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M.Sc. Thesis in Mechanical Engineering

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UNIVERSITY OF GAZÍANTEP GRADUATE SCHOOL OF NATURAL & APPLIED SCIENCE

A RESEARCH ON EFFECT OF SEWAGE SLUDGE ASH ON THE MECHANICAL PROPERTIES OF COMPOSITE MATERIAL

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Supervisor

Assoc. Prof. Dr. M. Akif KÜTÜK

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ABSTRACT

A RESEARCH ON EFFECT OF SEWAGE SLUDGE ASH ON THE MECHANICAL PROPERTIES OF COMPOSITE MATERIAL

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In the current study, effect of sewage sludge ash on the mechanical properties of particle filled composite material is investigated experimentally. The weight rate of sewage sludge ash which would be applied tensile test, three point bending test, impact test is changing between 0 % and 45 % of overall mixture. Tests are made in mechanics laboratory and for tensile test and three point bending test, the speed of test machine is adjusted as 1 mm/min and 2 mm/min respectively. According to the result of three point bending test, maximum stress tends to decrease with increased rate of sewage sludge ash in the composite. According to the result of tensile test, maximum stress tends to increase with the sewage sludge ash particles content up to 20 % ratio of overall mixture and beyond that ratio it tends to decrease. According to result of impact test; impact resistance increase until weight rate of ash reaches 10 %. The impact resistance decrease between the weight rate of ash 10 % and 25 % and then impact resistance remains constant until rate of ash reach 40 %.

By help of these results it can be said that weight rate of sewage sludge ash as 10 % is ideal ratio for usage of this type waste as additive in composite. However, it shouldn't be forgotten that addition of ash affects bending character negatively during usage of sewage sludge ash.

Keywords: Sewage sludge ash, mechanical properties, particle filled composite

ÖZET

ATIKSU ARITMA ÇAMUR KÜLÜ KATKISININ KOMPOZIT MALZEME MEKANIK ÖZELLIĞINE ETKISI ÜZERINE ARAŞTIRMA

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56 Sayfa

Bu çalışmada, atık su arıtma çamur külü katkısının partikül takviyeli kompozit malzemelerin mekanik özellikleri üzerine etkisi deneysel olarak incelenmiştir. Çekme, üç noktadan basma, darbe testleri uygulanmak üzere hazırlanan karışımlarda atık su arıtma çamur külü oranı ağırlıkça tüm karışımın % 0 ile % 45'i arasında değişmektedir. Testler laboratuarda yapılmış, çekme testi ve üç noktadan basma testi için test makinesinin hızı sırasıyla 1 mm/dak. ve 2 mm/dak. olarak ayarlanmıştır. Üç noktadan eğme testi sonuçlarına göre, kompozitte atık su arıtma çamur külü eklenmesi arttıkça maksimum stres azalmaya meyil eder. Çekme testi sonuçlarına göre, maksimum gerilme, atık su arıtma çamur külü partikülleri tüm karışımın % 20 oranına kadar artmaya meyil eder, daha yukarı oranlarda azalış eğilimi gösterir. Darbe testi sonuçlarına göre; darbe direnci, kül oranı ağırlıkça %10'a ulaşana kadar kül eklenmesiyle artar. Ağırlıkça kül oranı % 10-% 25 aralığında darbe direnci düşer ve daha sonra darbe direnci, kül oranı ağırlıkça % 40' a ulaşıncaya kadar sabit kalır.

Bu sonuçların yardımıyla şu söylenebilir ki ağırlıkça % 10 oranı olarak atık su arıtma çamur külü kullanımı kompozitlerde bu tarz atığın katkı olarak kullanımında ideal bir orandır. Ancak, atık su arıtma çamur külünün kullanımı esnasında kül eklenmesinin eğilme karakterini negatif etkilediği unutulmamalıdır.

Anahtar Kelimeler: Atık su arıtma çamur külü, mekanik özellikler, partikül takviyeli kompozit



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Before this study, used sewage sludge ash that is a product of an incineration system which has been established in Gaziantep and in TURKEY firstly, remain in waste state. This ash is an environmental problem for Gaziantep and also for Turkey and world if it is considered that the establishing of system is enlarged over the world.

Topic of this thesis is observing the effect of sewage sludge ash on the composite material and by this way to find a way to reuse of this ash in industry. Because if the reusing of sewage sludge ash is achieved, it is not only an environmental problem will be overcome but also it will contribute the national economy of Turkey in a positive side. Furthermore, by reusing of this waste a new and different approach will be brought for academic studies.

By using of this thesis, the studies about sewage sludge ash will continue in future.

I am deeply indebted to Assoc. Prof. Dr. M. Akif KÜTÜK, who has never been false in his advices through this master thesis. He has patiently supervised my studies and has shared his experiences.

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CONTENTS

ABSTRACT	v
ÖZET	vi
ACKNOWLEDGEMENTS	viii
CONTENTS	ix
LIST OF FIGURES	xii
LIST OF TABLES	xiv
CHAPTER 1	1
INTRODUCTION	1
1.1. Outline of the Study	1
1.2. Background	1
CHAPTER 2	
LITERATURE REVIEW	
2.1 Studies on Particle Filled Composites	7
2.2 Purpose of Thesis	
CHAPTER 3	
COMPOSITE MATERIALS	9
3.1 Definition of Composites	
3.2 Advantages and Disadvantages of Composites	
3.3 Application Areas of Composites	
3.3.1 Aerospace Industry	12
3.3.2 Automotive Industry	
3.3.3 Sports Industry	
3.3.4 Marine Industry	14
3.3.5 Health Industry	14
3.3.6 Corrosion Resistant Products	14
3.3.7 Consumer Goods Industry	
3.4 Classification of Composites	15
3.4.1 Matrix Composites	
3.4.1.1 Metal Matrix Composites	
3.4.1.2 Ceramic Matrix Composites	16
3.4.1.3 Polymer Matrix Composites	16

3.4.2 Reinforced Composites	17
3.4.2.1 Particle-Reinforced Composites	18
3.4.2.2 Laminated Composites	19
3.4.2.3 Fiber Reinforced Composites	19
3.5 Manufacturing Techniques of Composite Materials	20
3.5.1 Hand Lay-Up Method	21
3.5.2 Spraying Method	22
3.5.3 Vacuum Bagging Molding Method	22
3.5.4 Vacuum Assisted Resin Infusion Molding	23
3.5.5 Filament Winding Method	23
3.5.6 Resin Transfer Molding (RTM) Method	25
3.5.7 Structural Resin Injection Molding Method (SRIM)	26
3.5.8 Autoclave Curing	26
3.5.9 Pultrusion Method	27
3.5.10 Continuous Laminating Method	28
3.6 Types of Tests are being Applied Composite Materials	29
3.6.1 Impact Test	29
3.6.1.1 Charpy Impact Test	29
3.6.1.2 Izod Impact Test	30
3.6.1.3 Drop Weight Test	30
3.6.1.4 Gas Gun Test	31
3.6.2 Tensile Test	31
3.6.3 Three Point Bending Test	32
CHAPTER 4	33
EXPERIMENTAL METHOD	33
4.1 Polyester Resin	33
4.2 Hardener (Methyl Ethyl Ketone Peroxide - MEKP)	33
4.3 Sewage Sludge Ash	34
4.4 Stirring of Materials and Preparation of Specimens	36
4.5 Mechanical Test	37
4.5.1 Tensile Test	37
4.5.2 Three Point Bending Test	38
4.5.3 Impact Test	39
CHAPTER 5	41
RESULTS	41
5.1 Test Results	41

5.1.1 Tensile Test	41
5.1.2 Three Point Bending Test	44
5.1.3 Impact Test	46
CHAPTER 6	49
DISCUSSION AND CONCLUSION	49
REFERENCES	51

LIST OF FIGURES

Figure 3.1 : A car produced by carbon, kevlar and glass fibers	9
Figure 3.2 : Use of composite materials in different bodies of an aircraft	9
Figure 3.3 : Production of adobe material	
Figure 3.4 : Straw mixture of clay in the mud	10
Figure 3.5 : A car dashboard of GM automobile	13
Figure 3.6: Tennis rocket, fishing rod and some sports material which made	
by composite	14
Figure 3.7 : Prosthetic hand and legs made by composite	14
Figure 3.8 : Classification of composite materials	18
Figure 3.9: The most common use of reinforcement members composites;	
a)fiber reinforced, b)particle reinforced, c)laminates, d)filling composite	18
Figure 3.10 : Schematic representation of laminated composite	19
Figure 3.11: Schematic designation of continuous fiber reinforced	20
Figure 3.12: Fiber reinforced composite material types according to the shape	
(a)continuous fiber, (b)discontinuous fiber, (c)random oriented	20
Figure 3.13: Composite production by hand lay-up method	22
Figure 3.14: An illustration of spraying method and the spray gun	22
Figure 3.15 : Schematic illustration of vacuum bag molding	23
Figure 3.16: Schematic illustration of vacuum infusion process	23
Figure 3.17 : Schematic illustration of filament winding	25
Figure 3.18 : Schematic illustration of RTM	26
Figure 3.19 : A basic schematic illustration of RTM	26
Figure 3.20: A vacuum bagged composite aircraft part ready to enter Autoclave	27
Figure 3.21 : Schematic illustration of pultrusion	28
Figure 3.22 : Schematic designation of continuous laminating	28
Figure 3.23 : Schematic Illustration of Charpy Impact Test	30
Figure 3.24 : Schematic Illustration of Izod Impact Test	30
Figure 3.25 : Schematic Illustration of Drop Weight Test	31
Figure 3.26 : A Gas Gun Test mechanism	31
Figure 3.27 : A Typical Tensile Test Machine	32
Figure 3.28 : A Basic Illustration of Bending Test Types	32

Figure 4.1 : Beginning of Mixture	36
Figure 4.2 : Reaction of polyester and hardener	36
Figure 4.3: Universal Test Machine For Tensile Test	37
Figure 4.4: Tensile Test Specimens	37
Figure 4.5: Three Point Bending Test Machine	38
Figure 4.6: Three Point Bending Test Samples	38
Figure 4.7: The Schematic Designation of Impact Test Machine	40
Figure 4.8 : Impact Test Specimen	40
Figure 4.9: Impact Test Machine	40
Figure 5.1: Maximum Force - Specimen Diagram for Tensile Test	42
Figure 5.2 : Tensile Stress - Specimen Diagram for Tensile Test	42
Figure 5.3 : Stress - Strain Graph for Tensile Test	43
Figure 5.4 : Typical Failed Specimen for Tensile Test	43
Figure 5.5: Maximum Force - Specimen Diagram for 3 Point Bending Test	44
Figure 5.6 : Stress - Specimen Diagram for 3 Point Bending Test	44
Figure 5.7 : Stress - Strain Graph for 3 Point Bending Test	45
Figure 5.8: Typical Failed Specimen for 3 Point Bending Test	45
Figure 5.9: Impact Test, Absorbed Energy (J) - Specimen Diagram	47
Figure 5.10: Impact Test, Impact Resistance (J/m²) - Specimen Diagram	47
Figure 5.11: Typical Failed Specimen for Impact Test	48

