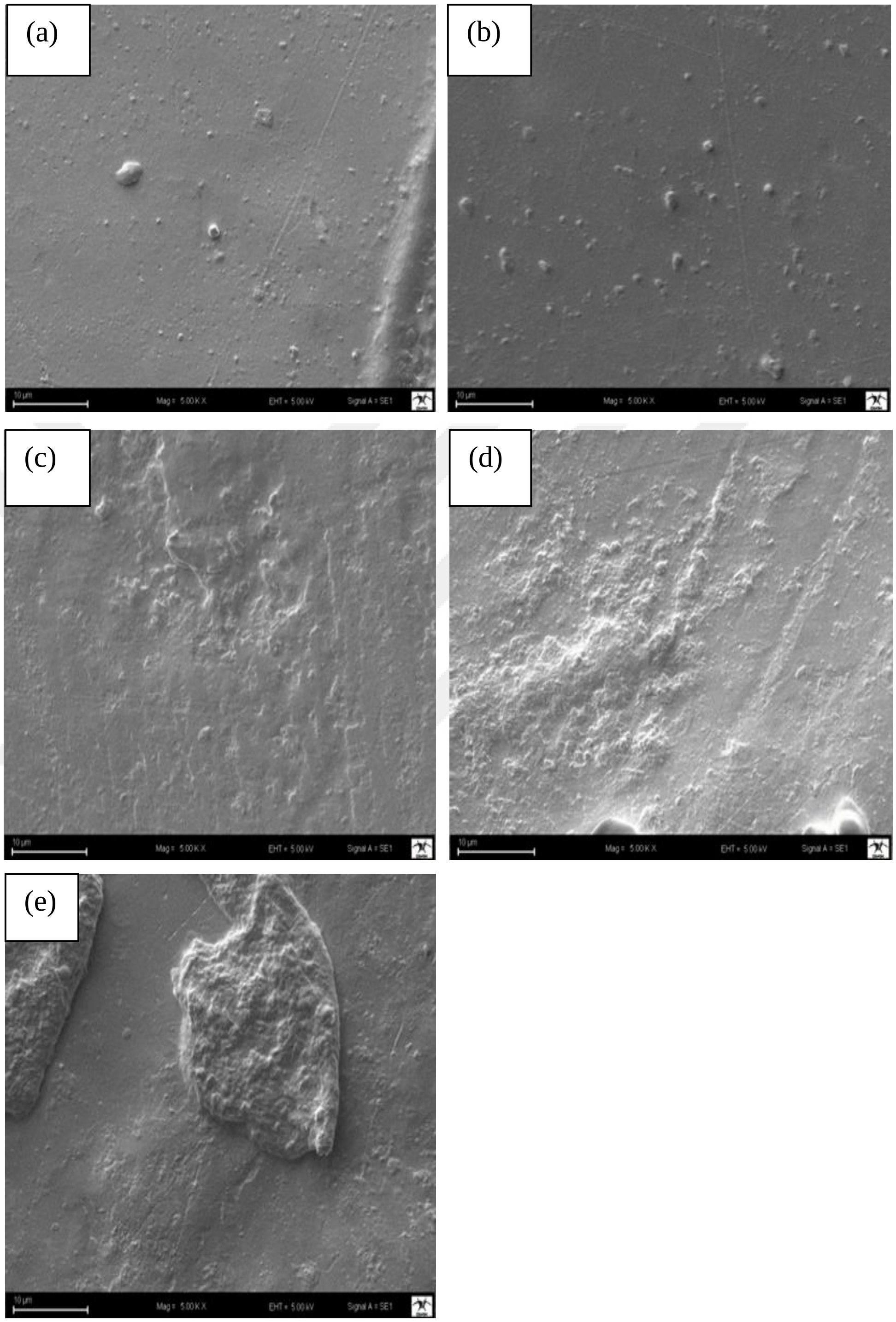


**Figure 5.1** SEM micrographs of the PS/polyester composites at particle content of (a) 5 wt%, (b) 10 wt%, (c) 15 wt%, (d) 20 wt% and (e) 25 wt%.

### 5.2.1 Tensile Test Results

As shown in Figure 5.2, the fracture occurs at maximum force and tensile strength increase until PS weight fraction of 10% of the total mixture (polyester-hardener-shell powder). It can be understood that homogenous (distribution of shell powder in the mixture) is good, adhesion and interaction of resin and shell powder are good, test result showed continuity improving each other between weight fraction of PS powder 0% and 10%. The maximum force was decreased from 10 wt% up to 25 wt% of PS powder content. The particulate filled composites are combined of severe fillers distributed in a polymer matrix regularly brittle by itself. Those fillers can act such as stress raisers which cause weakness in the synthesis by presenting irregularity in stress relocation process through the filler-polymer interface. The output stress focus at the failing sites lead to early starting of the failure procedure.

