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İZMİR KATİP ÇELEBİ UNIVERSITY

GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

**WOOD WASTE RECYCLING FOR THE PRODUCTION OF WOOD
POLYMER COMPOSITES**



M.Sc. THESIS

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Department of Materials Science and Engineering

JULY 2019

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İZMİR KATİP ÇELEBİ ÜNİVERSİTESİ

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**ATIK AĞAÇ GERİ DÖNÜŞÜMÜYLE AHŞAP POLİMER KOMPOZİT
ÜRETİMİ**

YÜKSEK LİSANS TEZİ

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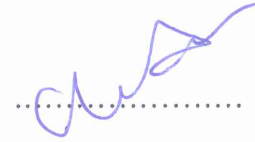


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To my family,

FOREWORD

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ABBREVIATIONS

WPCs	: Wood–Plastic Composites
PP	: Polypropylene
NFCs	: Natural Fiber Composites
MDF	: Medium Density Fiberboard
MAPP	: Maleic Anhydride grafted Polypropylene
FTIR	: Fourier Transform Infrared Spectroscopy
XRD	: X-ray diffraction analysis
SEM	: Scanning Electron Microscopy



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WOOD WASTE RECYCLING FOR THE PRODUCTION OF WOOD POLYMER COMPOSITES

ABSTRACT

Material selection plays important role not only for human life but also for engineering design of a successful sustainable product. Especially, importance of natural fiber reinforced composites is growing fastly both in terms of their applications and properties. Wood is popular natural fibers. They are available, renewable, low density, light, abundant and cost as well as satisfactory mechanical properties make them an attractive ecological alternative to glass, carbon and other man-made fibers used for the manufacturing of composites.

In this study, wood-polypropylene composites were manufactured via co-rotating twin-screw extrusion technique with coupling agent. In this study, as a wood hornbeam, pine and medium density fibreboard were used. Samples manufactured in certain proportions 10%, 20%, 30% wood and 3% or 6% MAPP (coupling agent) and PP. As a last step, composites materials were characterized in terms of their mechanical, spectroscopic and morphological properties. Tensile and flexural mechanic analysis were conducted to determine the mechanical properties of the materials.

The spectroscopic characterizations of the wood-polymer composites were carried out to verify the adhesion bonding between wood types and polypropylene. The morphology of the composite was investigated via scanning electron microscopy (SEM).

The results showed for Hornbeam /PP composite 13% increase in tensile strength and showed 33% increase in the elastic modulus, for Pine / PP composite reduce 15% in tensile strength and showed 37% increase in the elastic modulus, for MDF / PP composite increase 11% in tensile strength and showed 50% increase in elastic modulus. In flexural test, Hornbeam / PP 13% increase in flexural strength and 54% increase flexural modulus. They showed for Pine / PP almost same values for flexural strength and 48% increase flexural modulus. It showed for MDF / PP 19% increase in flexural strength and 52% in flexural modulus compared to the neat PP. According to spectroscopic characterizations of the composite samples, the interfacial interaction between matrix and wood phase was improved with addition of effective coupling agent with different amount.

Keywords: Wood, wood-polymer composite, composites, hornbeam, pine, medium density fiberboard.

ATIK AĞAÇ GERİ DÖNÜŞÜMÜYLE AHŞAP POLİMER KOMPOZİT ÜRETİMİ

ÖZET

Malzeme seçimi günlük yaşantımızda önemli bir role sahip olmasının yanısıra mühendislik uygulamalarında ve tasarımlarında ürün sürdürülebilirliği açısından da önemlidir. Özellikle, çevre dostu malzemelerin önemi günümüzde artmaktadır. Bu bağlamda, endüstriyel uygulama alanları ve eşsiz özelliklerinden ötürü ahşap takviyeli kompozit malzemelerin önemi artmaktadır. Ağaç popüler doğal liflerdir. Bunlar kullanılabilir, yenilenebilir, düşük yoğunlukta, hafif, bulunabilir ve uygun fiyata sahip olma gibi özelliklerinin yanı sıra tatmin edici mekanik özellikleri onları kompozit üretiminde kullanılan cam, karbon ve diğer insan yapımı elyafların çekici bir alternatifi yapmaktadır.

Bu çalışmada, ters dönüşlü, çift vidalı extruzyon tekniği ve bağlayıcı ajan kullanılarak ahşap-polimer kompozit üretilmiştir. Bu çalışmada, ağaç olarak gürgen, çam ve MDF kullanılmıştır. Numuneler %10, %20 ve %30 ağaç ve %3 ve %6 MAPP (bağlayıcı ajan) oranlarında ve polipropilen kullanılarak üretilmiştir. Son aşama olarak kompozit malzemeler mekanik, spektroskopik ve morfolojik olarak karakterizasyonu yapılmıştır.

Tekme testi ve dinamik mekanik analiz malzemelerin mekanik özelliklerini belirlemek amacıyla yapılmıştır. Kompozit morfolojisi taramalı elektron mikroskobu aracılığıyla araştırılmıştır.

Sonuçlar gösteriyorki, saf polypropilene kıyaslayınca, gürgen / PP kompozitinin çekme mukavemetinde %13, elastik modülü %33 artmış. Çam / PP kompozitinin çekme mukavemetinde %15 azalma, elastik modülü %37 artmış. MDF / PP kompozitinin çekme mukavemetinde %11, elastik modülü %50 artmış. Eğilme testinde gürgen / PP kompozitinde eğilme mukavemeti %13 artmış, eğilme modülü %54 artmış. Çam /PP kompozitinde eğilme mukavemeti neredeyse aynı kalmış, eğilme modülü %48 artmış. MDF / PP kompozitinde eğilme mukavemeti %19, eğilme modülü %52 artmış. Kompozit örneklerin spektroskopik karakterizasyonlarına göre, matris ve takviye fazı arasındaki etkileşimi etkili bağlama maddesinin farklı oranlarda eklenmesi ile geliştirilmiştir.

Anahtar kelimeler: Ahşap, Ahşap- polimer kompozit, gürgen, çam, orta yoğunluklu lif levha.

1. INTRODUCTION

1.1. Introduction to Composite Materials

The definition of composite materials referring 'advanced engineered materials'. It is consist of any combination of two or more materials at macroscopic level that shows better properties than any component which is used. The component of composites are matrix and reinforcement. The role of reinforcements is to increase the mechanical properties of the matrix.

The bulk part of a composite is called '**Matrix Phase**'. Materials in fibrous form are seen to be showing well strength feature and for succeeding this feature the fibers should be bonded by a matrix phase. During the manufacturing it can be liquid form, after that they became solid form. Polymer, metals and cermics can be matrix of composites.

In the composites, in order to strengthening the composite and improving the mechanical properties some additives were used. It is called '**Reinforcement**'. As a reinforcement different kinds of fibers can be used to affect the properties of materials. For example; one purpose of to add the reinforcement is to provides stiffness to the composite.

1.2. Composites Classification

The Composites are can be classified into some categories with respect to their components like matrix and reinforcing material type.

With respect to types of matrix material they can be classified as:

- a) **Metal -matrix composite:** They consists of metallic matrix like Fe, Al, Mg and Cu. Because of engineering properties metal matrix composites became very popular. The other important properties are, they are show good stiffness and light weight when compared to other kind of metals and metal alloys.

- b) **Polymer-matrix composite:** They are considered to be a very important and valuable group of composites when compared to metal matrix or ceramic matrix composites because of different applications. They consist of a matrix from thermoplastic (polyethylene, polystyrene, polypropylene, polyvinyl chloride) and including glass carbon, Kevlar fibers and steel or thermosetting (polyester, polyurethane, urea formaldehyde epoxy).
- c) **Ceramic-matrix composite:** This kind of composite include of a material consisting of a ceramic with a ceramic dispersed phase. When new technologies and new processing method develop, they became very popular. Actually, some high performance products were produced, however the brittleness is still remains a big disadvantage of ceramic material.

With respect to types of reinforcing material they can be classified:

- a) **Particulate composites:** They use as reinforcement material. It can be spherical, cubic, other regular or irregular shape. In general, particles are not very effective in improving fracture resistance but they increase the stiffness of the composite. Particle fillers are properly used to improve the properties of matrix materials such as to modify the thermal and electrical conductivities, increase performance at elevated temperatures, reduce friction, increase wear and abrasion resistance, increase machinability, improve surface hardness.
- b) **Fibrous composites:** Fibers can not be directly usable in engineering applications because of their small cross-sectional dimensions. Because of this reason, fibrous materials are take a place in matrix materials to form fibrous composites.

Fibers: These are generally classified into two groups:

- a) Natural Fibers
- b) Synthetic Fibers

Natural fibers: They produced from some plants, animal and mineral sources. Natural fibers may classified with respect to their origin material. For example: Cottons, wood parts and cashmere are very popular examples.

Synthetic fibers: These are man made fibers, they are discovered in research studies by scientists to develop natural occurring plant and animal fibers. For example: first synthetic fiber is nylon. Glassfibers and carbonfibers are used.

1.3. Advantages of Composites

Compared to the individual materials, composites have some advantages like; good resistance to corrosion, increase mechanical damping, high toughness, tensile strength, flexural strength, reduce the weight, low cost and easy processing.

1.4. Applications of Composites

- In transportation industry, some components like door frames, dashboards, lightning, engine parts etc.
- Daily products like toys, bottles, windsurf boards etc.
- Construction and building materials.
- Aircrafts and marine components.
- In some art activities.
- In some artistic materials or instruments like guitar.

1.5 Natural Fiber-Reinforced Polymer Composites

In last twenty years, natural plant fibers have been taken significantly attention as the substitute for synthetic fiber reinforcements like glass in plastics [1; 2]. The some advantages of plant fibers are; low density, low cost, admissible tensile or flexural strength, good thermal insulation, renewable resource and recycling possible without affecting the environmental bad effect and together with biodegradable capability [3; 4]. The literature shows, lots of works dedicated to the specialties of natural fibres from micro to nano scales are available. In these cases, the effects of reinforcement of thermoplastic starch matrix by using cellulose, commercial regenerated cellulose fibres are also suggested. One of the main subject of natural fiber reinforced polymer composite is wood-plastic composite industry.