LIST OF TABLES

Table 3.1: The mechanical properties of metallic and composite materials	12
Table 3.2: The rate of matrix used in automobiles between 1988 and 1993	13
Table 3.3 :The physical and mechanical properties of polymeric matrix	
materials	17
Table 3.4: The data of composite manufacturing techniques in World,	
Europe and Turkey	21
Table 4.1: Compound within ash and their percentage	34
Table 4.2 : Weight Rate of Ingredient of Tensile Test,3 Point Bending	
Test and Impact Test Specimens	35
Table 4.3: The chemical compounds of fly ash obtained from thermal	
power plants, in Turkey and Chemical compound of sewage sludge ash	36
Table 5.1: Test Results for Tensile and 3 Point Bending Test	46
Table 5.2 : Test Results for Impact Test	47

CHAPTER 1

INTRODUCTION

1.1 Outline of the Study

This thesis is organized into 5 chapters. In **first chapter** of thesis a small background about composite materials will be given and it will be introduction of thesis. In **chapter two**, literature review and purpose of thesis will be given. **Chapter three** will be concern about definition of composites and their advantages & disadvantages, application areas of composites, classification of composites, manufacturing techniques of composite materials, types of tests those are applied to composite materials. Each of these will be detailed also. In **chapter four** discussed parameters will be polyester resin, hardener (Methyl Ethyl Ketone Peroxide-MEKP), sewage sludge ash, stirring of materials, mechanical tests (tensile test, three point bending test, impact test). **Chapter 5** contains conclusion of experimental results and **chapter 6** contains conclusion and discussion.

1.2 Background

Composite materials, plastics, and ceramics have been the dominated emerging materials over the last forty years. The volume and number of applications of composite materials has grown steadily, penetrating and conquering new markets relentlessly [1].

A Composite material is a material system composed of two or more macro constituents that differ in shape and chemical composition and which are insoluble in each other. The history of composite materials dates back to early 20th century. In 1940, fiber glass was first used to reinforce epoxy.

A composite material can provide superior and unique mechanical and physical properties because it combines the most desirable properties of its constituents while suppressing their least desirable properties. At present composite materials play a key role in aerospace industry, automobile industry and other engineering

applications as they exhibit outstanding strength to weight and modulus to weight ratio. Composite materials offer diverse design requirements with significant weight savings as well as high strength-to-weight ratio as compared to conventional materials. Some advantages of composite materials over conventional one are mentioned below [2]:

- Tensile strength of composites is four to six times greater than that of traditional steel or aluminum,
- Improved torsional stiffness and impact properties,
- Higher fatigue endurance limit (up to 60% of the ultimate tensile strength),
- 30-45 % lighter than aluminum structures designed to the same functional requirements,
- Lower embedded energy compared to other structural materials like steel, aluminum etc.
- Composites are less noisy while in operation and provide lower vibration transmission than metals,
- Composites are more versatile than metals and can be tailored to meet performance needs and complex design requirements,
- Long life offers excellent fatigue, impact, environmental resistance and reduced maintenance,
- Composites exhibit excellent corrosion resistance and fire retardancy,
- Composite parts can eliminate joints/fasteners, providing part simplification and integrated design compared to conventional metallic parts.

There are several types of composite materials. Traditional examples are reinforced concrete, thermoplastics reinforced with short fibers, honeycomb composites, sandwich panels. Some of the most common types of composites are those made of layers, each of these layers is composed of long fibers embedded in a resin. Using various approaches (e.g., mixture theory), each layer can be represented at a macroscale as an orthotropic material [2].

CHAPTER 2

LITERATURE REVIEW

In the literature survey, some investigation is observed about effects of different fillers on mechanical, thermal and some other properties of composite material. Present study is about effect of particles on the mechanical properties of composite material hence similar studies in literature are investigated prior to any work. Although composites metal oxides are preferred in general in studies to obtain particle reinforced composites, fly ash is used in some studies. The closest studies to current study are summarized below.

2.1 Studies on Particle Filled Composites

PATNAIK et al. [3] studied a series of glass fiber reinforced polyester composites that are fabricated using fly ash, aluminum oxide (Al₂O₃) and silicon carbide (SiC) particles as filler materials. The effects of these three different ceramics on the mechanical properties of glass-polyester composites are investigated. It has been found that the incorporation of these fillers, the tensile strength of the composites decrease significantly. The flexural properties, inter laminar shear strength, density and hardness are also affected by the type and content of filler particles. It is found that the presence of SiC improves the hardness of the glass-polyester composites, whereas the other two fillers show marginal effect. The study reveals that the reduction in tensile strength is the minimum in case of fly ash among all the fillers. Further, the composite with low fly ash content (10 wt %) exhibits improved flexural strength. It is thus interesting to find that an industrial waste-like fly ash shows better filler characteristics compared to those of alumina and SiC. Moreover, being cheap and easily available, it would hopefully provide a cost effective solution to composite manufacturers.

RAJU et al. [4] studied the mechanical and two-body abrasive wear behavior of alumina (Al₂O₃) filled glass fabric reinforced epoxy (G-E) composites. From the

experimental investigation, it was found that the presence of Al₂O₃ filler improved the tensile strength and tensile modulus of the G-E composite. Inclusion of Al₂O₃ filler reduced the specific wear rate of G-E composite. The results show that in abrasion mode, as the filler loading increases the wear volume decreases and increased with increasing abrading distance. The excellent wear resistance was obtained for Al₂O₃ filled G-E composites. Furthermore, 10 wt % filler loading gave a very less wear loss.

RUSU et al. [6] investigated the mechanical and thermal properties of high density polyethylene/zinc powder composites. According to their study, the composites present poorer mechanical properties as compared to the unfilled polymer. The density and hardness of HDPE/zinc composites are higher than that for the unfilled polymer. The thermal stability of the HDPE charged with zinc powder is better that for the unfilled polymer. The incorporation of zinc powder in HDPE increases the thermal diffusivity and conductivity and decreases the specific heat.

GÜNGÖR [7] investigated the mechanical properties of high density polyethylene (HDPE) and HDPE containing Fe polymer composites experimentally. As compared to the mechanical properties of unfilled HDPE, Fe filled polymer composites showed lower yield and tensile strength, % elongation, and Izod impact strength, while the modulus of elasticity and hardness of the composites were higher than those of HDPE. Addition of 5 vol. % Fe reduced Izod impact strength and % elongation of HDPE about 40 % and 90 % respectively and increased the modulus of elasticity of HDPE about 31 %.

SAYER [8] studied the effects of the various ceramic particles on elastic properties and load carrying capabilities of filled E-glass/epoxy composite plates. Effects of particles on mechanical properties were determined experimentally and numerically. The composite plates are filled with 0 % (unfilled), 5 %, 10 % and 15 % particle weight fractions (based on the weight of composite), such as silicon carbide (SiC), which has two particle sizes, aluminum oxide (Al₂O₃) and boron carbide (B₄C). The results indicated that the load carrying capability of composites are influenced by particle weight fractions, different particle sizes and different ceramic particles (fillers). Accordingly, the load carrying capabilities of composites filled with 10 wt % ceramic particles were found higher for small particle sizes. Moreover, the

addition of 10 wt % boron carbide (B₄C) particles to composites increases the critical buckling load value of composite up to 42 %.

ASI [9] has carried out to investigate the mechanical properties of glass-fiber reinforced epoxy composite filled with different proportions of Al₂O₃ particles. As a comparison, the mechanical properties of unfilled glass-fiber reinforced epoxy composite were also evaluated under identical test conditions. The results showed that while ultimate tensile strength and shear strength of the composites decreased with increasing Al₂O₃ particles content, flexural strength increased with the Al₂O₃ particles content of up to 10 % beyond which it decreased. Compared with the flexural properties of the unfilled glass-fiber reinforced epoxy composite, with the addition of 10 wt % of Al₂O₃ particle in the matrix, flexural strength, and flexural modulus were increased by 33 and 78 %, respectively.

The effects of filler loading on the flexural and physical properties of natural-filled thermoplastic polymer composite panels were investigated by Zabihzadeh et al. [10]. Test results showed that the modulus of elasticity and flexural strength were mainly influenced by the filler content, and the flexural strength was shown to decrease with an increase in the filler content, while the flexural modulus increased in significance.

GULSOY et al.[11] investigated the physical and mechanical properties of Polypropylene (PP) and Fe-PP polymer composites containing 5, 10, and 15 volume % Fe experimentally. After preparing PP and Fe-PP polymer composites with a twin screw extruder and injection molding, the following properties were determined: yield and tensile strength, the modulus of elasticity, % elongation, hardness (Shore D), Izod impact strength (notched), melt flow index (MFI), vicat softening point, Heat deflection temperature (HDT), and melting temperature (Tm) of PP and metal-polymer composites. As compared to PP, it was found that by increasing the volume % of Fe in PP, notched Izod impact strength, yield and tensile strength, and % elongation decreased. On the other hand, the modulus of elasticity, hardness, MFI, vicat softening point, and HDT values increased with the amount of iron.

The influences of filler size and content on the properties (thermal conductivity, impact strength and tensile strength) of Al₂O₃/high density polyethylene (HDPE) composites were studied by Zhang et al. [12].Thermal conductivity and tensile

strength of the composites increase with the decrease of particle size. The dependence of impact strength on the particle size is more complicated. The SEM micrographs of the fracture surface show that Al₂O₃ with small particle size is generally more efficient for the enhancement of the impact strength, while the 100 nm particles prone to aggregation due to their high surface energy deteriorate the impact strength. Composite filled with Al₂O₃ of 0.5 µm at content of 25 volume % show the best synthetic properties. It is suggested that the addition of nano - Al₂O₃ to HDPE would lead to good performance once suitably dispersed.

SETSUDA et al. [62] studied the effects of fly ash in composites fabricated by injection molding. The results reveal that the content of fly ash is highly significant and contributive to the shrinkage ratio and bending strength. The shrinkage ratio, bending strength and flexural modulus of LLDPE composites containing raw fly ash were found to improve. The shrinkage ratio and flexural modulus of PP composites containing ground fly ash were also found to improve.