There are two reasons of this drop in the strength properties of the particulate filled composites comparable to the net risen ones. One probability is that the chemical response at the boundary among the filler particles and the matrix may be very feeble to carry the tensile stress.

The another probability is that the turning points of the asymmetrically formed particulates result in stress focus in the resin. These two reasons are accountable for decreasing the tensile strengths of the composites so considerably [118].

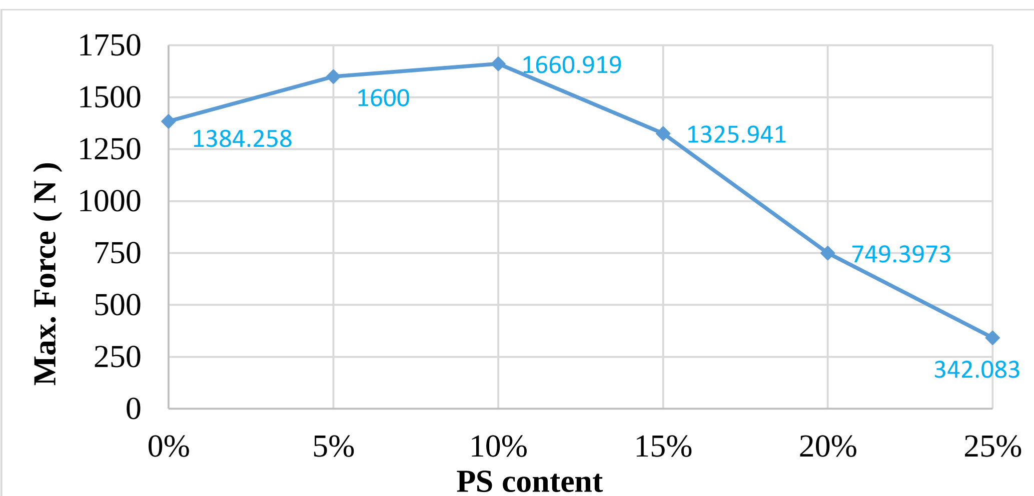


**Figure 5.2** Maximum force versus PS content

The tensile stress-strain curves of the PS/polyester composite specimens are shown in Figure 5.3. As illustrated in this figure, the tensile stress-strain behavior of the polyester matrix is significantly influenced by the addition of PS particles. It is found that the elastic limit increased up to PS content of 10 wt% then decreased. Furthermore, the elastic plastic curves exhibit semi-linear response.