1.6 Introduction to Wood-Polymer Composites (WPCs)

Wood-Polymer Composites (WPCs) materials are combination of the matrix polymers and woods and their individual properties. In general, wood-polymer composite materials can include different ingredients of wood (flour, fiber, or chips), plastics (polymers) and additives such as; pigments, light stabilizers and coupling agents. WPCs are processed via the exactly the same techniques as neat thermoplastic polymers like Polyethylene and polypropylene.

The usage areas of WPCs in different applications has been gradually growing since the 1990s. Thus far, the important markets for wood-polymer composites have been United States of America, eventhough the use of natural fibres and wood-polymer composites are also on the increase in Turkey and Europe markets. In these days, the main usage area of wood-polymer composites are building, construction, transportation and marine to substitute wood in outdoor applications like decking, railings, window and door frames. The usage areas of wood-polymer composite in transportation are automotive or train interior panels, dashboards, and some simple interior materials. Some example products of wood-polymer composites are shown in Figure 1.1 [5].



Figure 1.1: Some example products of Wood-Polymer Composites.

In application of natural cellulose material, using of wood fibres, flours or chips as fillers of reinforcement material into thermoplastic polymers has some advantages by the side of the synthetic and inorganic fillers conventionally used in polymer composites. The natural fibres are abundant, cheap, and light, also they have high specific stiffness and strength. Natural fibres are environmentally friendly; they are renewable, recyclable and biodegradable materials [5].

On the other hand, to use of natural fibres in thermoplastic composites cause some problems. Significant obstruction is the weak interfacial adhesion between the hydrophilic fibres and hydrophobic matrix phase. Another problem is quite difficult dispersion of fibres in the matrix phase. In order to eliminate this problem, they must be dry just before the compounding [5].

1.7 Raw materials

The polymers used are mostly cheap raw material thermoplastics which flow easily when it is melted, polyvinylchloride (PVC), polyethylene (PE), and polypropylene (PP) are the very common and commercial polymers that using in production. In this study, (PP) polypropylene was used.

Polypropylene (C₃H₆)_x is also one of the thermoplastic with a semi-crystalline polymer structure similar to Polyethylene and is manufactured via polymerization of propylene gas. Polypropylene has a very good resistance to stress, low specific gravity, and high mechanical properties such as perfect impact strength. The melting point of PP is 160-165°C, and low density of PP is (0.85 g/cm³ in amorphous structure, 0.95 g/cm³ in crystalline structure) and higher stiffness and strength than High density polyethylene. Polypropylene has a wide range of applications, including food packaging, plastic parts, reusable containers, daily parts, transportation industry and constructions.

As a specific raw material wood used in WPCs is either wood flour, fibres or chips. The term of fibre refers to a single spindle-shaped wood cell, although it is sometimes used to represent wood particles. Wood fibres have characteristic high strength; for this reason, their reinforcing potential is also quite good. On the other hand, Because of high cost and some difficulties in processing step, principally when

feeding to extruder, wood fibres are not used as easy as wood flour in industrial production of WPCs [3].

Flour form of wood is the most popular types preferred material in producing of WPCs; because of cheap and feeding to extruder wood flour is easier when compare to wood fibres. Wood flour contain small wood particles composed of fibre bunch. For this reason, the reinforcing performance of wood flour is not as well as fibres. Wood flour is provided in different particle size; but, generally the particle size of wood flour is between 180 μm to 840 μm . Wood flour can be acquired from the companies which produce product of wood or from some companies especially just produce wood flour [4]. Wood flour is primarily produced from the residues of wood processors, and the main processing stages are generally: 1) size reduction of pure wood with mills and 2) size classification of wood by screening with respect to demand [3].

In addition to wood flour or fibre and polymer as a matrix, WPCs include few amounts of some beneficial additives. These additives positively affect the production and material's performance. Popular additives of WPCs are coupling and foaming agents, lubricants, pigments, light stabilizers. Coupling agents are specifically important in WPCs. Natural fibres are generally hydrophilic materials and they do not bond well with the matrix polymer because of hydrophobic. For this reason; coupling agents are used to increase the interfacial adhesion link between the hydrophilic wood and hydrophobic polymer matrix. In WPCs, generally Maleic anhydride grafted polypropylene (MAPP) is used. MAPP is improve the interfacial adhesion link by these two different mechanisms: 1) anhydride forms an ester bond with a wood cell wall hydroxyl group, and 2) Polypropylene section attached to the anhydride entangles with the Polypropylene network in the melt [4]. Figures 1.2 and 1.3 shows the mechanism of compatibilization of components.

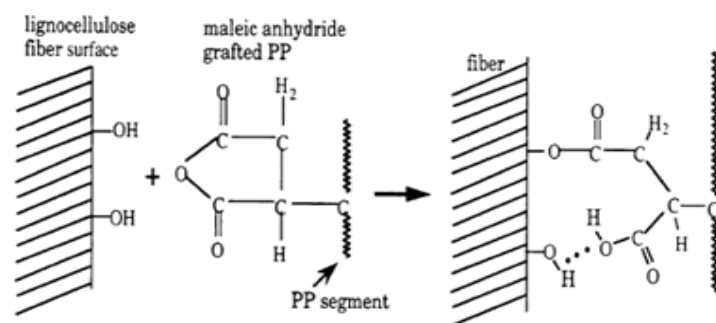


Figure 1.2: Reaction schema of lignocellulose fiber and MAPP [6].

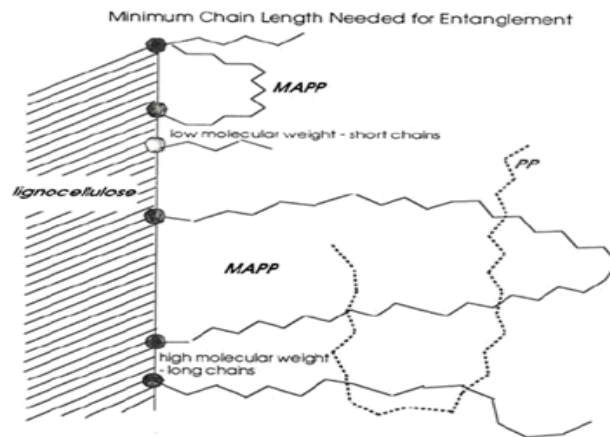


Figure 1.3: Bonding of melted PP to lignocellulose-MAPP combination [6].

As a first step, the anhydride end of the MAPP reacts with a hydroxyl group on the surface of the natural filler forming an ester bonding. After that, the Polypropylene tail on the grafted MAPP gets entangled with the molten thermoplastic resulting in a mechanical bonding between the hydrophobic thermoplastic Polypropylene and the hydrophilic natural fibre which also improve the mechanical properties of the occurring composite [7].

1.8 Advantage and disadvantage of Wood-Polymer Composites

There are lots of variety of wood-polymer composites are used in different industry, the performance and properties of the composites depends on the materials of wood polymer composite. The individual properties of matrix polymer and wood filler determine the total properties of wood-polymer composite. For example; stiffness is particularly influenced by the size, form, dispersion and ingredients of the wood filler in the composite material. The interfacial adhesion forces between the matrix and filler affects the tensile and flexural strength, and long-term properties of wood-polymer composites [2].

Generally, it can be said that addition of a wood improve the stiffness of the polymer matrix phase. On the other hand, when wood component amount increase in the composite, the materials become more brittle. Wood-polymer composites are generally less stiff then solid wood. When fibres form are used in place of wood flour as a reinforcing filler, properties of composite like the tensile and flexural strength, elongation usually increase [8].

The other important advantage of wood-polymer composite is the fungal resistance and dimensional stability is better than solid wood because they absorb moisture more slowly due to the thermoplastic matrix phase. Nevertheless, the mechanical properties of material like a stiffness, creep resistance and strength are lower than those of pure solid wood. For this reason, WPCs are currently used only as non-structural members in construction applications. Nevertheless, lots of research is being done to improve the mechanical performance of WPCs. Also, lots of the research topic is concentrated on studying the service life and durability of WPCs [8].

1.9 Wood and Properties

As a material wood is mainly used for building or construction materials, accessory and vehicles, as raw materials for paper, timber, and many others. Wood is a nature's composite material because of consisting cellulose structure in a combination of lignin, hemicelluloses and some extractives. Wood structure is strong, stiff, tough and lightweight material which can effectively perform tasks critical for tree survival, such as moisture transport. There are lots of wood depending on the species and growth conditions in the world, variations can occur in the properties and quantity of wood component and cell structure. Wood is stiffer, cheaper and stronger material than raw materials of synthetic polymers, when it compare with raw materials of synthetic polymers, because of these properties wood is a very good candidate for polymer filler or reinforcement material [3].

1.9.1 Structure of Wood and Wood Cell

The typical structure of a tree stem is illustrate in Figure 1.4. As can be seen from Figure 1.4, the stem can be divided into three planes: 1) radial surface, 2) transverse surface, and 3) tangential surface. View and properties of wood can be different with respect to plane [9].

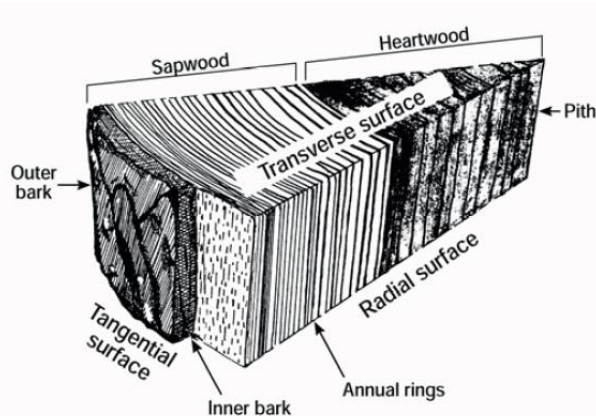


Figure 1.4: The detailed structure of a tree stem with surfaces [9].