SATAPATHY et al. [63] studied the mechanical, dynamic mechanical, and thermal characterization of fly ash and nanostructured fly ash-waste polyethylene/high-density polyethylene blend composites. An increase in storage/loss moduli with enhanced thermal stability was observed with the addition of FA/NFA and upon modifications. The analysis of the tensile fractured surfaces by scanning electron microscopy was in well correlation with the mechanical properties obtained.

GU et al. [64] studied the preparation and damping properties of fly ash filled epoxy composites. They found that the addition of fly ash certainly enhances the damping capacity of epoxy resin, the decomposition temperatures ascend with the increase of volume fraction of fly ash, which indicates that the heat resistance of the matrix can be optimized by controlling the fly ash content and the degree of dispersion of fly ash particulates in the matrix is good, and the fly ash particulates with surface treatment are found to have a good interfacial adhesion with the matrix.

SUDARSHAN [65] studied the synthesis of fly ash particle reinforced A356 Al composites and their characterization. In this study narrow size range (53–106 μ m) and wide size range (0.5–400 μ m) fly ash particles were used. Additions of fly ash lead to increase in hardness, elastic modulus and 0.2% proof stress. Composites

reinforced with narrow size range fly ash particle exhibit superior mechanical properties compared to composites with wide size range particles. A356 Al–fly ash MMCs were found to exhibit improved damping capacity when compared to unreinforced alloy at ambient temperature.

RAJA et al. [66] studied on mechanical properties of fly ash impregnated glass fiber reinforced polymer composites using mixture design analysis. From the study, it was observed that the addition of fly ash fillers enhanced the mechanical properties of FRP such as tensile strength, compressive strength, hardness and impact strength. Particularly the 10 wt% of fly ash fillers in the FRP (70 wt% resin +20 wt% fiber) yielded better results due to good adhesion.

As a conclusion on the summarized literature, the effect of fillers on the material can be clearly seen. Both type of filler and weight rate / volume rate of additive materials affect almost all properties of the composite material. Type of additives may affect the properties of base materials positively or negatively. This result can be obtained with different weight rate / volume rate of ash (additive) or different filler type.

2.2 Purpose of Thesis

In the literature, there can be seen that most material has been used either self state (Al, Cu, etc.) or has been used with compound state (SiC, Al₂O₃) as additive for composite materials. In this study, apart from literature sewage sludge ash that contains Al₂O₃, BaO, Br, CaO, Cl, Cr₂O₃, CuO, Fe₂O₃, K₂O, MgO, MnO₂, Na₂O, NiO, P₂O₅, ZrO₂, ZnO, TiO₂, SrO, SiO₂, SO₃, PbO with different percentage is used.

Population growth and urbanization increase waste water in our country as all over world; this increase also affects the number of waste water purification plants. The increase of plants cause to increase sewage sludge ash (SSA). SSA is stored, used as fill material, discharged to sea or used as agricultural material. However in any method aside from burning problems occur in time. For this reason incineration of SSA is preferred in the leading European countries and other developed countries. By this way while the major source of environmental problem is eliminated both energy is produced and a harmless waste generated as ash. SSA contains above compound as oxide by not harmful to the environment. The annual amount of sewage is 3.5 billon m³ in Turkey [13]. When the 4 percentage of this amount is

considered as waste sedimentary, 140 million tons SSA potential is available in Turkey. SSA incineration system was first developed in Gaziantep [14]. System generates electric by burning approximately 150 tons SSA per day. About 15 tons ash remain at the end of combustion. When the established mechanism is thought as a recycling system the use of remain ash increase the value of mechanism and also use of ash can be regarded as a versatile earning due to its environmental problem.

Evaluation of the SSA ash in composite materials has been examined in the present study. For this purpose effect of sewage sludge ash on the mechanical properties of composite material is aimed to be observed. Tensile test, three point bending test, impact test are applied on the specimen with different proportions of SSA.

CHAPTER 3

COMPOSITE MATERIALS

3.1 Definition of Composites

Composite materials are made from two or more constituent materials with significantly different physical or chemical properties, that when combined, produce a material with different characteristics from the individual components. The individual components remain separate and distinct within the finished structure. The new material may be preferred for many reasons: common examples include materials which are stronger, lighter or less expensive when compared to traditional materials.

Although; the car seen in figure 3.1 has been produced carbon, kevlar and glass fibers for obtaining minimum stiffness and maximum lightweight, the different structures of composite materials were used in the construction of aircraft (Figure 3.2) [15].



Figure 3.1 : A car produced by carbon, kevlar and glass fibers [15]

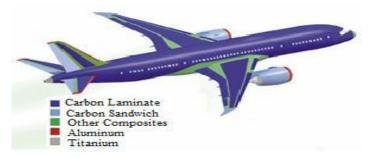


Figure 3.2 : Use of composite materials in different bodies of an aircraft [16]

The first example of composite materials was found in nature. Since the earliest ages, people used the vegetable or animal fibers in brittle materials to eliminate the fragility feature of brittle materials. One of the best examples of this is adobe material. In the production of adobe material; clay-mud, straw, vine-like branches, stalks and fibers were used to increase the strength of adobe structure (Figure 3.3-3.4).



Figure 3.3: Production of adobe material [15]



Figure 3.4 : Straw mixture of clay in the mud [15]

3.2 Advantages and Disadvantages of Composites

Composite materials bring a significant advantage by collecting the original characteristics of the contained material. Today, composite materials are produced in order to ensure adaptation with outstanding features include the difficult working conditions. Superior features expected from a material generally is as below;

- High strength,
- High rigidity,
- High fatigue resistance,
- Excellent wear resistance,

- High temperature capability,
- High corrosion resistance,
- High thermal conductivity,
- Formability,
- Low density,
- They reduced the number of complex parts because of the part produced as a single. Thus, the reduction in production time is shortened.

All of features mentioned above in a composite material can be obtained if it is regarded to suitable matrix/reinforcement element pairs, manufacturing techniques, the strength features of the components and other factors.

The choice of suitable matrix/reinforcing elements is important on the mechanical and physical properties of the system. For a composite material the interface bond between matrix and reinforcement elements should be strong in order to transmit the load from matrix to reinforcing element. This depends on the harmony of pair. Besides, apart from the production technique selection, the homogeneous distribution of the reinforcing elements in the matrix/resin depends on the choice of matrix alloy and reinforcing element [17, 19].

Besides the above-mentioned excellent properties, some disadvantages are also available for composite materials. These are as follows;

- Production difficulties,
- It is expensive,
- There is no recycling for composite material,
- Operating problems,
- High operating costs,
- It is difficult to achieve the required surface roughness,
- The air particles in the polymer based composite materials adversely affects the fatigue properties of material [18, 19, 20].

3.3 Application Areas of Composites

The application areas of composite materials are very wide. Composite materials are preferred for many industries. Therefore, composite materials are not difficult to find

an industry branch. Most extensive usage of composite materials can be seen in the transportation industry. The reason for this is that the composites have good mechanical properties and lightness. The usage areas of composite materials are discussed below:

3.3.1 Aerospace Industry

In recent years; the composite materials are widely used in aerospace industry instead of metallic materials. When comparing to conventional materials, boron/epoxy and carbon/epoxy have superior mechanical properties for the construction of aircraft. The mechanical properties of composite materials and metallic materials are given in Table 3.1 [21].

Table 3.1: The mechanical properties of metallic and composite materials [22,23].

Material	Density	Elastic Specific		Tensile
	(g/m^3)	modulus(GPa)	modulus(E/p)	strength(GPa)
Steel (3140)	7.90	200	25	1.85
Alum.				
ASG(6061)T6	2.70	70	26	0.35
Alum.AU4GI				
(2024)74	2.80	73	26	0.29
Alum.A25G4				
(7075)T6	2.80	76	27	0.45
Titanium T6V	4.40	119	27	1.14
Boron/Epoxy	2.10	270 ^a	129ª	2.00^{a}
Boron/Alum.	2.70	225 ^b	83ª	1.25 ^b
Graphite /Epoxy	1.70	208 ^a -10.3 ^b	122ª	1.34 ^a -0.03 ^b
Carbon/Epoxy	1.50	142 ^a -10.3 ^b	95ª	1.60 ^a -0.07 ^b
Kevlar/Epoxy	1.35	80°-5.5°	59 ^a	1.38 ^a -0.03 ^b
Glass/Epoxy	2.20	53 ^a -12.4 ^b	24ª	1.45 ^a -0.04 ^b
Carbon/Polyester	1.68	127.5 ^a -7.6 ^b	76ª	1.52 ^a -0.04 ^a
Kevlar/Polyester	1.40	76°-5.5°	54ª	1.20 ^a -0.02 ^b
Glass/Polyester	1.80	39 ^a -9.6 ^b	22ª	1.13 ^a -0.02 ^b

a: The direction of fibers and b: the direction of perpendicular to the fibers

Some examples about application of composite materials at aerospace industry are below;

- Airframe panels of B2 type bomber aircraft,
- Wing panels of A380 type passenger aircraft,
- The nose section A380 type passenger aircraft,
- Horizontal stabilizers of F-14 aircraft, horizontal and vertical stabilizers of F15 aircraft [24].

3.3.2 Automotive Industry

In the automotive industry; the composite materials are used for their lightness to save more fuel. The automotive industry uses glass fibers as main reinforcement material. Table 3.2 represents the use of matrix in the automobile industry according to application areas and production methods.

Table 3.2: The rate of matrix used in automobiles between 1988 and 1993 [25]

Applications	Use	Matrix materials	Use	Production	Use
	(10^6kg)		(10^6kg)	method*	(10^6 kg)
Fender	42	Polyester	42	RM	40
Seat	14	Polypropylene	22	RET	20
Hood	13	Polycarbonate/PBT	10	IM	13
Radiator	4	Polyethylene	4	MEP	5
Ceiling	4	Epoxy	4	FW	3
Other	12	Other	7	Other	8
Total	89	Total	89	Total	89

^{*}Where RM is ready molding, RET is reinforced extruded thermoplastic, IM is injection molding, MEP is mould extra power, and FW is fiber winding.

Some examples about application of composite materials at automotive industry are ; pedals, filter box of Mercedes, air intake manifold of Mercedes, Ford, BMW



Figure 3.5 : A car dashboard of GM automobile [24]

3.3.3 Sports Industry

The use of composite materials is very common in the sports and entertainment industry. Composite materials are preferred according to other materials due to their, light weight, strength and vibration characteristics. Composite materials are extensively used in sports industry such as golf clubs, racing boots, tennis rackets.