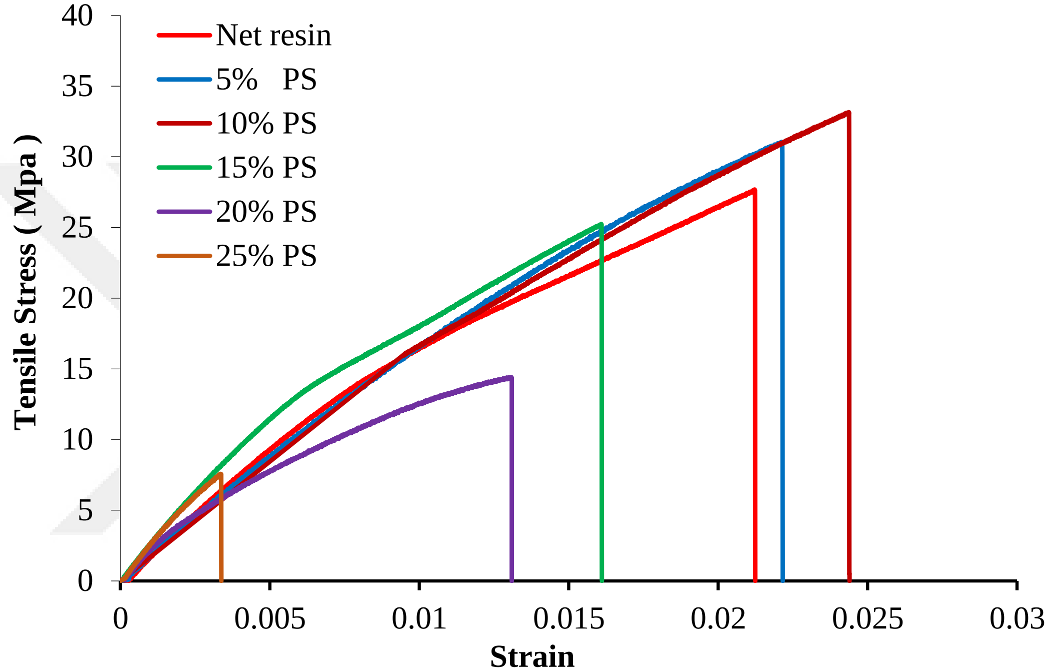


**Figure 5.3** Tensile stress-strain curves of the PS/polyester composite

Figure 5.4 show samples of the failed specimens of the tensile test, for all specimens the fracture surfaces were flat without nicking. This behavior refers to the specimens totally fractured in a brittle style through the testing. Also, there was no active reducing in the area (cross-sectional) of the specimens. Table 5.1 presents the mechanical properties of PS particle-filled polyester composite for various filler contents. Figure 5.5 illustrated the PS content versus tensile strength and strain at break of the PS/polyester composites. As seen in the Figure 5.5 and Table 5.1, the tensile strength improved up to PS content of 10 wt% and then followed the trend of decreasing. Therefore, the maximum enhancement of tensile strength was 19.9% occurred at 10 wt% content of PS. The decreasing in tensile strength can be refer to the relation of adhesion and interaction among PS particle and polyester matrix. The PS particle aggregation occurred led to increasing in particle size and caused stress concentration points which can be weakened the interfacial adhesion between the matrix and particle. In other words, the bonding at particle/matrix interface is very weak to carry the tensile stress [7, 119, 120]. In addition, the corner points of the unequal shaped for PS particle result in stress focus in the resin [118].

Figure 5.5 demonstrates also the strain at break was decreased at high PS particle content due to mechanical restraints of the particles interference in the deformability of the matrix, thus dropping the strain at break. However, the strain at break was increased by 9.2% at PS particle content of 10 wt% compared to the unfilled polyester matrix.