As can be seen from Figure 1.4 in the centre of the stem is the pith, which is a small core of soft tissue formed during the first growth year. The pith is surrounded by heartwood. This part include dead wood cells and function of this part is mechanically supporting to tree. As a color heartwood is generally darker than sapwood because of the fact that resinous organic compounds residue in its cell walls. The section of tree which is called ‘sapwood’ is the physiologically alive part, and also a structural support, it perform as a food tank and it provides water transport up from the roots. The cambium section is a thin layer of tissue between the sapwood and bark. Cell divisions in the vascular cambium result in wood thickness growth. The inner bark, or the phloem, is a narrow layer of tissue through which the sap moves up and down. Inner bark is generally light coloured. The outer bark which is called ‘rhytidome’ contain dead wood cells so it is dar coloured [9; 10].

Cambium has a growth cycle yearly in temperate climates, which is the reason for the annual rings of a tree. The volume of wood growth is generally very fast in the beginning of the growth step and it started to slow and stops at the end of the growing season. Because of this reason, colour changed from light to dark wood each year. If the wood formed early is called earlywood, if the wood formed late is called latewood with respect to season [9].

Wood cells can be divided into three categories with respect to their function in a tree:

- 1) Storage cells
- 2) Conducting cells
- 3) Supporting cells.

Prosenchymatous (prosenchyma) is the name of conducting and supporting cells, *parenchymatous* (parenchyma) is the name of storage cells. Prosenchyma cells are thin and long cells which are narrower at the ends, and parenchyma cells are short cells which are either round or rectangular shapes [11]. In all wood cells there are also small *pits*, recesses which interrupt the secondary wall. Through these pits fluids and gases are able to pass from cell to cell. [9] Hardwoods and softwoods differ somewhat in terms of cell structure. Generally, structure of hardwoods is complex than and a larger number of different kinds of cells than softwoods structure [9].

1.9.2 Chemical Composition

The major ingredient of wood are;

- Cellulose,
- Hemicelluloses,
- Lignin
- Extractives.

The proportion of each constituent as percentage in hardwood and softwood are shown in Table: 1.1.

Table 1.1: Chemical composition of typical wood [10].

COMPONENT	SOFTWOOD	HARDWOOD
Cellulose	42 ± 2 %	45 ± 2 %
Hemicellulose	27 ± 2 %	30 ± 5 %
Lignin	28 ± 2 %	20 ± 4 %
Extractives	3 ± 2 %	5 ± 3 %

Cellulose is the most abundant and the main structural part of wood. Cellulose determines the character of fibers. The molecule of cellulose is long and straight linear chain of homo-polymer contain three elements which are called hydrogen, oxygen and carbon. They are organised into anhydro d-glucofuranose linked by β 1, 4 glycosidic bonds (Figure 1.5). The chemical formula of cellulose is $(C_6H_{10}O_5)_n$, where n is the indicate the repeating sugar units number also it shows the DP ‘degree of polymerization’. The repeating unit contain two glucose molecules. Name of this unit is *cellobiose*. The degree of polymerization can be change depending on the source of cellulose and the treatment it has received. The properties of cellulose related to the degree of polymerization of cellulose [10].

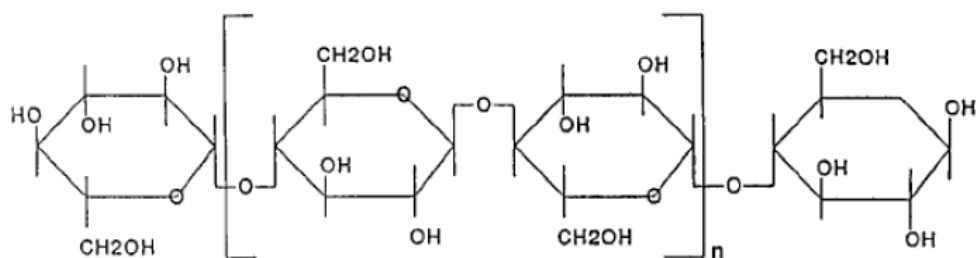


Figure 1.5: Typical structure of cellulose molecule [12].

Hemicelluloses contain a collection of polysaccharide with lower DP than cellulose molecule, so it has lower molecular weight. Structure of hemicellulose is very similar to that of the cellulose, it is consist of five carbon sugars (glucose, mannose, galactose, xylose, and arabinose) in chains. However, chains are comparatively short and less regular then the cellulose, for this reason, they aret easily degrades and dissolves more easily than cellulose. The DP is only tens or hundreds of repeating units. The other part of wood is Lignin. It is an amorphous polymer with a very complex structure. It holds cellulose fibres together like kind of binding agent because of it is a brittle and relatively inert material acting. The affect of lignin improve the stiffness of wood cell and allows for stress transfer between matrix and fillers in the WPCs when it diffuse the fibre wall. It is also consist of carbon, hydrogen and oxygen elements.

Except that cellulose, lignin and hemicellulose, some substances, e.g. fatty acids, resin acids, alcohols, are additionally present in wood. These compents are called extractives, and they are provide some properties to wood like a odor, color, density, taste, decay resistance and flammability. They can be removed from the wood by the method of extractions with solvents, so ther are called “extractives”.

1.9.3 Properties of pine (*Pinus sylvestris* L.)

It is an evergreen tree that develops to 30-40 m with an erect, tube shaped, exposed and straight trunk beaten by an adjusted or flattopped mass of foliage. The bark is textured dim dark brown outwardly and ruddy inside, with pieces and crevices [13].

Buds are reddish. Leaves are industrious acicular, inflexible, dim green gathered on the tip of solid shoots, turned by sets round in cross segment no longer than 6 cm. Flowers have male inflorescences of sulphur-like yellow. Each scale represents a female flower. It has a place with the Pinaceae family. It sprouts in spring and seeds

maturation take place at fall of the next year. This is the most popular pine in Europe, Asia and Turkey [13]. This kinds of pine especially abundant in blacksea, and Anatolia region of Turkey.

1.9.4 Properties of hornbeam (*Carpinus betulus*)

Hornbeam, any of about 25 species of tough, moderate developing decorative and timber trees comprising the class *Carpinus* of the birch family (Betulaceae), disseminated all through the Northern Hemisphere. The hop-hornbeam (*q.v.*) is in a different genus of the birch family. A typical hornbeam has smooth, grayish bark, a short, fluted trunk, and evenly spreading branches. A leaflike, three-lobed husk surrounds each fruit, a nutlet about 1 cm ($\frac{1}{2}$ inch) long. The egg- or lance-shaped alternate leaves may remain on the tree into winter [14].

The hornbeam (*C. betulus*) in Europe has a twisted trunk that branches profusely; the tree may grow to 20 m (65 feet). Generally European hornbeam abundant in Marmara region. Hornbeam are growned Ege, marmara and trakya regions of Turkey.

1.9.5 Medium density fibreboard (MDF)

Fibreboard, in all its densities, is made from wood fibres. Those would be little tiny pieces of hardwood or softwood. Mix these with some resin and wax, then heat it up and press it together, and you get fibreboard [16].

1.10 Wood Waste Arising and Sources

Municipalities, commercial users, industrial users especially, after construction as a waste wood can be residue in many vary forms. Some reviewed reports have tried to count the overall Turkey wood waste (tonnages), and to the extent possible, identify the quantities increasing from sources. The most common wood waste arising after used MDF, Pines and hornbeam. There is a statistical data in city of Balıkesir / Turkey.

Table 1.2: The ratio of the MDF and Pine amount to other wood amount [17].

MDF	Pine	Others
39.21%	35,05 %	25,74%

According to the Balıkesir Carpenters Chamber of Tradesmen and Craftsmen Report amount of waste total for 2018 is 390648kg.

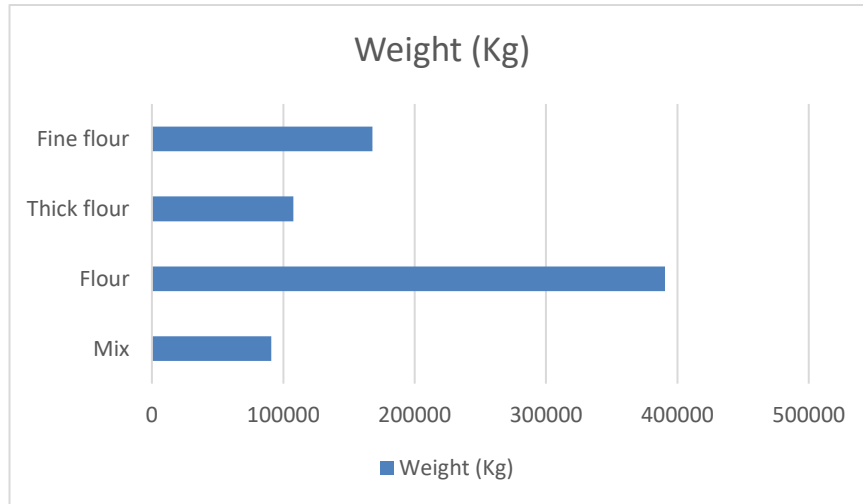


Figure 1.6: Annual waste wood flour weight in 2017 Balıkesir [17].

Figure 1.6 show us; waste amount of flour is 390648 kg, thick flour amount 109730 kg, fine flour amount 167772 and mix flour amount is 90972 kg [17].

1.11 Production of Wood-Polymer Composites

Production of WPCs generally consists of three stages:

- 1) Raw materials processing,
- 2) Compounding stage,
- 3) Moulding of the products by injection or compression moulding and profile extrusion [2].

The aim of the raw materials (wood) processing stage is to improve as a raw material wood quality by separating it with respect to different species and sizes and drying it prior to the stages of compounding.

In compounding stage the basically composite is produced by blending the raw materials like reinforcement or additives with the matrix polymer. For this main step of production twin-screw extruder is used. Examples of the twin-screw extruders are; parallel co-rotating, conical counter-rotating and parallel counter-rotating. After the compounding stage, material can be fastly pressed or shaped as an end product or formed into pellets in order to use next processing. As a last stage, these products can be produced for different applications like; profile extrusion, injection building or compression moulding [2; 4].

Extrusion is the most popular method for manufacturing of Wood-polymer composite. In this method, the raw materials are melted and compounded, then extruded into a continuous profile by forcing by the engine the molten material through the die in the same process step [4]. In direct extrusion either profiles or sheet materials for compression moulding can be produced. A schematic illustration of an extrusion line for production is shown in Figure 1.7 [2].