Figure 3.6: Tennis rocket, fishing rod and some sports material which made by composite [19, 24]

3.3.4 Marine Industry

Composite materials have good corrosion resistance, light weight, and fuel efficiency. These properties provide more speed and more comfortable for marine industry. In general, glass fiber reinforced plastic composites are used in marine industry. The ship, yacht, passenger ferries, sea boats, lifeline etc. are manufactured from composite materials.

Some examples about application of composite materials at marine industry are; pole, the rear platform of yachts, steps [24].

3.3.5 Health Industry

Composite materials used in the health sector are produced in the outer housing of the medical device. Composite materials have become popular vehicle types in the health sector due to designed to be light weight and easily by the human body.





Figure 3.7: Prosthetic hand and legs made by composite [19,24]

3.3.6 Corrosion Resistant Products

Composites used in corrosion-resistant products are generally resist to corrosion of materials. Some examples about application of composite materials at corrosion

resistant products are; water tank, underground pipes, frozen food section coating in stores [24].

3.3.7 Consumer Goods Industry

Composite materials are used for sewing machines, doors, bathtubs, desks, computers, printers, etc. in the consumer goods industry. Most parts of these products are made of short fiber composites. For this kind of products, the ready molding and injection molding methods are used [15].

3.4 Classification of Composites

Although to not be able to draw precise boundaries for the grouping of composite materials that a number of different materials can be used in composite structures, composites can be classified in two groups according materials which formed by and the shape of component. Depending on the type of the base material (plastic composites, metallic composites, ceramic composites, etc.), a grouping can be made according to construction components also according to form of composite (particle composites, fiber-based composites, filled " cage " composites, laminated composites) can be classified. That is composite materials classified into two groups, these are according to matrix and according to reinforcements.

3.4.1 Matrix Composites

There is no significant effect on the load carrying capacity of the composite material for matrix. However, the selection of matrix is a profound effect on sliding properties of the composite material layer and the internal layer. The shear strength between layers is important material to be exposed to bending stress; shear strength inside the layer is important material to be exposed to torsion stress [27].

3.4.1.1 Metal Matrix Composites

The main structure of these composite materials is the metal matrix. The selection of these materials is virtually no limit. Metal matrix composites are increasingly used as an alternative material to traditional metallic materials. Metal matrix composites provide high toughness and strength, high temperature properties and higher heat stability, high corrosion resistance in most environments, higher ductile properties

and can be combined by conventional methods. Due to these superior properties, metal matrix composites are used in automotive, aerospace and defense industry [15, 26]. Some properties about metal matrix composites are below;

- High elastic modulus,
- High strength (tensile, compression, wear, abrasion resistance),
- Operate at higher temperatures,
- They combine the features of ductility and toughness of metals and high strength and high module of ceramic,
- Have reproductible properties,
- They give low density values,
- They show low sensitivity against to temperature changes, or thermal shock,
- High electrical and thermal conductivity properties are available [20,28].

3.4.1.2 Ceramic Matrix Composites

Ceramic materials have high temperature resistant and light weight. Ceramic matrix composites exhibit very low elongation at break, low toughness, and tend to thermal shocks. On the other hand, they have very high elasticity modulus and operating temperatures [29]. These product are mainly used in place of sandwich armors, a wide variety of military structures and space vehicles. Carbon, ceramic and glass fibers are added to ceramic matrix for the special conditions such as high temperature. The strength and toughness of ceramic matrix composites can be increased by added ceramic fibers into ceramic matrix materials [15].

Especially metal-ceramic composite materials are used in both industrial areas and aerospace industry due to the high temperature stability and corrosion resistance. Also they are used in turbine engine parts, hot gas filters, in the heat exchanger tubes, components exposed to corrosion in oil pipelines, heat treatment furnace, the exhaust valves of diesel engine [20].

3.4.1.3 Polymer Matrix Composites

Polymers such as polypropylene, nylon or polyethylene are observed that some important differences when compared to conventional materials. Therefore, polymers are increasingly used day to day. The polymers have several advantages; such as

easily molding, lower density, resistant to chemical influences and corrosion, using an insulator against to heat and electricity. Although, the strength and modulus of polymers are high, the density of them is low per unit weight or volume. So, polymers can be found in the aerospace and aviation industries. Polymer matrix is divided into two groups such as thermosets and thermoplastics. After heated and shaped the conversion of the old structure of thermoset is not possible due to the change in microstructure. Thermoplastics can be dissolved when heated again due to weak bonds in the structure of polymers. This type of plastic is heated to soften and harden when cooled after being shaped. The most important in these polymers are acrylic, cellulose acetate, nylon polyamide, polycarbonate. Epoxy, polyester and vinyl ester are widely used as matrix materials in reinforced composites. Their physical and mechanical properties (Table 3.3) depend on the length of cross link, density and the size of molecules. Mainly application areas of polymeric matrix composites are marine applications, automotive and other transportation industries and sports equipment due to their corrosion resistance and light weight [15].

Table 3.3: The physical and mechanical properties of polymeric matrix materials [15]

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)	<i>35-100</i>	40-90	80
Compression strength (MPa)	<i>100-200</i>	90-250	
Thermal conductivity (W/m°C)	0.1	0.2	
Thermal expansion (°C)	<i>50-300</i>	50-110	
Heat distortion temperature (°C)	<i>50-300</i>	50-110	
Solidifies tensile (%)	1-2	4-8	
Water absorption (%)	0.1-0.4	0.1-0.3	0.150

3.4.2 Reinforced Composites

Essentially a composite material consists of with a matrix and a reinforcement component. Reinforcement material is the main load-bearing element in structure. Different materials are used as the reinforcement and depending upon the use of these composite materials were classified. The structure to be used as a reinforcement material has a great importance due to the importance of determining the physical and mechanical properties of the composite material. Hence the

selection must be done correctly [27]. The classification of composite materials is showed in figure 3.8 and the most common use of reinforcement members composites are showed in figure 3.9 at below.

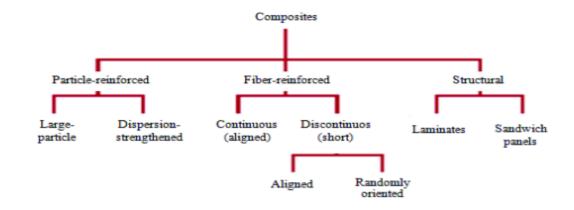


Figure 3.8 : Classification of composite materials [33]

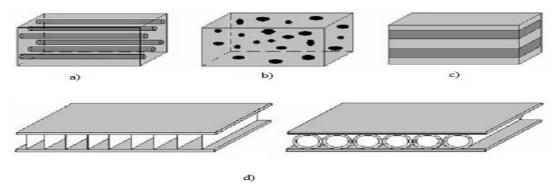


Figure 3.9: The most common use of reinforcement members composites; a) fiber reinforced, b) particle reinforced c) laminates, d) filling composites

3.4.2.1 Particle-Reinforced Composites

If the size is almost the same in every direction, a reinforcement is named as 'particle' [26, 31]. Particle-reinforced composites are obtained by the presence of particles in a matrix material. They are nearly isotropic structures. Strength of the particle-reinforced composites depends on the hardness of the particles. Structures containing ceramic particles are more hardness and have high temperature than the metal matrix. The production of aircraft-engine parts is preferred from this kind of structures. The macroscopic or microscopic particles can be formed in matrix. Besides reinforcement particles have spherical, cubic, uniform or different geometry, they are positioned in random or directed. Directed by strengthening reinforcement particles in a certain direction can be made against the special force [30]. At particle reinforced composite, due to the particles contained in the base material mechanical properties

are improved by dispersion hardening. This is achieved by preventing slippage occurring in the grain boundary of particles [20].

3.4.2.2 Laminated Composites

The laminated composite material which has the oldest and most widely used type. With the combination of different fiber orientation layers, very high strength values are obtained. These structures are resistant to heat and moisture. When compare with metals, they are preferred due to their light weight and high strength features. Continuous fiber reinforced laminated composite materials have a very widespread use in aircraft construction, as a surface coating on the wing and tail group. Additionally, sandwich structure can be given as examples of the laminated composite material that a common use areas of aircraft structures. While many layered composite maintaining high strength or wear resistance with low cost, light weight, they includes improved appearance and excellent thermal expansion characteristics [20, 32].

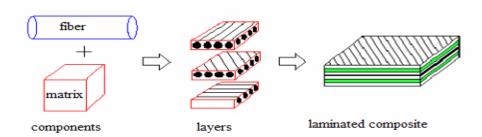


Figure 3.10: Schematic representation of laminated composite [24].

3.4.2.3 Fiber Reinforced Composites

This composite type is occurred by taking place the thin fibers in the matrix structure. Placement of fibers in the matrix is an important phenomena due to affect the strength of the composite materials. The fiber-reinforced composites can be long fibers or short fibers. The long fibers are placed parallel to each other in the matrix, hence, the mechanical properties in the longitudional directions are higher than the transverse directions. The short fibers are distributed homogeneously in the matrix, therefore, the mechanical properties are nearly equal strength in all directions. The strength of fibers is the most important terms of strength of composite structures. Furthermore, by increasing the length/diameter ratio of fibers, the amount of load transmitted to fibers increase. Fiber orientation is an important factor in terms of

strength of the composite. Thus oriented fiber reinforced composites show different characteristics at different direction. Material is accepted as isotropic when fibers distributed within the matrix randomly. In this case composite properties do not change depending the direction. Another important factor in the strength of composite structure is the bond between the fiber and matrix [15, 31].

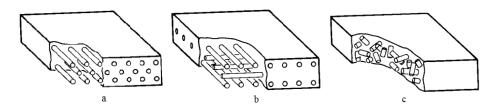


Figure 3.11: Schematic designation of continuous fiber reinforced MMC

a) Orientated in one direction, b) Orientated in two directions with 90°, c) Short fiber reinforced

Fiber reinforced composite materials are produced in different ways by applying different shapes of the fiber placement depending on the expected properties from the material. By using continuous fiber, one direction and two directions location is made by using discontinuous fiber, one direction and random location is made [27].

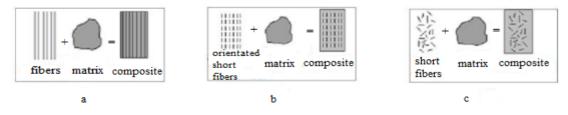


Figure 3.12: Fiber reinforced composite material types according to the shape (a) continuous fiber, (b) discontinuous fiber, (c) random oriented [28].