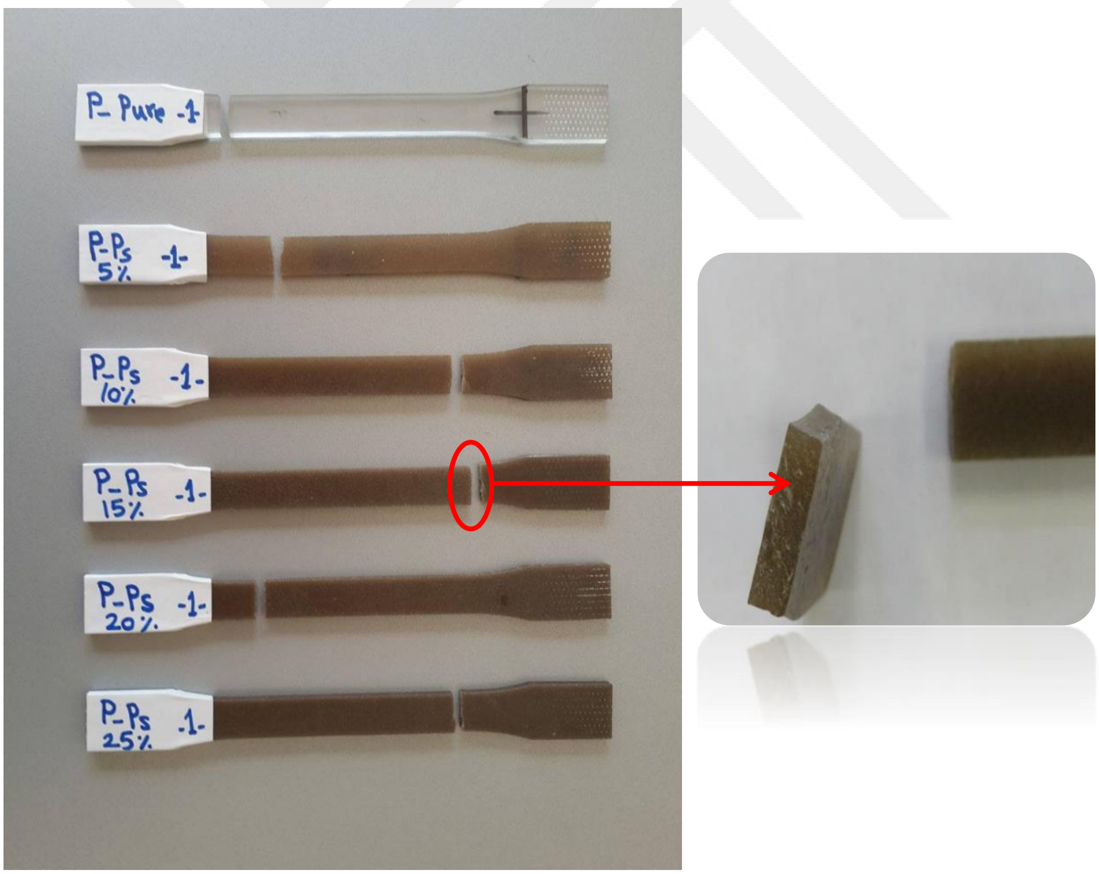


**Figure 5.4** Samples of the failed specimens of tensile test of the PS/polyester composites.

**Table 5.1** Mechanical properties of the pistachio shell powder/polyester composite

| Composite Type | Filler content (wt%) | Tensile strength (MPa) | Flexural strength (MPa) | Flexural modulus (MPa) | Absorbed energy (J) | Impact strength ($KJ/m^2$) |
|---|---|---|---|---|---|---|
| Neat resin | 0 | 27.6 ±1.07 | 82.5 ±0.99 | 2310 ±74 | 0.33 ±0.05 | 8.25 |
| $P\text{-}PS_5$ | 5 | 31.0 ±1.40 | 85.8 ±1.39 | 2523 ±93 | 0.74 ±0.07 | 18.5 |
| $P\text{-}PS_{10}$ | 10 | 33.1 ±1.95 | 67.8 ±0.98 | 2985 ±81 | 0.45 ±0.09 | 11.25 |
| $P\text{-}PS_{15}$ | 15 | 25.2 ±0.48 | 47.3 ±0.47 | 3374 ±105 | 0.29 ±0.05 | 7.25 |
| $P\text{-}PS_{20}$ | 20 | 14.4 ±2.06 | 45.7 ±0.31 | 3678 ±64 | 0.24 ±0.08 | 6.00 |
| $P\text{-}PS_{25}$ | 25 | 07.5 ±1.49 | 41.2 ±1.86 | 3769 ±79 | 0.18 ±0.06 | 4.50 |

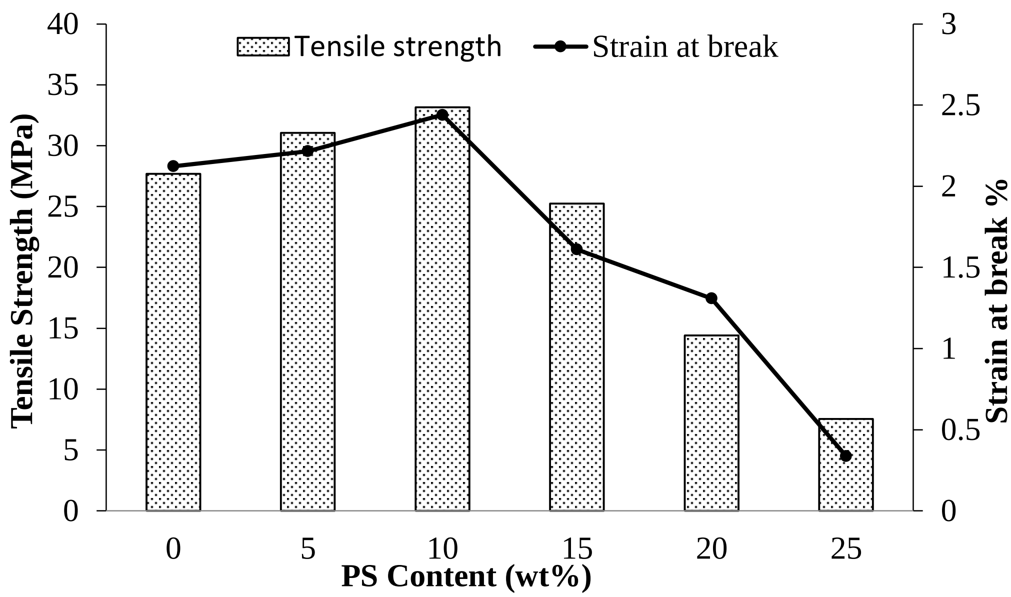


**Figure 5.5** Pistachio shell particle content versus tensile strength and strain at break.

### 5.2.2 Three-Point Bending Test Results

As seen at Figure 5.6, the maximum force that occurs at fracture and associates with flexural strength and the strength values increase until PS weight fraction of 5% of the total mixture, after that these values drop. This decrease was very close between the weight fraction of 20% and 25%.