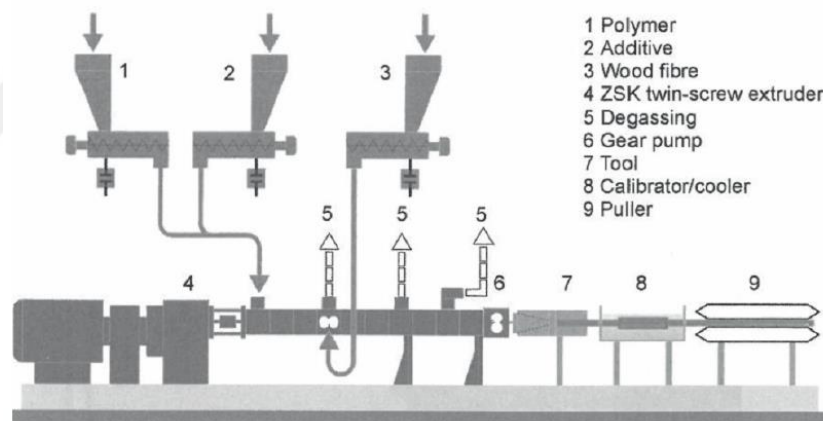


Figure 1.7: Typical schema of compounding and extrusion [5].

In the method of pelletizing, raw materials are melted and compounded before to moulding steps to shape a product. The common method of producing pellets is the strand pelletizing method, since the composite melt is pressed through to holes, after that strands passing to a water bath to cool. As a next of cooling step the strands are carried to an air blowing unit to help to reduce excessive water on products, and then the strands are fed into a pelletizer unit. As a last step strands can be cut by the extra machine into small granules. The pellets can be used in extrusion or injection moulding with respect to applications [5].

Mengelöglu et al [18] the most common problem of wood polymer composite processing is nonconformity. Although wood are hydrophilic, polymers are hydrophobic materials. The mixture of these two different material can not be homogeneous, however it can be mix by the help of coupling agents. In order to obtain homogenous mixture as a coupling agent MAPP is used.

Dalvag et al [19] in wood-polypropylene composites, if as a coupling agent MAPP is use, tensile strength, flexural strength and elastic modulus can be show better performance. Han et al [20] they reported that using of MAPP as a coupling agent in PP/cellulose composites by the action of reactive hydroxyl groups on the fill surface, mechanical properties of composite were improved. Bledzki et al [21] investigated that effect of MAPP on wood polymer composite mechanical properties and released that when use small amount MAPP became more effective. A.K. Bledzki et al. [22] reported that three compounding techniques (two-roll mill, high-speed mixer and twin-screw extruder) were tested for preparation of Wood flour and PP composites. The best mechanical strength and lowest values of absorbed water exhibited composites compounded in extruder. Arrakhiz et al [23] results show a clear improvement in mechanical properties from the use of both alkali treated Pine cone and Pine cone compatibilized with maleic anhydride, a gain of 43% and 49% respectively in the Young's modulus, as a results of improved adhesion between fibers and matrix at the interface.

2. MATERIAL and METHODS

2.1 Materials

2.1.1 Wood flours of Hornbeam, Pine and MDF.

As a filler of composites, Hornbeam (*Carpinus betulus* L.), Pine (*Pinus sylvestris* L.), Medium density fibreboard (MDF) sawdusts have been taken from the residues of furniture workshop (OSABYA Design). MDF lamina was purchased from STARWOOD construction market in November 2018. Typical sample of wood flour and wood chips is shown in Figure 2.1.



Figure 2.1: Wood flour and wood chips

2.1.2 Matrix Phase, Polypropylene

In this study, Polypropylene (Petoplen EH 251), density 0.905 g/cm³, melt flow index 24 g/10 min. (230°C/2.16 kg), T_m=163 °C, Flexural Modulus 1450 MPa and Tensile strength at yield 38 MPa was used as composite matrix. Polypropylene was purchased from PETKİM Petrochemical Holding A.Ş. Typical PP pellets are shown in Figure 2.2.



Figure 2.2: Polypropylene pellets.

2.1.3 Coupling Agent

As a coupling agent Maleic Anhydride-Modified Polypropylene (Bondyram 1001), was purchased from RESINEX BMY A.S. It is a kind of Homo-polypropylene recommended for coupling of glass and other fillers in polypropylene composites. Typical properties data are shown in Table 2.1.

Table 2.1: MAPP Properties Data

Property	ISO Test Method	Unit	Bondyram 1001
Melting Flow Index	1133, 190° C/2.16 kg	g/10min	100
Density	1183	g/cm ³	0.90
Melting Point	DSC	°C	160
Maleic Anhydride level	FTIR	%	1

2.2 Processing of wood-polymer composite and samples

LabTech Engineering Company LTD. Twin-Screw Extruder (screw diameter: 20mm, L/D ratio of 32:1) was used to produce wood-polypropylene (Wood/PP) composites. First of all, pure sawdust of hornbeam, pine and MDF collecting by Koparan KP2000 dust collector in workshop. Secondly, polypropylene, MAPP and wood sawdust separately were put into LabTech Engineering Company LTD mixer for 5 minutes to make it homogeneous. Laboratory mixer is shown Figure 2.3.



Figure 2.3: Laboratory Mixer

Then, the prepared mixture was placed to the hopper of extruder and feed with 20 rpm. Zone temperatures of extruder were selected between the temperatures 170°C and 185°C. Screw speed is adjusted as 190 rpm. Pressure was 10bar. Twin screw co-rotating is shown in Figure 2.4.



Figure 2.4: Twin screw Co-rotating Extruder

After the extruder die, composite was cooled with water in cooling bath and transferred to pelletizer to obtain pellet type composite materials.



Figure 2.5: Product in spaghetti form **Figure 2.6:** Pellet machine

The amounts of compositions are shown in Table 2.2.

Table 2.2: Composition and amount of samples

Sample name	Composition of Wood	Composition of MAPP	Hornbeam amount	MAPP amount	Polypropylene amount
Neat	0%	0%	0	0	500g
H10M3	10%	3%	50g	15g	435g
H20M3	20%	3%	100g	15g	385g
H30M3	30%	3%	150g	15g	335g
H30M6	30%	6%	150g	30g	320g
P10M3	10%	3%	50g	15g	435g
P20M3	20%	3%	100g	15g	385g
P30M3	30%	3%	150g	15g	335g
P30M6	30%	6%	150g	30g	320g
M10M3	10%	3%	50g	15g	435g
M20M3	20%	3%	100g	15g	385g
M30M3	30%	3%	150g	15g	335g
M30M6	30%	6%	150g	30g	320g

Notifications: H: Hornbeam, P: Pine, M: MDF, P10M3 means %10 Pine %3 MAPP.



Figure 2.7: Pellets of Pine, Hornbeam and MDF

As a last step, hot press was used to form the composite panels from compounded pellets. Then hot press is heated to the until processing temperature reach (200°C), manufactured composite pellets were put into the mold cavity (15 cm x 15 cm)

between teflon sheets which were used to avoid direct contact of PP composites with the hot press metal platens during heating and pressing.



Figure 2.8: Hydraulic Laboratory Press Machine

Then, plates of sample were obtained from the mixture by using Hydraulic Laboratory Press [Labtech] via heating and cooling platens. Samples were pressed under 40 bar pressure with 200°C upper and lower temperature of heating platens for 20 seconds in hot part and 2 minutes in cold part of the press. Therefore, samples were ready for test and characterization. They are shown in Figure 2.9.



Figure 2.9: Samples of Neat, MDF, Hornbeam and Pine plates

2.3 Characterization

2.3.1 Tensile testing

Tensile testing samples were obtained by using a mould which cut the plate with blades with respect to standard of ASTM D638. Tensile test machine SHIMADZU AGS-X 5kN (Figure 2.10) was used for wood reinforced composites at room temperature with a crosshead speed of 50 mm/min. From each composite samples, 2 specimens were tested to obtain to check the result.

2.3.2 Flexural testing

The flexural strength and flexural strength of samples were tested with respect to DIN EN ISO 178 in SHIMADZU AGS-X 5kN (Figure 2.10.) machine, which include a three-point bending test at a crosshead speed of 1 mm/min. The dimension of samples was 25 mm×40 mm. Two samples of each group were tested to check.

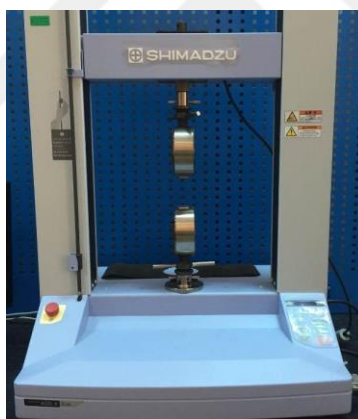


Figure 2.10: Tensile and tree-point bending device

2.3.3 Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR technique was used to study the main functional groups present in materials used in this study. Thermo Scientific™ FTIR spectrometer (Figure 2.11) was used to obtain the spectra of woods, MAPP and PP. Attenuated total reflection infrared (ATR-IR) spectra of the materials were collected at a resolution of 4 cm^{-1} and a number of 16 scans per sample. Spectral outputs were recorded in absorbance mode as a function of wave number.

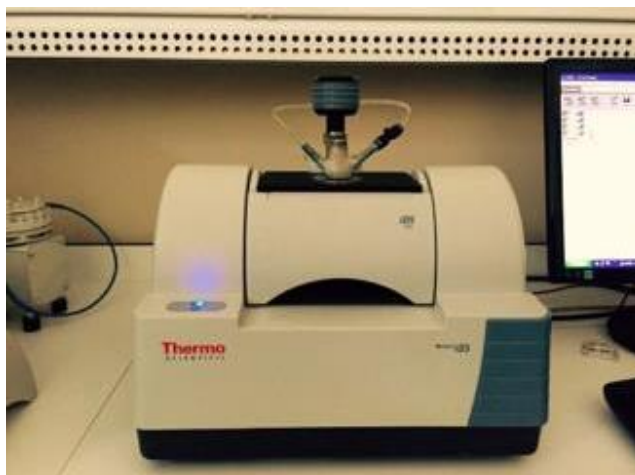


Figure 2.11: FTIR Device

2.3.4 X-ray diffraction (XRD) analysis

The XRD analysis of “as received” and ECAPed samples were performed by Bruker D2 Phaser X-ray diffractometer (Figure 2.12) with $\text{CuK}\alpha$ radiation ($\lambda=1.5406 \text{ \AA}$) at 30kV in the 0.02° intervals of $5\text{--}90^\circ$. In the present study, XRD analysis was performed to determine their phase content and to understand that how the crystallographic orientations of the grains changes after the ECAP process of the samples (Figure 2.12).