3.5 Manufacturing Techniques of Composite Materials

Composite materials can be produced by different manufacturing techniques and manufacturing depends on geometry, desired quality, cost and experience. These techniques may roughly divide into two categories as open and closed molding. In open molding, the process is done under the effect of atmosphere. In closed molding, the process is done with the aid of molds or vacuum bags that block the effect of atmosphere.

Table 3.4: The data of composite manufacturing techniques in World, Europe and Turkey [35]

Turkey [33]	T		
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%

3.5.1 Hand Lay-Up Method

Hand lay-up method is generally used for boats, tanks and building panels. In the production, the woven fibers as a reinforcing material are usually selected. But in order to gain additional strength and modulus of elasticity also glass, carbon or other fibers can be added in to manufactured structure [34]. Desired thickness can be reached and this process has low tooling costs. Complex geometries can be produced with experienced operators. Slow process, unwanted gaps and lack of adaptation to mass production can be said as disadvantages. Turbine blades and marine applications like boat hulls and kite boards can be produced with this technique [35]. The hand lay-up procedure is mentioned below step by step according to figure 3.12 [36].

(1) The wax should be applied to the mould to remove the product easily from the mould, (2) applying the second separator by using polyvinyl alcohol with sponge, (3) applying the high viscosity resin with brush, (4) preparing fibers, (5) mixing the hardener with resin, (6) applying the mixed resin by brush, (7) applying the paint roller to avoid the air bubbles in structures, and (8) the process is continued until the desired thickness and hardening and then the product is removed from the mould [36].

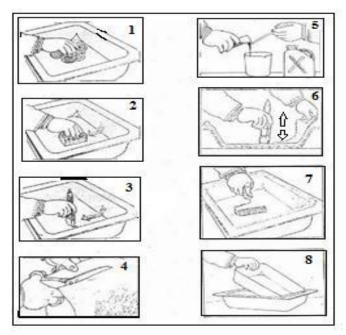


Figure 3.13: Composite production by hand lay-up method [36]

3.5.2 Spraying Method

Spray-up is also open molding production technique usually used for small boats and sandwich panels. Gel coat is optional and depends on manufacturer for a surface finish quality. With this technique, continuous fibers are chopped with a chopping mechanism and discontinuous short fibers are provided and liquid resins are sprayed onto mold together with the aid of a nozzle or a gun. This process is simple, low cost and has easy setup. Because of chopped fibers, desired strength may not be available and this can be said as a disadvantage [24, 35]

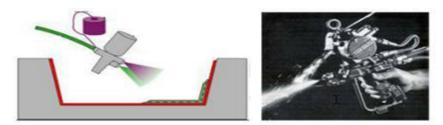


Figure 3.14: An illustration of spraying method and the spray gun [24, 15].

3.5.3 Vacuum Bagging Molding Method

Vacuum bag molding has appeared in order to eliminate the shortcomings of open molding processes. This technique has steps as follows (Figure 3.13).

- Fibers and resins are placed on mold as wet lay-up.
- A flexible film (nylon, PVA etc) is placed over wet lay-up.

• A vacuum is started and atmospheric pressure compresses laminates.

Vacuum bagging has important advantages over hand lay-up. First of all efficient laminating can be done. Improved strength can be achieved because of removing trapped air and emptying excessive resin from laminates, with this feature this technique decreases resin cost.

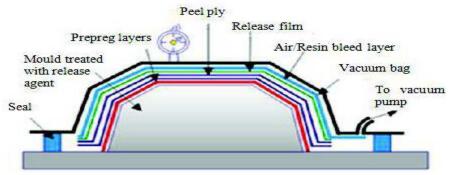


Figure 3.15: Schematic illustration of vacuum bag molding [37]

3.5.4 Vacuum Assisted Resin Infusion Molding

Vacuum assisted resin infusion molding is a various application of vacuum bag molding. The difference between bag and infusion is that resin is entered to mold after vacuum is started and air is almost evacuated (Figure 3.14). So reinforcements are already ready and resin comes after vacuum operation. Also position of reinforcements may be well defined and excessive resin problem is resolvable. Desired mechanical properties of composite materials which are produced with vacuum infusion technique can be achieved.

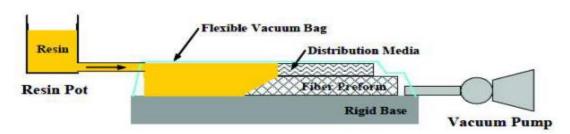


Figure 3.16: Schematic illustration of vacuum infusion process [38]

3.5.5 Filament Winding Method

A continuous band or tape of resin - impegranted fibers is wrapped over a mandrel to produce a hollow part. A large number of fiber roving is pulled from a series of

payoff devices, collimated into a band through the use o textile thread board or a stainless steel comb, passed into a liquid resin bath and a wiping device, and then wrapped over a mandrel. Either the mandrel or the application head can rotate to give the fiber coverage over the mandrel. Although the rotating mandrel is far more common, simultaneous rotation/translation of both mandrel and application head permits filament winding of complex non-uniform shapes [39,40].

The process which incorporates the resin impregnation as part of winding process is called wet filament winding. The process which uses prepreg tow as the winding medium is called dry filament winding. In wet winding, fiber tension is controlled by the fiber guides or scissor bars located between each payoff device and the resin bath. A wiping device is normally a set of sequeeze rollers where the clearence gap between rollers can be adjusted to the resin content. The traversing speed of the carriage and the winding speed of the mandrel are controlled to provide the desired winding angle patterns. A helical winding pattern is created with a rotating mandrel and a translating a carriage. By adjusting the carriage speed and the mandrel rotation rate, any wind angle between near 0 and 90 can be achieved. For a circular mandrel rotating with a constant rotational speed, N (revolutions per minute) and a constant carriage feed of V, the wind angle is given by;

$\theta = 2\pi Nr/V$

where (r) is the radius of the mandrel. A constant (θ) can be maintained in a thick part if the ratio N/V is adjusted from layer to layer.

Most standard matrix resins can be used for filament winding, provided that they are low in volatile content and have proper viscosity characteristics. Viscosity of the resin should be high enough so that resin dripping in the mandrel can be avoided and yet low enough so that good fiber wet out can be achieved. Resins such as epoxy, polyester, etc. can be used for filament winding. The parts can be left on the mandrel and a mold is placed over the part for autoclave curing. In some cases, parts can be simply oven cured. The effects of gravity may be minimized if the part is rotated while being cured. Characteristic of filament winding [40];

- Parts of widely varying size may be produced,
- Non-cylindrical shapes can be made,

- Panels and fittings for reinforcement or attachment can be easily included in the winding process,
- Low void content and good fiber/resin ratio can be achieved,
- Parts with high pressure ratings can be fabricated.

Some applications of filament winding include pressure vessels, fuel and water tanks, rocket motor cases, pipelines, automotive drive shafts, and helicopter blades. In sporting goods, the filament winding process is used also to manufacture tennis rackets from prepreg sheets. The sheets are formed by slitting the wound shape parallel to the mandrel axis or at an inclined angle to provide adequate normal and shear strengths to the tennis rackets.

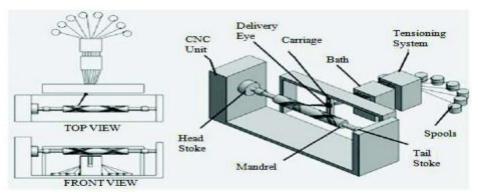


Figure 3.17 : Schematic illustration of filament winding [35]

3.5.6 Resin Transfer Molding (RTM) Method

Resin transfer molding is a closed molding process which has two molds, reinforcements are placed in these molds and resin is injected into these molds for producing advanced continuous fiber reinforcements. All fibers can be used with forms such as mat and woven. After resin transfers, molds are heated and curing cycle starts and resin solidifies. Gel coat may be used for better surface finish quality. In this method, vacuum can be used for removal of the air bubble. One of the advantages is fast production and this process is adaptable to mass production. Higher fiber-resin ratio can be provided so after manufacturing, finalized product is lighter and has more strength. Complex shapes can be manufactured by cavities. Tooling costs are high and also molds are controlled by hydraulic presses. Some automotive products such as auto body panels, wind turbine blades can be manufactured [15,35].

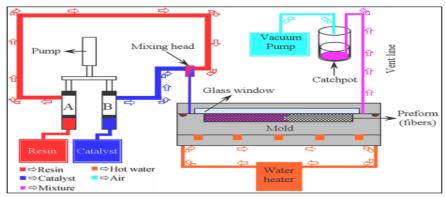


Figure 3.18: Schematic illustration of RTM [26]

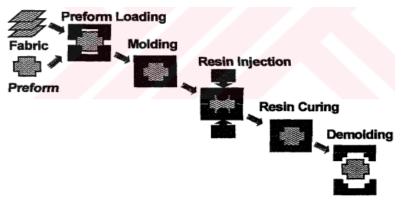


Figure 3.19: A basic schematic illustration of RTM [41]

3.5.7 Structural Resin Injection Molding Method (SRIM)

Structural Resin Injection Molding (SRIM) is a derivative of RTM process. Once the mold has been closed, the resin and the cross linking agent is injected separately into the mold and reacts quickly within the mold to carefully within a few seconds. This chemical reaction proceeds as the resin penetrates through the preform and, therefore, SRIM requires fast fiber wet-out and air displacement. Through impregnation of reinforcement is quickly followed by complete cure of resin. The resin rapidly becomes to thick to permit resin flash through vents. SRIM parts normally do not require post curing [40, 43]

3.5.8 Autoclave Curing

Autoclave is a container that can control the temperature, pressure and absorption. This method is similar to the vacuum bagging method. Different from vacuum bagging method more regular and controllable pressure is applied from outside. For this, compressed gas is given to container of composite material. Instead of oven, autoclave is used. Hence, curing conditions can be fully controlled. Autoclave curing which is usually performed for aerospace and ballistic applications is a vessel that

controls temperature and pressure for polymeric composite materials to remove unwanted air. The machine applies pressure with temperature. After curing operation composite material has better resin-fabric ratio so strength properties are improved to better levels. Cost can be said as a disadvantage and the process is not suitable for small parts [26, 27, 35, 42].