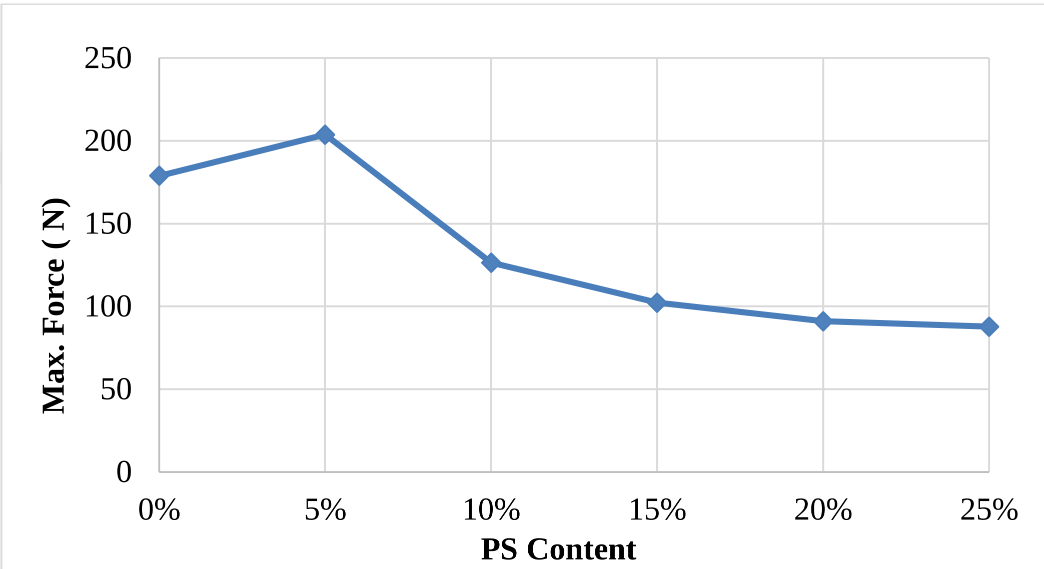


**Figure 5.6** Maximum force versus PS Content

To evaluate the bending loading performance, the flexural properties were acquired from the three-point bending test of PS/polyester composite specimens, the stress-strain curves (Figure 5.7) were indicate that the stress-strain curves followed linear behavior up to failure stress (flexural strength) and failed suddenly at highest stress this behavior attributed to the brittle nature of the composite matrix. Besides, the shape of the broken specimens in Figure 5.8 indicates this behavior.

In general, the flexural strain at break was decreased with inclusion PS particles due to the rigid particles can restrict the mobility and deformation of the polyester matrix.