Figure 2.12: X-ray diffractometer (XRD) (Bruker D2 Phaser).

2.3.5. Scanning electron microscopy (SEM)

Scanning electron microscopy (SEM) was used to observe the microstructure of Neat PP and polymer composites. SEM images were taken from the fracture surfaces of the tensile specimens Carl Zeiss 300 VP (Figure 2.13) was used as SEM. The samples were coated with gold and the images and accelerated voltage was adjusted as 30 kV and images were taken at different magnifications.



Figure 2.13: Scanning Electron Microscopy

3. RESULTS and DISCUSSION

3.1 Mechanical Properties

3.1.1 Tensile Testing

Table 3.1: Tensile test result of Wood-polymer composites.

	Max Stress (MPa)	Strain (%)	Elastic Modulus (MPa)
PP	38.16	3.28	1360.71
H10M3	36.74	1.67	1554.08
H20M3	38.69	1.71	1672.13
H30M3	36.86	1.29	1715.62
H30M6	39.65	1.30	1813.20
P10M3	32.63	1.44	1395.95
P20M3	31.65	1.15	1594.09
P30M3	29.65	0.78	1762.92
P30M6	33.22	0.89	1862.47
M10M3	34.13	1.46	1446.77
M20M3	39.89	1.62	1752.11
M30M3	40.81	1.59	1938.71
M30M6	42.41	1.52	2050,04

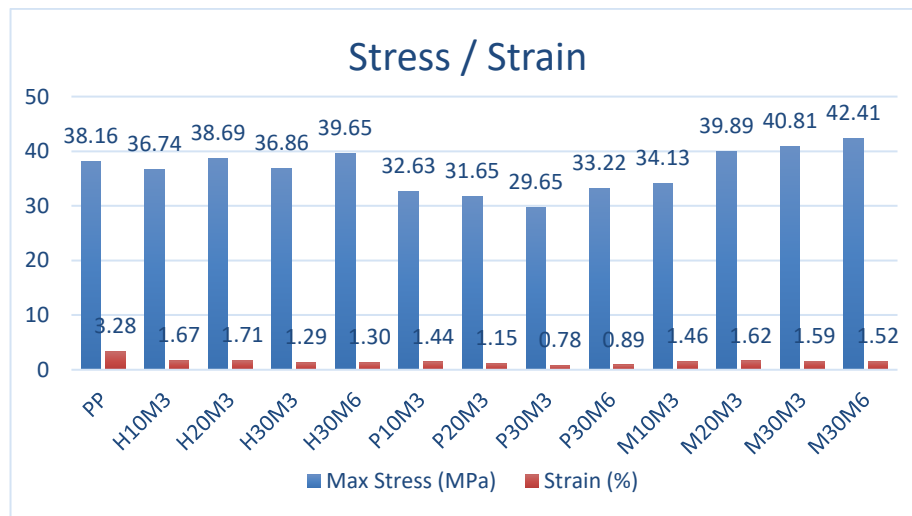


Figure 3.1. Tensile Stress / Strain graphic of Wood-polymer composites.

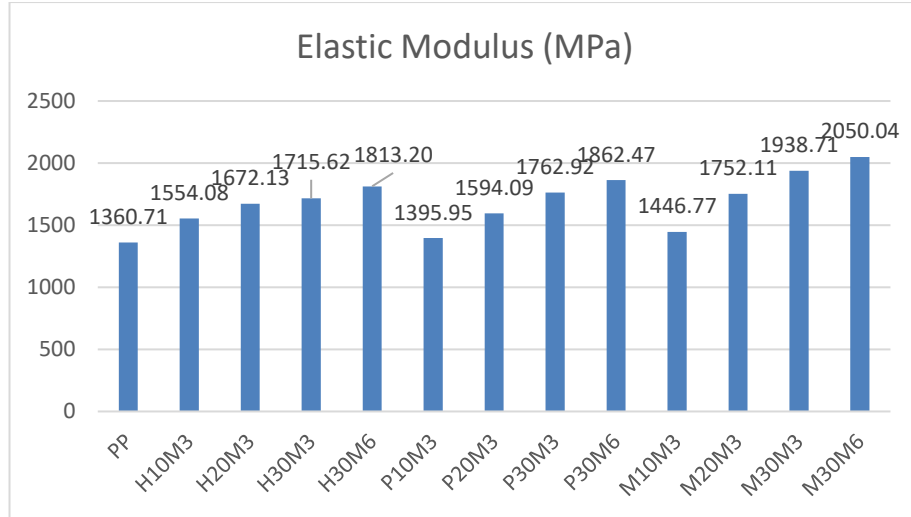


Figure 3.2. Elastic Modulus graphic of Wood-Polymer Composites.

Tensile strength, maximum load that a material can support without fracture when being stretched, divided by the original cross-sectional area of the material [15]. In Hornbeam / PP composites, tensile strengths are very close to each other. Effect of hornbeam on the tensile properties of WPC very few. Max tensile strength at H30M6 because MAPP improve interfacial adhesion of Hornbeam / PP. As an Elastic modulus (Stiffness) increase with respect to amount of wood. When MAPP amount increase Stiffness of material increase. Strain was decreased because of weak bounding between matrixes and coupling agent.

In Pine/PP composites, tensile strengths and strain are getting reduced with respect to amount of pine flours. Also, they are lower then neat PP. On the other hand, elastic modulus increased when pine flour amount is increased. When MAPP amount increase stiffness, strain and elastic modulus of material increase.

In Medium Density Fiberboard, tensile strengths and elastic modulus increased with respect to amount of MDF. Strain are very close to each other. Effect of MAPP is quite positive. When MAPP additives increase, tensile strength is increased.

As a result, when amount of MAPP is increased from 3% to 6% elastic modulus and tensile strength of three different wood types composites also increased. MAPP is make strength bounding to polymer matrix. This result was supported in the study Ayrilmis et al.,(2013) when MAPP amount increase, tensile strength also increase [24].

3.1.2 Flexural testing

Table 3.2: Flexural test result of Wood-polymer composites.

	Max Stress (Mpa)	Strain (%)	Flexural Modulus (Mpa)
PP	52.23	8.20	1308.24
H10M3	59.26	7.10	1749.81
H20M3	57.33	5.76	1700.20
H30M3	55.55	4.63	1862.10
H30M6	58.81	5.35	2012.92
P10M3	53.34	7.97	1573.25
P20M3	52.61	5.97	1834.93
P30M3	50.85	4.30	1941.41
P30M6	54.14	4.37	1932.66
M10M3	58.42	7.34	1656.09
M20M3	58.76	6.31	1878.80
M30M3	63.18	5.55	2103.03
M30M6	62.05	5.65	1982.23

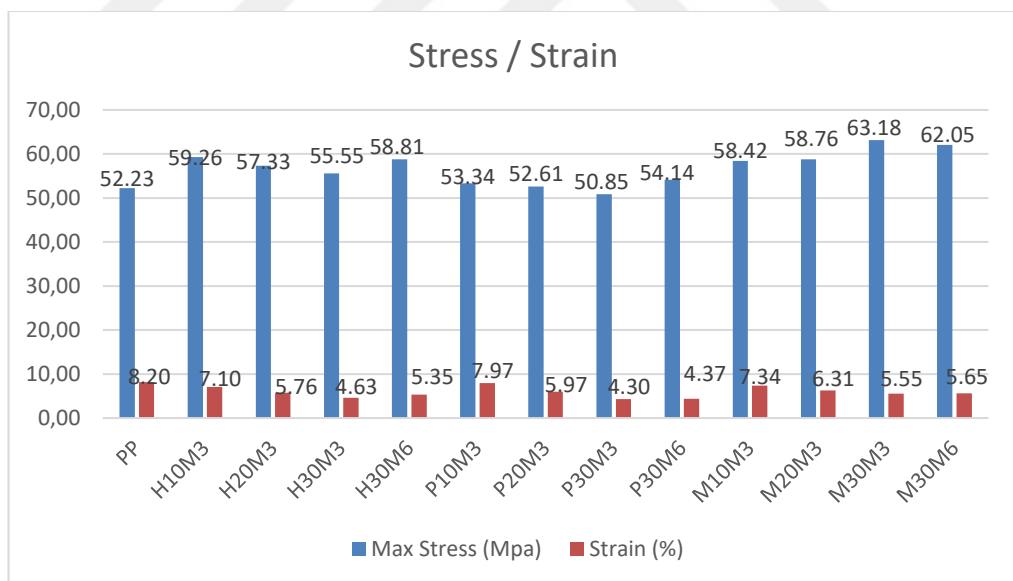


Figure 3.3: Flexural Stress /Strain graphic of Wood-polymer composites.