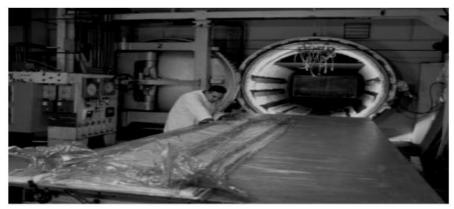


Figure 3.20 : A vacuum bagged composite aircraft part ready to enter the Autoclave [26]

3.5.9 Pultrusion Method

Pultrusion is a continuous process for manufacturing products that have constant profiles such as pipe, beams and structural shapes. Roving fibers go through resin bath with a guide puller and then formed. Multiple rows can be used with an automated process. After forming, curing process takes place. Lastly cutting is done after forming and curing operation. These products which are manufactured with this technique have high strength properties with providing enough fiber contents. High mechanical strength is obtain at reinforcement direction due to the use of continuous fiber. This technique has a disadvantage because of limited to uniform cross sections [26, 27, 35, 42].

Advantages of pultrusion;

- Pultrusion has a much higher material usage (95%) than lay up (75%) therefore, it is more productive in time and material,
- High throughput rate,
- High resin contents available

Disadvantages of pultrusion;

- Part cross sections must generally be uniform,
- Problems can arise when resin or fibers accumulate and build up at the die opening. This can increase the friction to the point that the equipment will be jammed or the fibers will break.
- Voids can result if the dies are run with too much opening for the fiber volume [41].

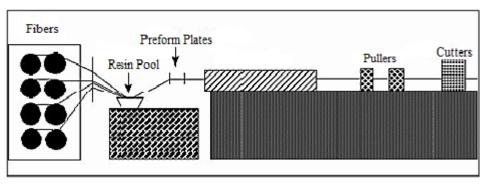


Figure 3.21: Schematic illustration of pultrusion [26]

3.5.10 Continuous Laminating Method

In this method, the glass fiber reinforcement materials and polyester poured onto a plastic film carrier and then with a second film overlying layer, the mixture is drawn between two sheets of plastic by compressing and is ovened. Two side smooth corrugated, corrugated transparent or opaque plate production is provided.

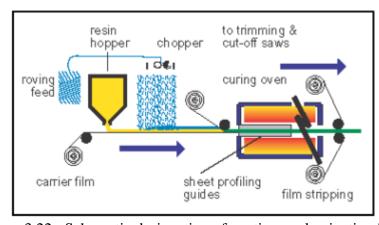


Figure 3.22: Schematic designation of continuous laminating [24].

3.6 Types of Tests are being Applied Composite Materials

In order to determine the strength properties of composites mechanical tests are applied. By these tests some information is obtained from produced composites which carries or not the expected properties. By utilizing obtained data continuity of production is provided or by changing of some parameters like the applied production type, amount and types of materials were used, can expected mechanical properties can be manufactured.

In general mechanical tests applied to composite materials are impact test, tensile test, and the three point bending test.

3.6.1 Impact Test

Besides the advantage of composite materials there are some usage restrictions exist. The most important of these limitations is reaction against regional impact which they are exposed to the from foreign material under operating conditions. To measure the impact response of composite materials, lots of researches were carried out.

Test apparatus used to measure the response to the impact load of composite are Izod and Charpy pendulum impact test, drop weight test and gas gun test.

3.6.1.1 Charpy Impact Test

In the initial stages of the impact study for composite materials, designed primarily for metal Charpy test system is used. Two basic reasons for the use of this mechanism is that it is relatively simple and adjustable. Thus able to provide information about energy absorption mechanisms for composites. The test sample is generally a thick beam and sometimes a notch is opened in the middle of beam. The sample is supported on a horizontal plane and exposed to oscillating loading by a hammer blow from opposite of the notch (figure 3.24). During impact test absorbed energy is read by help of a scale which on test device.

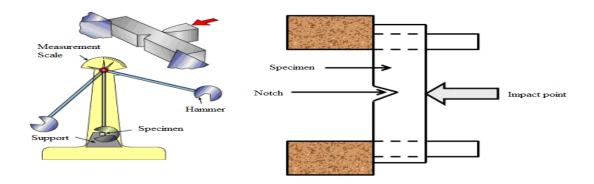


Figure 3.23: Schematic Illustration of Charpy Impact Test [42].

3.6.1.2 Izod Impact Test

In the Izod impact test, test apparatus and process steps are similar to mentioned in the Charpy test. In the Izod test, the sample is attached from at one edge as a fixed beam at the vertical plane and is exposed to impact loads by a released pendulum from the unsupported end.

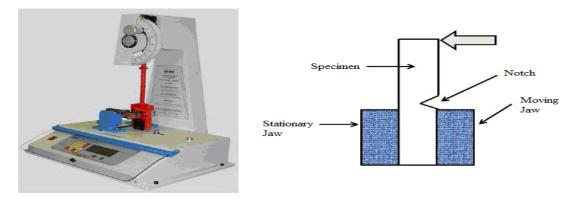


Figure 3.24: Schematic Illustration of Izod Impact Test [42].

3.6.1.3 Drop Weight Test

In the drop weight test, the weight release from a certain height to the sample which is fixed in the horizontal plane. Impact does not cause deformation of the sample completely. The speed of impact can be measured with the aid of optical sensors placed just 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 use of mostly semi-spherical geometry tip point, sharp and flat type tip point can also be used for impact.

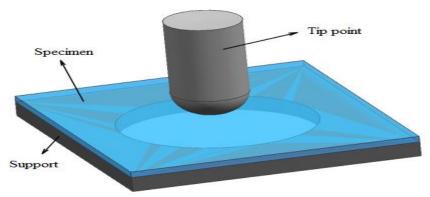


Figure 3.25: Schematic Illustration of Drop Weight Test [42].

3.6.1.4 Gas Gun Test

In this test mechanism, nitrogen or a similar gas is filled to reservoir which is placed end of the gun barrel. Where the gas is stated by a plastic diaphragm. The diaphragm explode when the gas reaches the designated pressure and accelerates the impact tip point which is inside the gun barrel and this enables to hit specimen which fixed to other end. The hit speed of tip point can be measured by help of optical sensors or with a simple wire breaking mechanism [44, 45].

It can be used for large test pieces and therefore used to measure high speed impact behavior of composite materials.

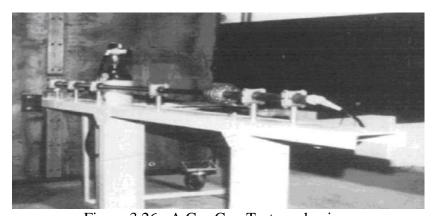


Figure 3.26 : A Gas Gun Test mechanism

3.6.2 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 the tensile test device and the variable and axial forces are applied.

Tensile test device composed of two jaws which can move basically up and down relative to each other. The test piece is connected to these jaws which give force and movement. By one of the jaws move with a constant speed, varying amounts of tensile force is applied to the test piece and elongation corresponding to this force is recorded.

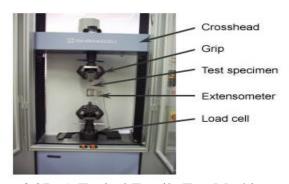


Figure 3.27: A Typical Tensile Test Machine

3.6.3 Three Point Bending Test

Three-point bending test can be defined as the deformation occurring in the sample when force is applied in the middle of a test sample which is a circular or rectangular cross-section that placed freely on the two supports. If the sample is broken, applied load measured during the fracture. Bending test specimens can be circular section or rectangular section. There is no problem about connection of samples to bending test device. According to the total number of supports which contact with specimen and load applications bending test is divided into different groups, such as three-point bending test or four-point bending test. The main purpose of the bending test is to determine the ability to endure the cold state cracking of the material. Tensile and compressive stresses occur at the cross section of sample is subjected to bending [47].

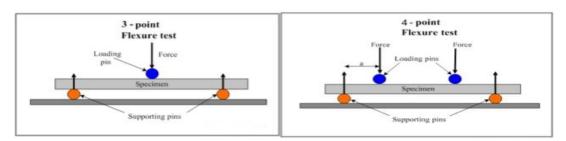


Figure 3.28: A Basic Illustration of Bending Test Types [48]

CHAPTER 4

EXPERIMENTAL METHOD

4.1 Polyester Resin

Polyester resins are the most common resin in manufacturing of composite materials. These are collected in two groups as unsaturated and saturated polyesters. Saturated polyester resins are used in the injection molding process [49]. Unsaturated polyester resins are used in the construction of fiber-reinforced materials. Polyester resins are classified according to storage lifetime. Generally, a small amount 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 polyester resin. These products are usually the catalyst accelerator additives such as pigments, fillers, supplements for chemical resistance. The polymerization rate is very slow for polyester resins; therefore, the catalyst and accelerator must be added to polyester resins to obtain a faster polymerization process. The polyester resins show good properties in terms of mechanical and chemical resistance, low prices, and flexible usage of intended design [15]. Polyester resins are preferable [35].

In this experimental study, the resin material is used in composite manufacturing POLIPOL 3401-TAB CTP type thixotropic accelerated general purpose polyester resin obtained from Poliya.

4.2 Hardener (Methyl Ethyl Ketone Peroxide - MEKP)

The most common initiator for operations which are made at room temperature is methyl ethyl ketone peroxide (MEKP) as an organic peroxide. They provide the energy required to initiate the polymerization. Hardener (MEKP) is used as 2 percentage of polyester weight for all specimens. Some properties about MEKP are given as [55];

The physical condition : transparent liquid

Density (kg/m³, 20°C) :1170

Flash point ($^{\circ}$ C) : 60

Viscosity (mPa.s, 20 °C): 25

4.3 Sewage Sludge Ash

Sewage sludge ash that is used as filler is provided from Gaziantep Metropolitan Municipality Water and Sewerage Department (GASKİ). The compounds and their percentages in ash are shown at table 4.1 [50]. One of the most critical parameters is size of particle that affects mechanical properties of composite materials, homogeneous distribution, adhesion and holding to resin/matrix [51, 52, 53, 54].

Provided ash is sieved from 45 µm sieve. Short and small size additives should be preferred due to it provides high specific surface area and uniform distribution in the composite [51, 53]. Ash is sieved in order to achieve homogeneous size distribution, good adhesion and provide a good uniformity between specimens which will be molded and tested. Otherwise, if a composite specimen contains different size ashes, specimen will exhibit different strength behaviors due to the adhesion of ash.

The weight rate of ash is used between 0% and 45%. However for weight rate of 45% and above molding couldn't achieve due to polyester-ash mixture become saturated so quickly. Materials and weight rate of them are tabulated at table 4.2. In the table, tensile test sample is referred as T, 3-point bending test samples are called as B, impact test samples are called as I.

Table 4.1 : Compound within ash and their percentage [50].