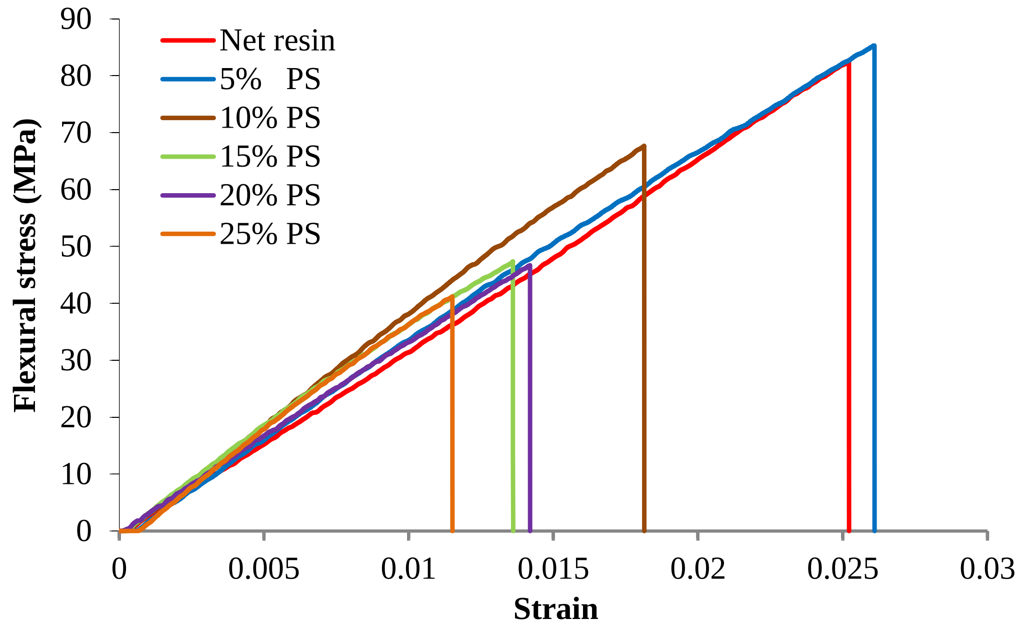


**Figure 5.7** Flexural stress-strain curves of the PS/polyester composites.

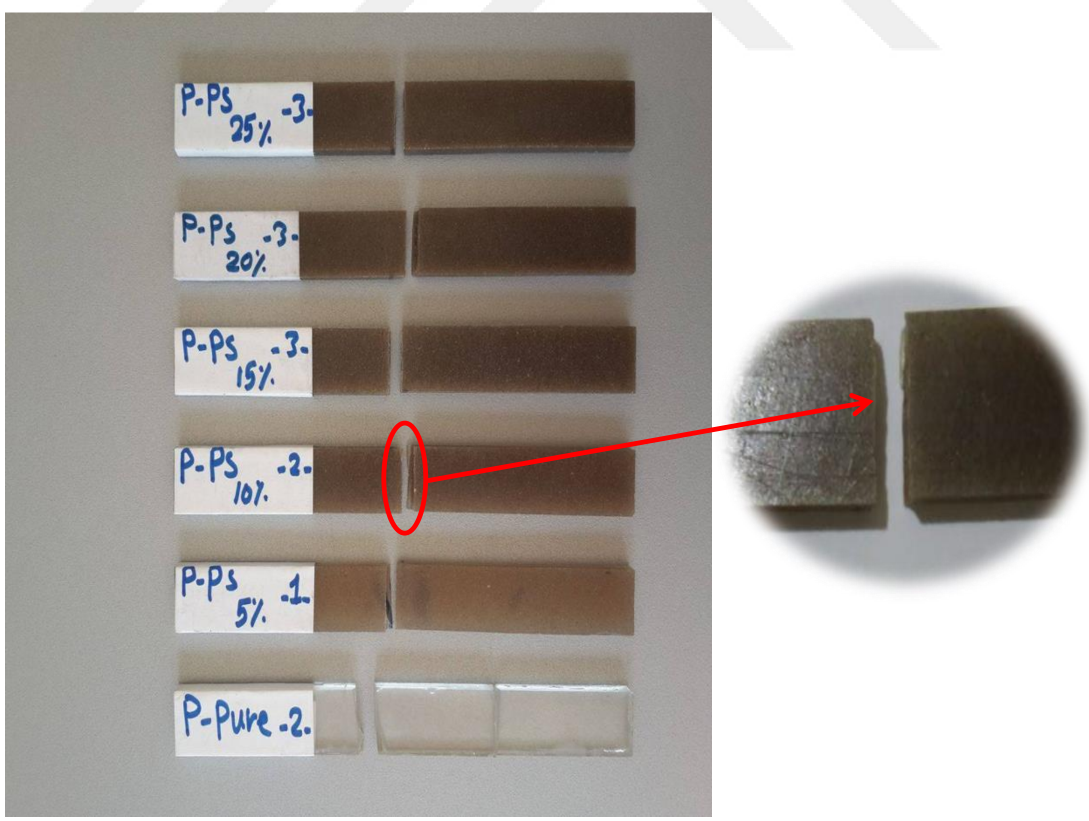


**Figure 5.8** The failed specimens of the three-point bending test of the PS/polyester composites.

As seen in the Figure 5.9 and Table 5.1, the flexural strength were significantly influenced by the addition of PS particles. The flexural strength was slightly improved about 4.0% with PS inclusion at 5 wt% and then followed the trend of decreasing. This behaviour as discussed in SEM section was ascribed to particle aggregation phenomena resulting in a particle-particle interaction led to weak in particle/matrix interfacial bonding [121]. On the other hand, this parameter does not influence the modulus since for a little loads or displacements, the interfacial bonding of particle/matrix is not yet observed. The flexural modulus was greatly improved by the incorporation of particles in the polymer matrix due to rigid particles have much higher stiffness than the polymer matrix, the same behavior was observed by [7] for olive nuts flour filled unsaturated polyester composite. Furthermore, the maximum improvement for the flexural modulus was obtained at PS inclusion of 25 wt% with the highest increment of 62.7%.

As listed by the results of the three-point bending test, as the weight rate of PS content in the composite increase, the flexural strength values increases at 5%. After that, the results showed decreasing in flexural strength after adding pistachio shell powder into the mixture; also, this drop in strength is nearly constant after 20 wt%.