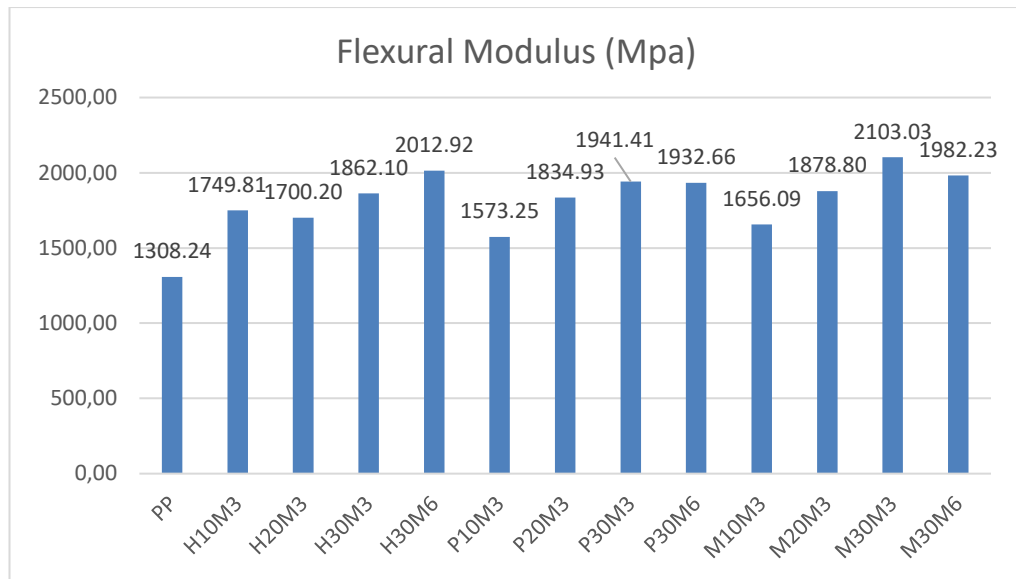


Figure 3.4. Elastic Modulus graphic of Wood-Polymer Composites

Flexural strength and elastic modulus values of composite samples are shown respectively in Figure 3.3 and Figure 3.4. Flexural strength (Stress) PP with hornbeam higher than neat pp, then reduce with respect to amount of hornbeam. If we compare H30M3/H30M6 MAPP improve interfacial adhesion of PP/Wood so Stress, Strain and Flexural modulus was increased. As a Flexural modulus (Stiffness) increase with respect to amount of wood except H20M3. When MAPP amount increase, stiffness of material increase. Strain was decreased because of weak bounding between matrixes and coupling agent. Another reason some gap badly affect to material. In Pine /PP composite sample, although elastic modulus increase, flexural strength and strain is reduced when amount of wood flour increase. Also, similar case was observed in tensile test. As a wood flour, pine is badly affect to mechanical properties of composite. Even this case, when MAPP amount increase, flexural strength, strain and flexural modulus increase.

In Medium density fibreboard / PP composite sample, tensile strength and elastic modulus increase with respect to increasing amount of MDF except M30M3 and M30M6. It is expected to increase mechanical properties when MAPP is increase, but in this case reverse effect was observed. Probably some gap or bounding problem badly affect the flexural strength and elastic modulus.

3.2 Scanning Electron Microscopy

3.2.1 Hornbeam / PP Composites

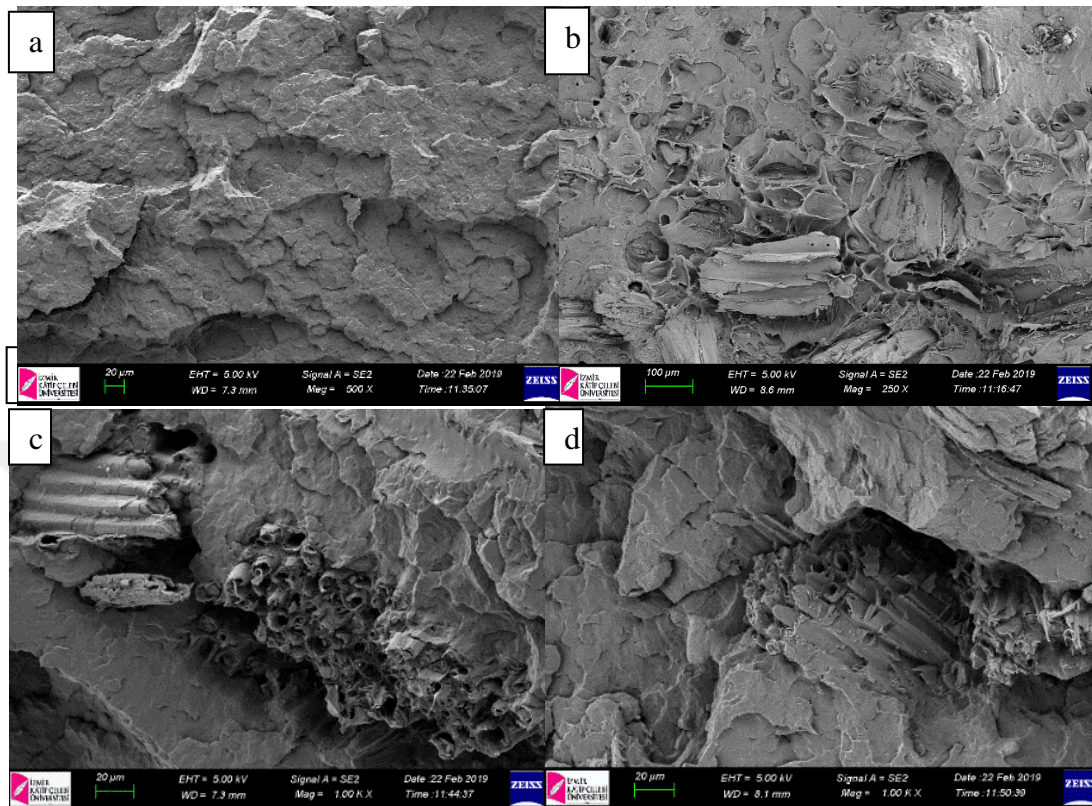


Figure 3. 5. SEM images of Hornbeam and Polypropylene composites samples
a) Neat PP b) H10M3 c) H30M6 d) H30M6

SEM images show us for H10M3 it is good dispersion of Hornbeam in the Polypropylene matrix. The degree of agglomeration decreases as the hornbeam and PP matrix is compatibilized.

In the H30M3 and H30M6 have some air gaps, they causes to affect mechanical properties. It was fractured on gaps. H30M6 more compatible then H30M3 because of effect of coupling agent (MAPP) amount higher so interfacial adhesion bounding better than H30M6.

3.2.2 Pine / PP Composites

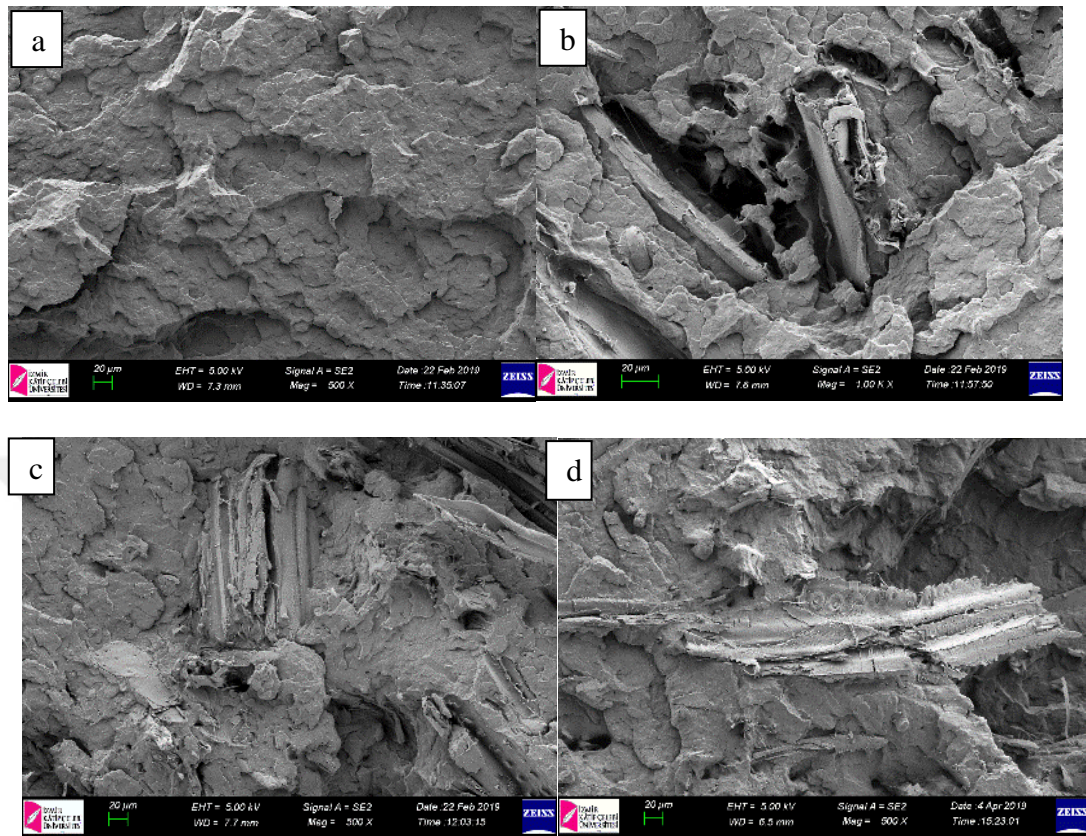


Figure 3.6: SEM images of Pine and Polypropylene composites samples a) Neat PP
b) P10M3 c) P30M3 d) P30M6

SEM images show us for P30M3 it is good dispersion of Pine in the Polypropylene matrix. In the P10M3 and P30M6 have some small air gaps, they causes to affect mechanical properties. P30M6 more compatible then P30M3 because of effect of coupling agent (MAPP) amount higher so interfacial adhesion bounding better than P30M6. It is shown that P30M3 and P30M6 polymer matrix and wood flour made strong adhesion.

The reason of air voids within the structure could be explained as polymer was not subjected to drying process prior to extrusion, and therefore polymer would absorb moisture from the air. Another solutions can be mixed more or increasing operation pressure of extruder.