Oxide	Percentage	Oxide	Percentage	Oxide	Percentage
P_2O_5	23.655	K ₂ O	4.874	CuO	0.189
CaO	19.588	ZnO	2.096	MnO ₂	0.188
SiO ₂	16.602	TiO ₂	1.079	NiO	0.068
SO ₃	8.533	Cl	0.539	Br	0.063
MgO	8.221	Na ₂ O	0.442	SrO	0.049
Fe ₂ O ₃	7.461	Cr ₂ O ₃	0.354	ZrO ₂	0.036
Al ₂ O ₃	5.727	BaO	0.206	PbO	0.031

Table 4.2 : Weight Rate of Ingredient of Tensile Test,3 Point Bending Test and Impact Test Specimens

Specimen	Polyester (%)	Hardener (%)*	Ash (%)
T0 - B0 - I0	98	2	0
T5 - B5 - I5	93	2	5
T10 - B10 - I10	88	2	10
T15 - B15 - I15	83	2	15
T20 - B20 - I20	78	2	20
T25 - B25 - I25	73	2	25
T30 - B30 - I30	68	2	30
T35 - B35 - I35	63	2	35
T40 - B40 - I40	58	2	40

^{*}The weight rate of hardener is 2% of weight of polyester.

When compound of sewage sludge ash and fly ash compared, it will be seen that the ingredients of compounds are similar. The type of compound and their percentages are at table 4.3 at below.

Table 4.3: The chemical compounds of fly ash obtained from thermal power plants, in Turkey and Chemical compound of sewage sludge ash [50, 61]

Compound	Flyash (Elbistan)	Flyash (Çatalağzı)	Flyash (Seyitömer)	Flyash (Soma)	Flyash (Tunçbilek)	Flyash (Orhaneli)	Flyash (Yatağan)	Sewage Sludge Ash
SiO2	27.4	56.8	40.6	39.8	56.4	48.8	51.2	16.602
CaO	47	1.4	19.9	25.4	2.1	10.1	13	19.588
Al2O3	12.8	24.1	9.1	22.3	23	19.6	22.9	5.727
Fe2O3	5.5	6.8	7.7	4.4	10.1	6.5	7.8	7.461
MgO	2.5	2.4	3.1	1.9	3.3	4	2.8	8.221
SO3	6.2	2.9	10.6	4.8	0.4	4.2	0.3	8.533
Ti(O2)	0.7	1.1	-	0.6	-	-	0.9	1.079
P2O5	-	-	-	-	-	-	-	23.655
K2O	-	-	-	-	-	-	-	4.874
ZnO	-	-	-	-	-	-	-	2.096
Cl	-	-	-	-	-	-	-	0.539
Na2O	-	-	-	-	-	-	-	0.442
Cr2O3	-	-	-	-	-	-	-	0.354
BaO	-	-	-	-	-	-	-	0.206
CuO	-	-	-	-	-	-	-	0.189
MnO2	-	-	-	-	-	-	-	0.188
NiO	-	-	-	-	-	-	-	0.068
Br	-	-	-	-	-	-	-	0.063
SrO	-	-	-	-	-	-	-	0.049
ZrO2	-	-	-	-	-	-	-	0.036
PbO	-	-	-	-	-	-	-	0.031
Si + Al + F	39.7	87.7	57.4	66.5	89.5	68.4	81.9	-
Alkali	0.3	3	1.4	0.4	0.9	1.7	2.9	-
Lost	2.4	0.6	1.4	0.4	1.1	-	0.4	-

4.4 Stirring of Materials and Preparation of Specimens

For each class of samples (T0 - B0 – I0; T5 - B5 – I5; etc.) mixing and molding processes are performed simultaneously. Stirring has been made in laboratory with 1000 rev / min speed drills with mixing equipment. Attention is paid to the homogeneity of the mixing apparatus fitted to drill. By adding the particles and hardener to mixture the mixing process continued until mixture became to gel form [56]. This form is extremely critical to avoid the negative factors, such as flocculation and precipitation may occur in the freezing process after casting the composite. Particles which are added to polyester may be a factor for increasing stress. These fillers can act as stress raisers that cause weakness in the structure by introducing discontinuity in stress transfer process across the filler–polymer interface [7]. These error sources that will be effective on the part is directly related with homogeneous of composite during casting and freezing. The presence of agglomeration and voids in the composites obviously deteriorates their mechanical properties [57, 58].

Stirring is initiated by addition of the hardener by 2% weight of polyester. Ash is added to polyester - hardener before mixture become gel form. In this way, by hold on ash to mixture, it is intended to prevent the agglomeration and sedimentation. Each sample is kept at room conditions 3 weeks before testing.

Figure 4.1 and figure 4.2 are the beginning of polyester-hardener mixture and the reaction of polyester - hardener just before adding ash to mixture respectively. Discoloration of polyester resin is observed by reaction of hardener and polyester. The color of discoloration can vary according to kind of polyester.



Figure 4.1: Beginning of mixture



Figure 4.2: Reaction of polyester and hardener

4.5 Mechanical Test

4.5.1 Tensile Test

Three samples are molded in order to apply tensile test accordance with standard ASTM D 3039. Samples are tested with a 300 kN capacity universal test machine Schimadzu with 1 mm/sec constant speed [7], three weeks after casting. Tensile strength is calculated again for each samples with final surface area after test was finished. Each test is performed for three different specimens. Results are interpreted taking the average of three samples. The tensile test machine is shown at figure 4.3 and the fractured specimens are shown at figure 4.4.



Figure 4.3: Universal test machine for tensile test



Figure 4.4: Tensile test specimens

4.5.2 Three Point Bending Test

In order to apply three point bending test 3 samples are molded into prepared mold. Samples are tested with a 300 kN capacity universal test machine with 2 mm/sec

constant speed [7] three weeks after casting. A large span to depth ratio in bending test increases the maximum normal stress without affecting the inter-laminar shear stress and thereby increases the tendency for longitudinal failure. If the span is short enough, failure initiates and propagates by inter-laminar shear failure. The maximum shear stress in a beam occurs at the mid-plane. So in the shear test, failure consists of a crack running along the mid-plane of the beam so that crack plane is parallel to the longitudinal plane [4]. In the present investigation, during flexural test, the span length is 16:1. Results are interpreted taking the average of three samples. The calculation of bending stress is made by using formulas 4.1 and 4.2 [59]. Figures 4.5 and 4.6 are for showing three point bending test machine and fractured samples for three point bending. Samples are fractured as expected from the midpoint of specimen.

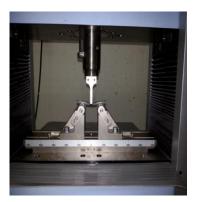


Figure 4.5: Three point bending test machine



Figure 4.6: Three point bending test samples

$$\mathbf{O}_m = \frac{|M|}{S} \tag{4.1}$$

$$S = \frac{1}{6}bh^2\tag{4.2}$$

 σ_m = Bending Stress b = width of the cross section M = Bending Moment h = depth of the cross section

4.5.3 Impact Test

There are several mechanisms for toughening of polymer. For the inorganic particles toughened polymer, at least three factors are necessary: inherent ductility of the matrix, weak inter phase supporting the filler/matrix debonding and suitable inter particle distance. The stress concentration first leads to debonding of the filler particles and voids formation. The particle content affects the inter particle distance and the stress state of the matrix polymer surrounding the voids [12]. Load is carried by the material during the impact test is an important parameter for the material that can be related to fracture or damage.

Impact test represents the energy needed to break the sample. This magnitude is called as failure energy and denoted with W;

$$K = \frac{W}{A}$$
 (4.3) [60]

 $K = \text{Impact Resistance} : (J/m^2), \qquad W = \text{Failure energy} : J,$ $A = \text{the area of the specimen cross-section where fracture occur: } m^2$

Impact strength is calculated again for each samples with final surface area after test finished. Results are interpreted taking the average of three samples. The schematic designation of impact test machine, impact test specimens, and impact test machine are showed at figure 4.7, 4.8, 4.9 respectively. Impact test is made accordance with ISO 179.

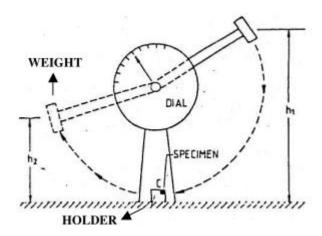


Figure 4.7: The Schematic Designation of Impact Test Machine



Figure 4.8 :Impact test specimen



Figure 4.9: Impact test machine

Tensile test, three point bending test and impact test were applied to composite material in order to define the mechanical effect of sewage sludge ash on the composite material. The interpretation of tests will be handed in chapter five.

CHAPTER 5

RESULTS

In chapter four, information about current study is given. These informations are about polyester, hardener, sewage sludge ash and its compound, preparation of specimen which would be tested and mechanical tests (tensile test, impact test, three point bending test). In this chapter the test result will be given and the interpretation about tests will be done.

5.1 Test Results

Tensile test is performed for three different specimens and results are interpreted taking the average of three samples. Also for three point bending test and impact test results are interpreted taking average of three samples.

5.1.1 Tensile Test Results

As shown in figures 5.1 and 5.2, maximum force that occurs at fracture and associate with tensile strength increase until weight rate of ash reach 20% of total mixture (polyester-hardener-ash). It can be interpreted that homogenous (distribution of ash in the mixture) is good, adhesion and interaction of resin and ash is good due to the test result showed a continuity each other between weight rate of ash 0% and 20%. Maximum force and tensile stress decrease from 20% to end. This decline was unexpected at 30%. The particulate filled composites are composed of hard fillers dispersed in a polymer matrix often brittle by itself. These fillers can act as stress raisers that cause weakness in the structure by introducing discontinuity in stress transfer process across the filler-polymer interface. The resulting stress concentrations at the defect sites lead to early initiation of the failure process. The increase of filler loading (discontinuous phase) further aggravates these problems in such a composite [7]. There can be two reasons for this decline in the strength properties of these particulate filled composites compared to the unfilled ones. One possibility is that the chemical reaction at the interface between the filler particles

and the matrix may be too weak to transfer the tensile stress; the other is that the corner points of the irregular shaped particulates result in stress concentration in the polyester matrix. These two factors are responsible for reducing the tensile strengths of the composites so significantly [3].