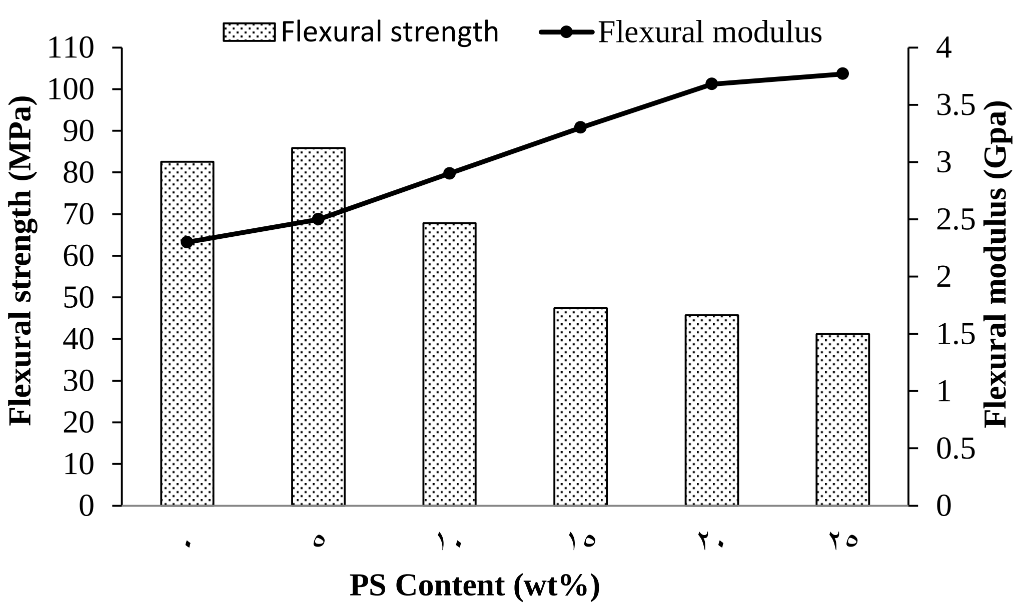


**Figure 5.9** Pistachio shell particle content versus flexural strength and modulus.

### 5.2.3 Charpy Impact Test Results

Charpy impact test was utilized to discover the impact energy absorbed by the specimens. As shown in Figure 5.10, the impact strength and absorbed energy firstly increase at PS content of 5 wt% then followed the trend of decreasing up to final PS content of 25 wt%. Therefore, the highest impact strength was 18.5 (KJ/$m^2$) compared to 8.25 (KJ/$m^2$) for unfilled polyester with increment about 124%. This behavior indicated that the impact strength was important influenced by the inclusion of PS particle. These results were affected by the adhesion bonding strength between particle and matrix, hence the best adhesion bonding occurred for PS content in a range of 5-10 wt%.

The failed specimens of charpy impact test of the PS/polyester composites for various particle contents are illustrated in Figure 5.11.