3.2.3 Medium Density Fiberboard (MDF) / PP Composites

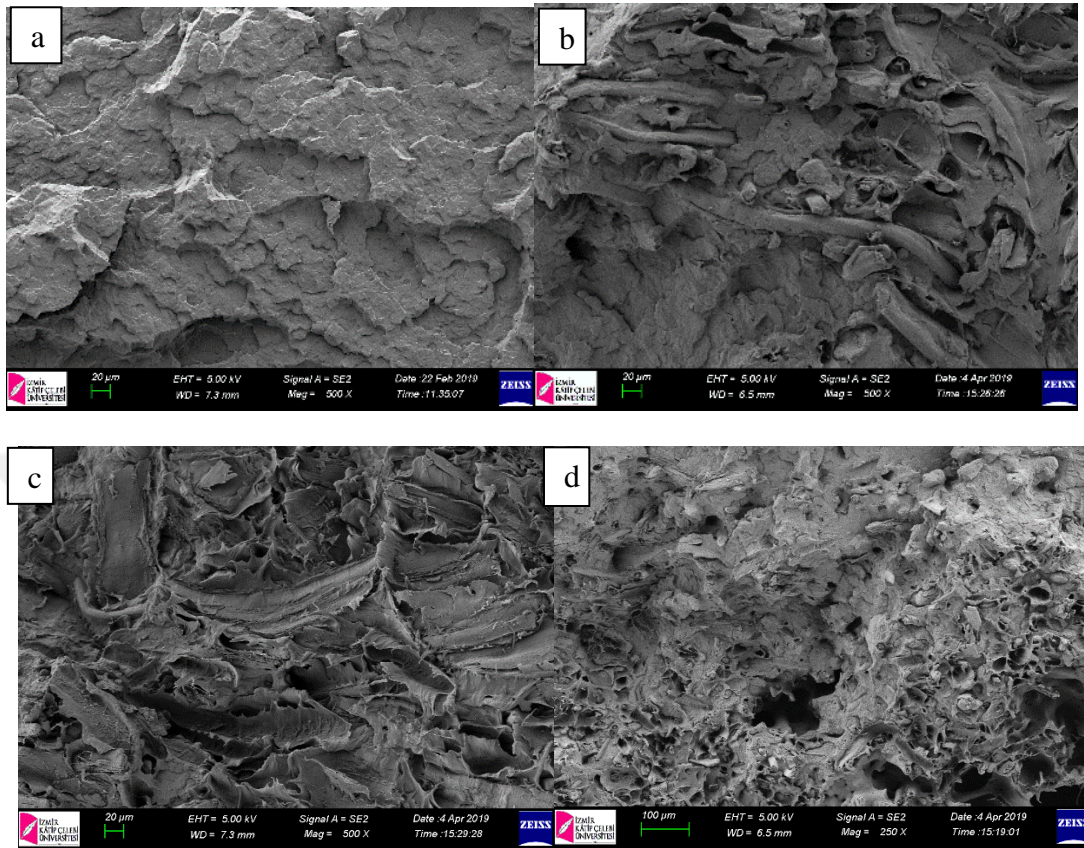


Figure 3.7: SEM images of MDF and Polypropylene composites samples a) Neat PP
b) M10M3 c) M30M3 d) M30M6

Medium density Fiberboard is consist of mix wood fibres, because of this reason it could shown in figures, it is very good dispersion. It is shown that M10M3 and M30M3 polymer matrix and wood flour made strong adhesion bounding. In M30M6 some gaps was shown in figure d.

The reason of air voids within the structure could be explained as polymer was not subjected to drying process prior to extrusion, and therefore polymer would absorb moisture from the air.

3.3 Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR technique was used to study the main functional groups present in wood fibres composites with coupling agents. Chanel stated that the bands assigned in O-H stretch at 3300 cm^{-1} , the C-H asymmetric stretch at 2900 cm^{-1} , the C-H symmetric stretch at 2850 cm^{-1} , the C=O stretch of a carboxylic acid and ester at 1700 cm^{-1} , and the C=O stretch of an acid salt at 1600 cm^{-1} [25]. The most distinct spectral peaks about wood fibers are shown in Table 3.3.

Table 3.3. Infrared band assignment for the cellulosic fibres.

Position / cm^{-1}	Assignment	References
~3335	Lignin and cellulose Intra-molecular OH stretching vibration	[26,27,30]
~2900	C-H asymmetrically stretching vibration in cellulose and hemicellulose	[26-28]
~1635	Adsorbed water and oxygen-containing group	[27-30]
~1735	Stretching vibration of C=O Hemicellulose or carboxylic acid	[26-28,30]
~1060	C-O, C-H stretching vibration of Cellulose	[28-30]
~1163	Cellulose and hemicellulose C-O-C bending vibration	[30-32]
~1372	Cellulose and hemicellulose C-H bending vibration	[29-32]
~2899	Cellulose CH stretching vibration	[29-32]
~1315	CH in plane deformation of CH ₂ groups	[33-34]
~898	Correspond to β -1, 4 glycosidic linkages between sugar units in hemicelluloses and celluloses	[33-34]

The FTIR spectroscopic analysis of neat PP, Hornbeam / PP, Pine /PP, MDF/PP composites are shown in respectively, Figure 3.8.-3.9-3.10.

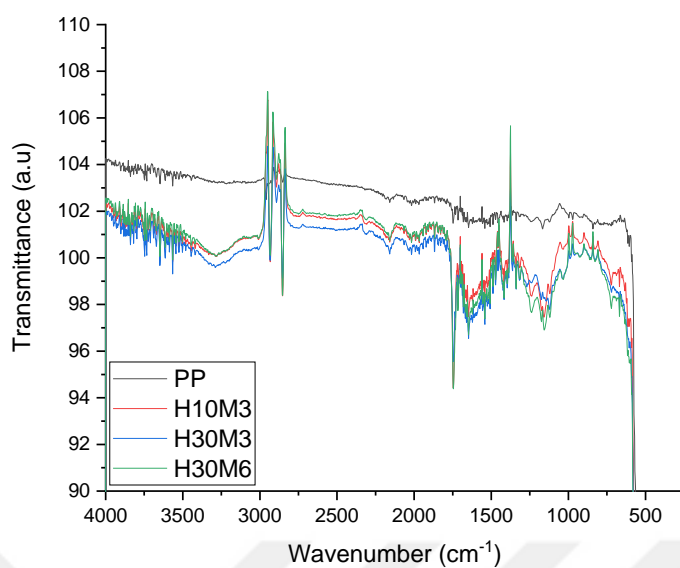


Figure 3.8. FTIR spectra of PP, H10M3, H30M3 and H30M6.

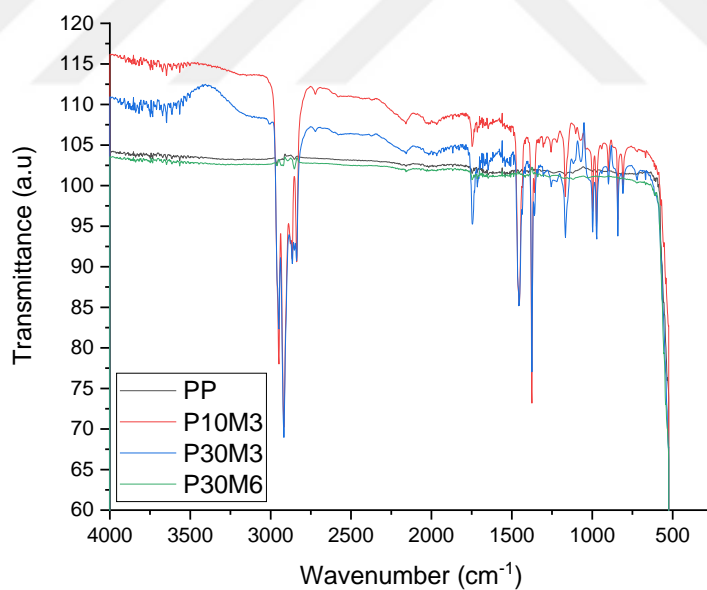


Figure 3.9: FTIR spectra of PP, P10M3, P30M3 and P30M6.

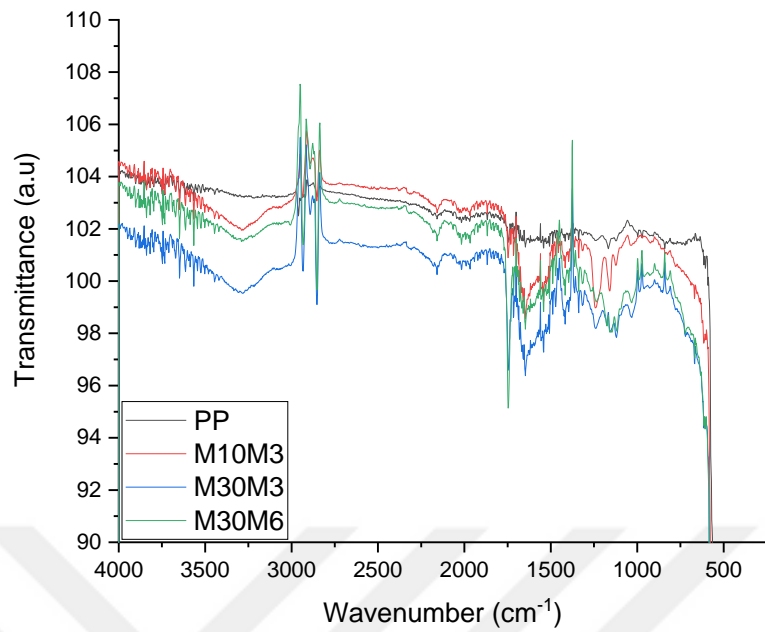


Figure 3.10: FTIR spectra of PP, M10M3, M30M3 and M30M6.

Woods are organic materials. For this reason in the three different wood types of composites at nearly 2900 cm^{-1} C-H asymmetrically stretching vibration of CH and CH_2 groups in cellulose and hemicellulose are shown in spectras [31].