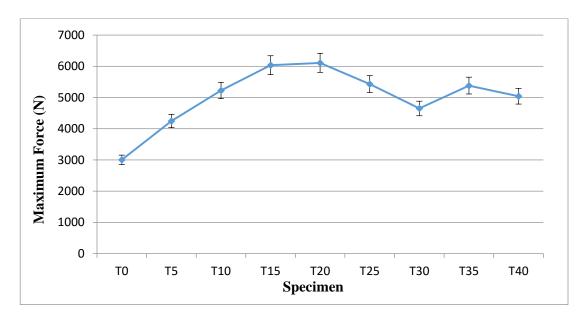


Figure 5.1: Maximum Force - Specimen Graph for Tensile Test

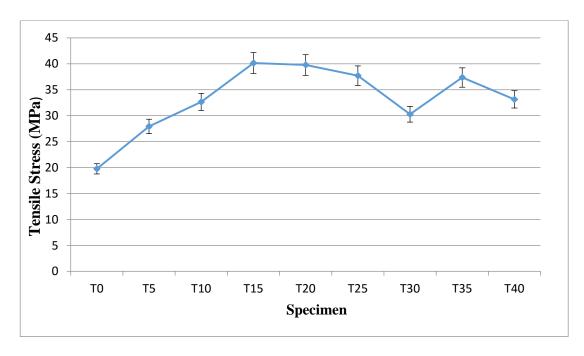


Figure 5.2: Tensile Stress - Specimen Graph

The results of tensile test for sewage sludge ash particle reinforcement composite material have been plotted in figure 5.3 as stress - strain graph in order to demonstrate the tensile loading performance. Stress - strain graph show nearly same

character with force and tensile stress graph. That is, elastic limit increase until weight rate of ash reaches 20%. Then the area for elastic limit decreases.

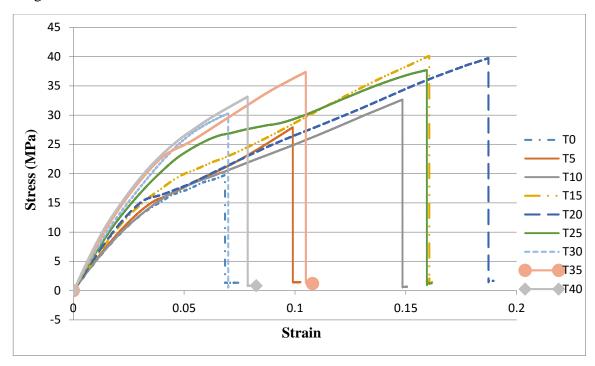


Figure 5.3 : Stress - Strain Graph for Tensile Test

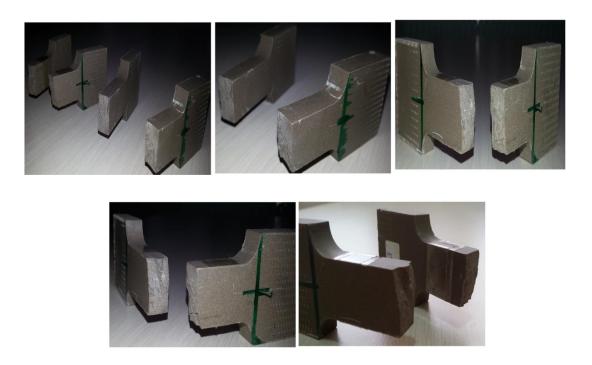


Figure 5.4 : Typical Failed Specimen for Tensile Test

5.1.2 Three Point Bending Test

As seen at figures 5.4 and 5.5, according to result of three point bending test, as the weight rate of ash content in the composite increase, the strength values decrease. This decrease is very close between rate of 20% and 35%, in fact it showed a constant character. At 40% the strength value decreases again. Actually for weight rate of 5% the maximum force showed an increase tendency. However this increase is eliminated by measuring final cross section area where fracture occur.

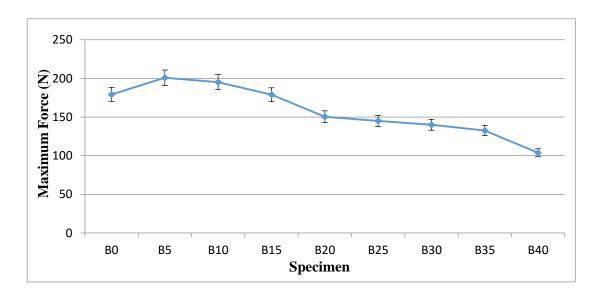


Figure 5.5: Maximum Force - Specimen Graph for Three Point Bending Test

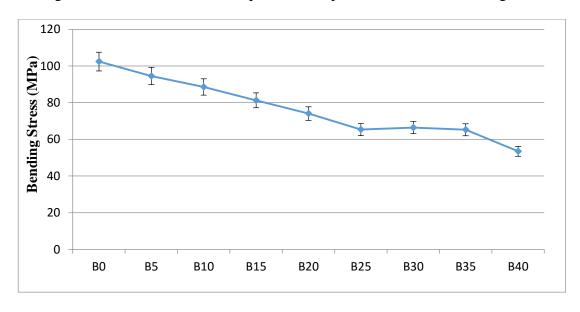


Figure 5.6 :Stress - Specimen Graph for Three Point Bending Test

The results of three point bending test for sewage sludge ash particle reinforcement composite material have been plotted in figure 5.7 as stress - strain graph in order to

demonstrate the bending loading performance. Stress - strain graph show same tendency with force and bending stress plots. By adding of sewage sludge ash to polyester resin, elastic limit decrease.

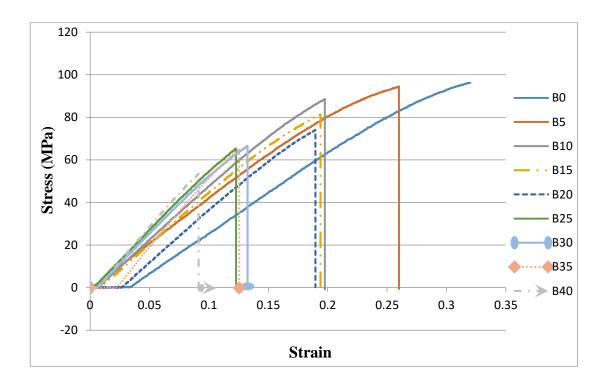


Figure 5.7 : Stress - Strain Graph for Three Point Bending Test



Figure 5.8: Typical Failed Specimen for Three Point Bending Test

Table 5.1 : Test Results for Tensile and Three Point Bending Test

Test Type	Specimen	Maximum Force (N)	Strength (MPa)	Maximum Strain (%)
	T0	3003.79	19.76	1.78
	T5	4245.85	27.93	2.53
	T10	5225.13	32.66	3.80
	T15	6036.38	40.14	4.11
Tensile Test	T20	6108.76	39.77	4.80
	T25	5429.17	37.70	4.08
	T30	4649.83	30.27	1.78
	T35	5380.82	37.37	2.72
	T40	5038.88	33.15	2.05
	В0	179.29	102.39	5.08
	B5	200.94	94.50	3.65
	B10	195.22	88.57	2.83
	B15	179.05	81.23	2.78
Three Point Bending Test	B20	150.49	74.03	2.60
	B25	145.10	65.31	1.76
	B30	140.09	66.42	1.85
	B35	132.56	65.21	1.72
	B40	103.81	53.47	1.22

5.1.3 Impact Test

As seen from figure 5.7 and 5.8 impact resistance and absorbed energy increase until weight rate of ash reaches 10%. Impact resistance decreases between 10% and 20%. After from 20% impact resistance and absorbed energy showed nearly constant value.

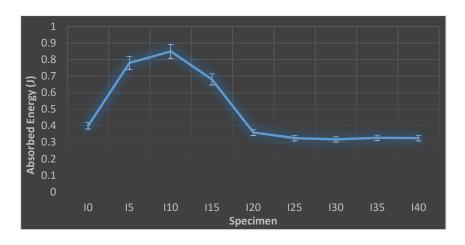


Figure 5.9: Impact Test, Absorbed Energy (J) - Specimen Diagram

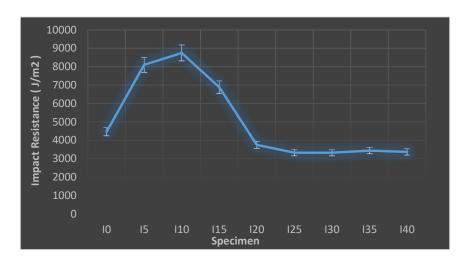


Figure 5.10 : Impact Test, Impact Resistance (J/m^2) - Specimen Diagram

Table 5.2: Test Result for Impact Test

Test Type	Specimen	Absorbed Energy (J)	Impact Resistance (J/m2)
	ΙΟ	0,4	4458,38
	I5	0,78	8100,86
	I10	0,85	8746,29
T4	I15	0,68	6880,32
Impact Test	I20	0,36	3751,5
1681	I25	0,325	3319,28
	I30	0,3175	3319,57
	I35	0,327	3435,17
	I40	0,325	3362,62





Figure 5.11 : Typical Failed Specimen for Impact Test

CHAPTER 6

DISCUSSION AND CONCLUSION

In the current study a research on effect of sewage sludge ash on the mechanical properties of composite material is carried out. Tensile test, three point bending test and impact test are handed as mechanical tests.

Polyester is used as resin material during the preparation of mixture. In the first step of study, the homogenous of mixture must be achieved in order to take faultless results. If not, results show a wavy character. This was achieved by adding correct weight ratio of hardener at correct time. That is sedimentation is prevented by adding hardener at true time.

For homogenous of mixture, correct hardener ratio or correct mix type are not enough alone. In addition to these factors there is a critic point about homogenous which is particle size. In the first stage of laboratory studies the sewage sludge ash is used as mixed sizes (without sieving). Ash is used as got from plant. However this caused inconsistent test results for each specimen group (T0, T5, B0, B5, I0, I5, etc.). Then sewage sludge ash is sieved and test result showed uniform data.

After preparation of polyester - hardener – ash mixture, dies are molded. Specimen is waited for same period and then mechanical tests are applied to composite.

Test results showed that adding sewage sludge ash to polyester affect the mechanical properties of composite significantly. For tensile test result, tensile stress increase until weight rate of ash reach 20% and beyond it decreases. For three points bending test, adding of sewage sludge ash affects negatively to composite. In other words adding ash decreases the bending stress of material. Finally for impact test, absorbed energy and impact resistance increase until adding ash reaches 10% and beyond it decreases.

By current study, sewage sludge ash which has not been used before is used and tested in the literature for the first time. This is not the single point for importance of

study. As mentioned before this kind of ash is a waste of the combustion process. Sewage sludge ash is not useful yet for any industry and is useless waste, generally disposed as landfill. If the use of ash is achieved an environmental problem will be overcome.

By diversifying the usage of sewage sludge ash for industry, use of waste may be possible for industrial applications.

Different type of resin materials and/or use of ash with fabrics to produce composite may be future academic topics which are not available yet in the literature.

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