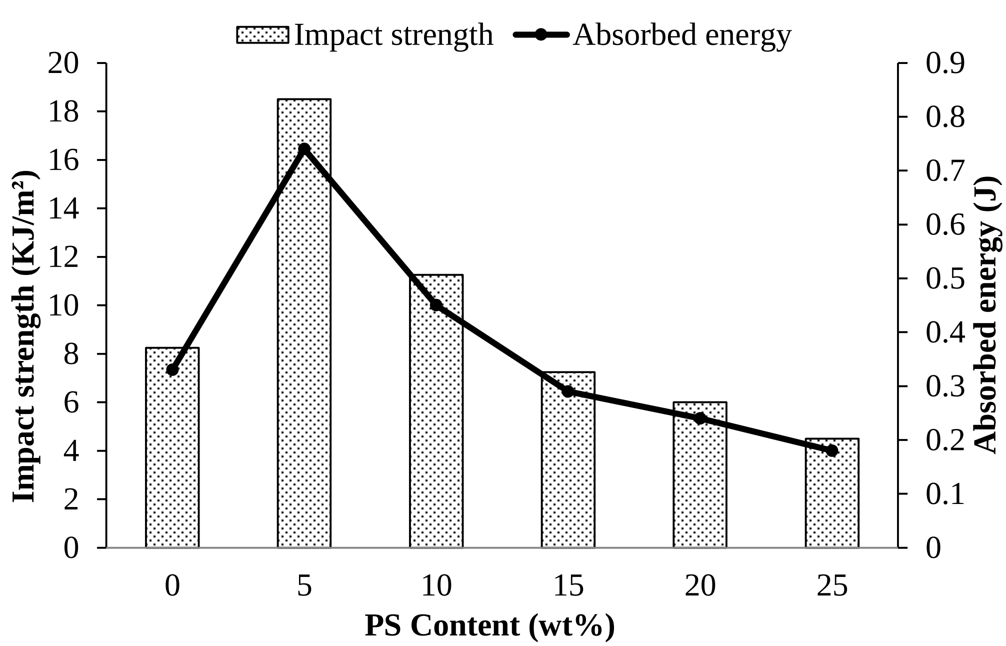


**Figure 5.10** Impact strength and absorbed energy versus PS content.

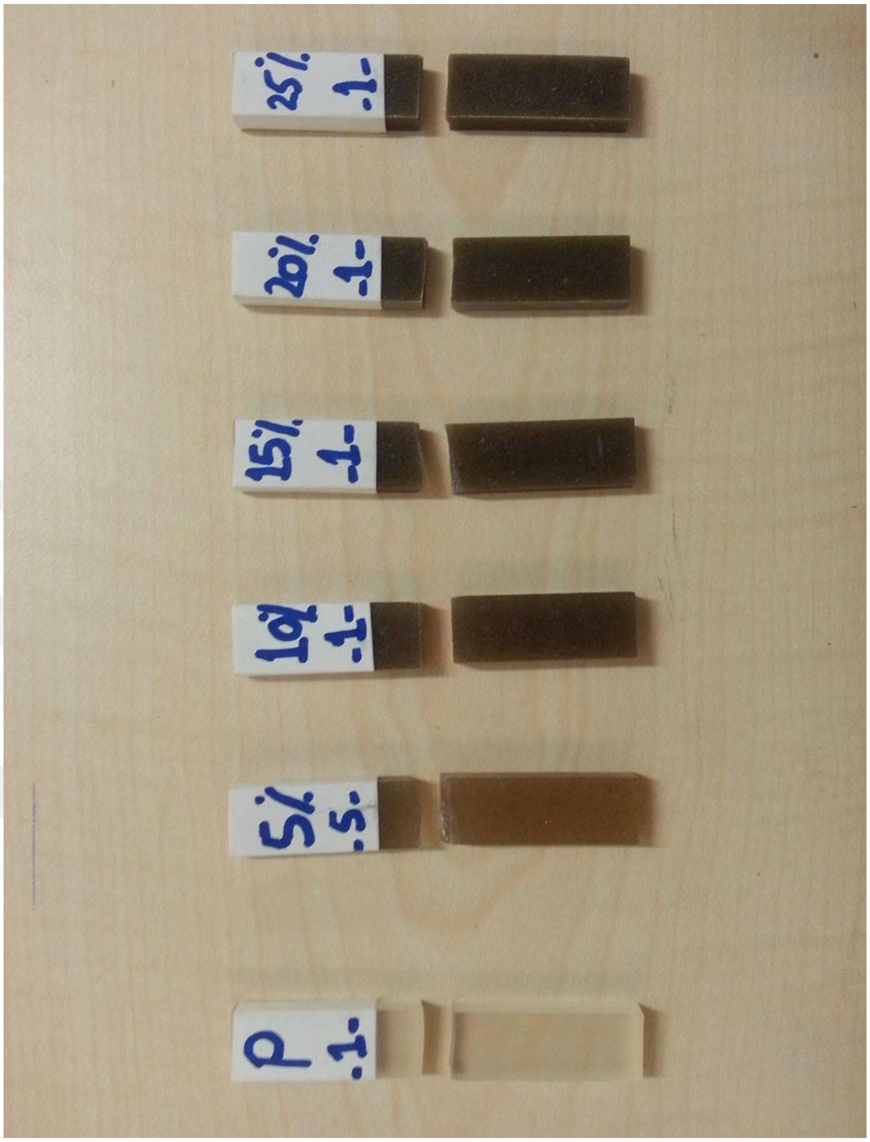

**Figure 5.11** The failed specimens of charpy impact test of the PS/polyester composites.

# CHAPTER 6

# CONCLUSION

In this study, the micro-scale pistachio shell powder is incorporated with various particle contents within polyester matrix. Tensile, three-point bending and charpy impact tests were applied on PS/polyester composite specimens in order to estimate the mechanical behavior of the particulate polymer composites. The following conclusions are obtained from the present study:

1. From the results of three different tests, the addition of pistachio shell powder noticeably effects the mechanical properties of polyester matrix composites.
2. The SEM images indicated a good distribution of pistachio shell particles was obtained for PS content of 5 and 10 wt%. Also, particle aggregation occurred at content above~10 wt%.
3. For tensile test results, the adding of pistachio shell particle improved the tensile strength up to PS content of 10 wt%.
4. For three-point bending test results, maximum flexural strength obtained at PS content of 5 wt%, then the followed the trend of decreasing. On the other side, the flexural modulus was gradually enhanced up to PS content of 25 wt%.
5. Finally, for Charpy impact test result, impact strength and absorbed energy reached a maximum value at PS content of 5 wt% and then decreased.
6. Even though some loss in mechanical properties can be found at high particle content, fabricating environmentally friendly and low price particulate polymer composite can be regarded as benefits of using pistachio shell as particulate filler.

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