3.4 X-Ray Diffraction Analysis

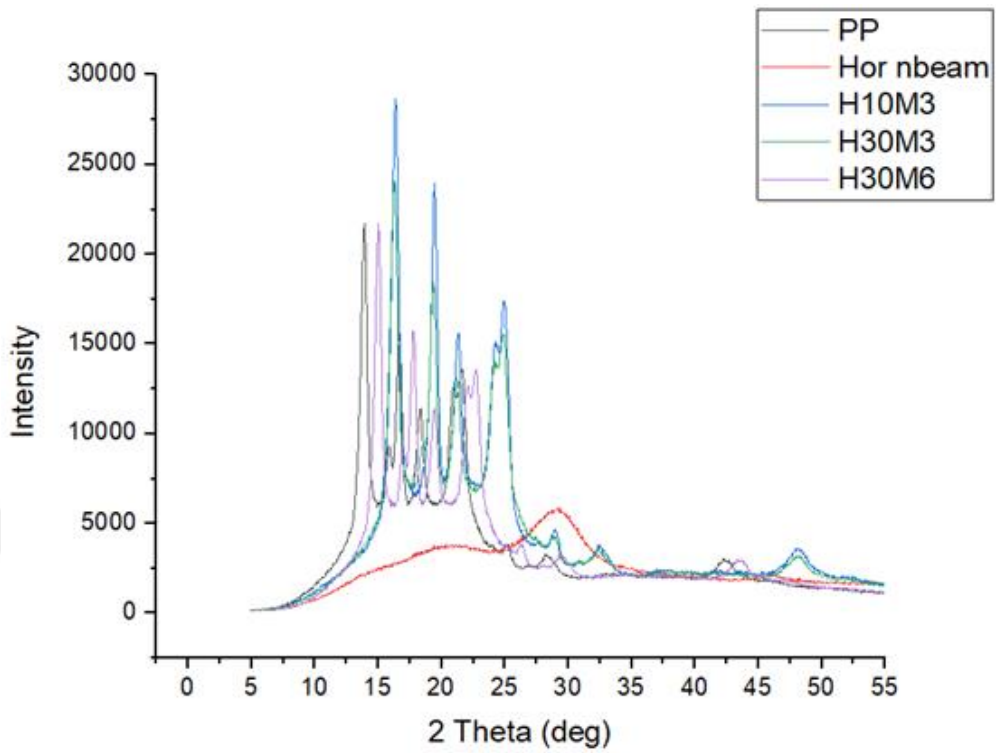


Figure 3.11: XRD patterns of Hornbeam and polypropylene composites.

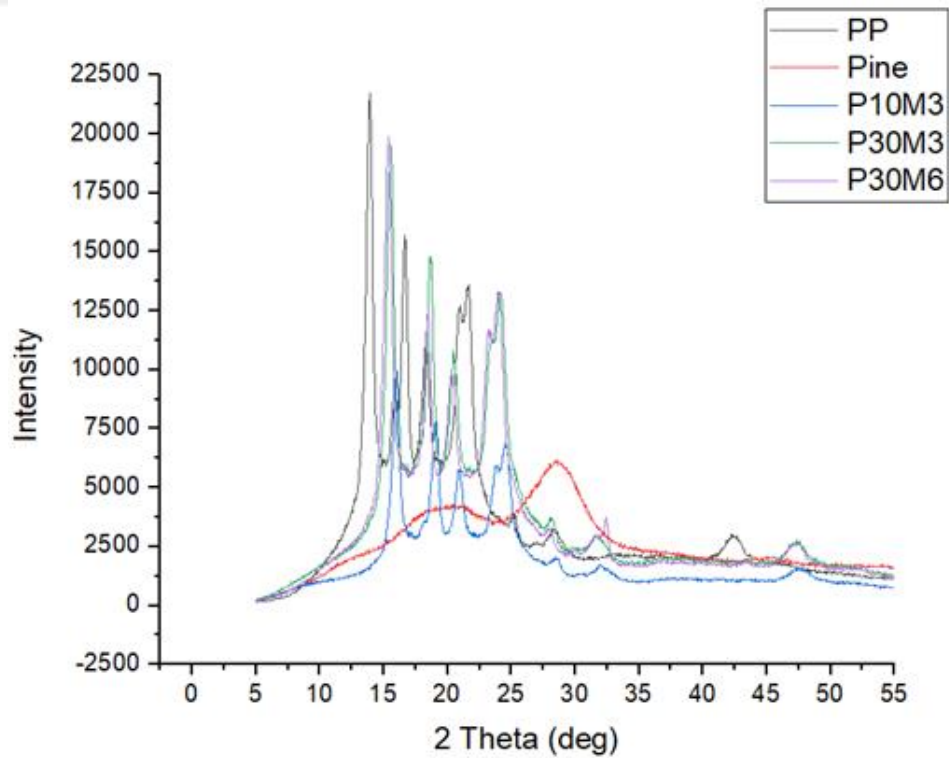


Figure 3.12: XRD patterns of Pine and polypropylene composites.

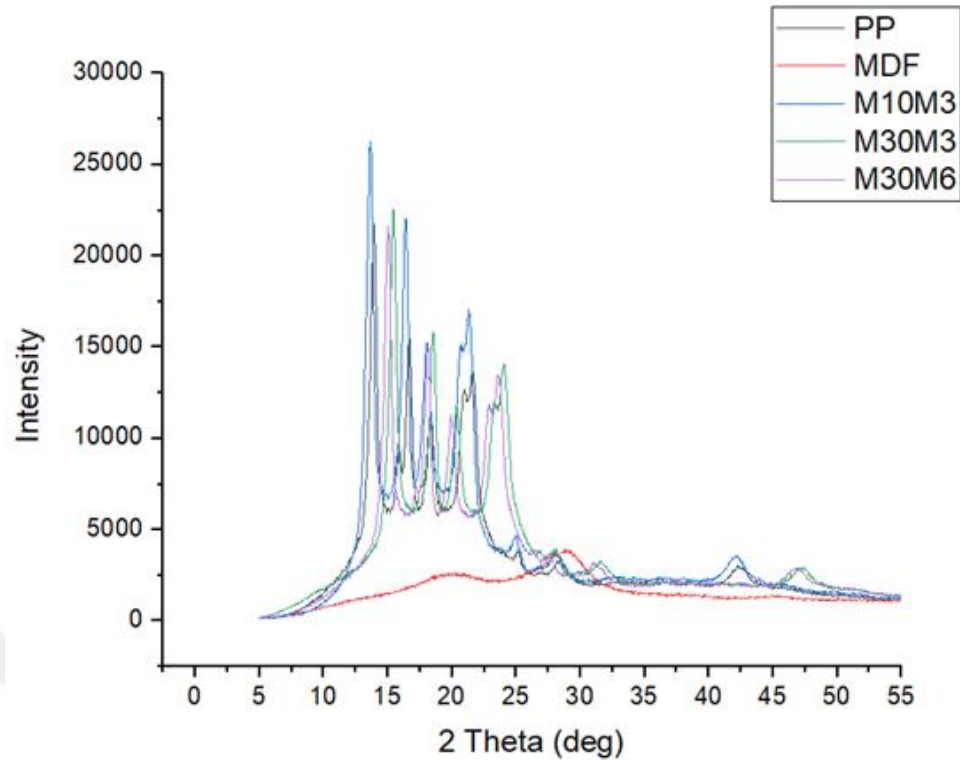


Figure 3.13: XRD patterns of Medium density fiberboard and polypropylene composites.

X-ray diffraction is useful, very simple and widely used characterization technique for determination of wood flour dispersibility. Intensity versus 2θ values graphs were obtained after XRD analysis. Figure 3.11, 3.12 and 3.13. Shows respectively, XRD patterns of neat PP, Hornbeam, pine and MDF. Pure PP and PP-wood composite shows very similar pattern, peaks occurs almost same locations. Peaks occurred at 15° , 17° , 19° , 21° , 25° , 29° and 42° , PP and composites shows semi crystalline forms.

Intensity of PP is lower than PP-Hornbeam composites. Intensity of PP is higher than PP-Pine composites. Intensity of PP almost same like PP-MDF composite. Intensity of H30M3, P30M3 and M30M3 higher than H30M6, P30M6 and M30M6 at 19° . It means when amount of MAPP increase from 3% to 6% these peaks intensities are reduced. L. Danyadi et al. [34] reported that wood was shown to modify the crystalline structure of PP and to induce transcrystallization. Same affects were shown in our study.

4. CONCLUSION

The aim of this study was to prepare wood-polymer composites and discussed their possible to use waste woods as a raw material. Within the developments in the technology, woods became very popular for Material Science, which have many advantages like cheap, light, abundant and environmently friendly. Wood-polymer composite have a pioneering role in composite technology and large number of applications in automotive, constructions, marine and transportation. A series of wood-polymer composite were manufactured with hornbeam, pine, medium density fibreboard with coupling agent. According to the tensile test results, if we compare the neat PP tensile strength for three different wood, generally tensile strengths are increase with respect to increase amount of hornbeam, pine and MDF, strain generally decreased and elastic modulus strongly increased. When amount of coupling agent MAPP is increase Max stress, strain and elastic modulus increase also. MAPP has good affect for mechanical properties. Flexural strength also increased with respect to amount of wood flour, strain a little bit decreased and elastic modulus increased for three different wood types. Although, in hornbeam case, when amount of MAPP is increase flexural modulus increase but in case of Pine and MDF when amount of MAPP increase, flexural modulus did not increase.

According to the mechanical testing results, Hornbeam / PP, Pine / PP and MDF / PP composites can be used instead of neat PP in many applications, in some applications composite can performed better than neat PP with high elastic and flexural modulus.

Interaction in wood-polymer composites were analysed with (FTIR). XRD diffraction patterns obtained from all the analyses exhibit individual spectacular changes in peaks. The interaction of neat polymer, woods and coupling agents were investigated.

Morphology of interface was observed by SEM analysis. It is seen that hornbeam and pine flours has medium homogeneous, in MDF has homogeneous. When amount of MAPP increase, adhesion bounding also increase. Also, in some sample, composites produced has air voids. As a result, preferring to use of wood-polymer composite over neat PP will reduce the cost and weight of material, therefore reduce the cost and weight of car. For this reason it will be innovation for transportation and construction industry. For the future of study, when preparing the mix of WPCs lubricant and stabilizer can be added. Wood flours could be feed from a side-feeder to extruder in order to enhance the dispersion of wood in polymer melt. Also different coupling agents can be tried to obtain WPCs. Another important step can be drying stage after get the pellet form of wood-polymer composites. Different types of wood can be used. Instead of wood flour, wood chips can be tried. Additional analysis for characterization could be performend to wood reinforced composites such as DSC (Differential Scanning Calorimetry), contact angle measurement, water absorption capacity and UV test.

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