KAUNAS UNIVERSITY OF TECHNOLOGY

FACULTY OF MECHANICAL ENGINEERING AND DESIGN

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STUDY ON MECHANICAL, THERMAL AND MORPHOLOGICAL PROPERTIES OF WOOD POLYMER COMPOSITES

Master's Degree Final Project

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Faculty of Mechanical Engineering
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KTU, Lithuania.

KAUNAS, 2016

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"Study on Mechanical, Thermal and Morphological Properties of Wood Polymer Composites"

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MECHANICAL ENGINERRING AND DESIGN (621H30001)

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"Study on mechanical, thermal, and morphological properties of wood polymer composites"

Final project

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MASTER STUDIES FINAL PROJECT TASK ASSIGNMENT
Study programme MECHANICAL ENGINERING - 621H30001

Approved by the Dean's Order No.V25-11-7 of May 3rd, 2016 y

Assigned to the student **BALACHANDER KESAVALU**

(Name, Surname

1. Title of the Project

Study on mechanical, thermal and morphological properties of wood polymer composites

2. Aim of the project

To study and compare the properties of wood polymer composites based on its compositions and processing conditions.

3. Tasks of the project

- Material prepared according to the compositions and processing conditions of WPC
- Composition of WPC: 0%, 20% and 40% of wood content
- Processing conditions are drying, sieving and extruding
- Study on Mechanical, thermal and morphological properties of WPC
- Comparing test results between 0%, 20% and 40% to analyses the effects of WPC on its composition and processing conditions

4. Specific Requirements

Conducting the final experimental project thesis according to KTU regulations and requirements.

5. This task assignment is an integral part of the final project

6. Project submission deadline: 2016 JUNE 10th.

Task Assignment received ______________________ ______________________

(*Name, Surname of the Student*) (*Signature, date*)

Supervisor ______________________ ______________________

(*Position, Name, Surname*) (*Signature, date*)

BALACHANDER Kesavalu. “Study on mechanical, thermal and morphological properties of wood polymer composites”. Master's Final Project / supervisor Assoc. prof. doc. dr. PAULIUS Griškevičius; Faculty of Mechanical Engineering and Design, Kaunas University of Technology.

Research field and area: Technological science, Mechanical engineering and material engineering

Keywords: LDPE, Wood polymer composites, DSC, Strength, Composition, Material characterization

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SUMMARY

In this research study, the Spruce Pine wood flour is used as a filler material with the low density Polyethylene to prepare composites without adding any inorganic fillers. Considering the advantages and need of environment safety, cost effect, low maintenance, good weather resistance and high dimensional stability the wood polymer composites are used and it requires more improvements in future. This Research is based on the effect of processing conditions and compositions of wood polymer composites tested under some technical properties like Mechanical, thermal and morphological properties.

For preparing specimens to this study, the wood flour and LDPE material mixed together and extruded at 150°C in co-rotating twin screw extruder. Later the extruded materials is used to melt in the glass tube by using gravimetric casting machine for 130°C at 3mm/min heater movement speed to prepare test specimen. For material characterization, the tensile test, bending test, charpy test, DSC test and optical microscope for microstructure are the tests conducted and results are plotted in graph and compared with the composition of wood materials. For the testing, 0%, 20% and 40% of wood filler content is used. However, the results from mechanical, thermal and morphological properties represents that the addition of wood filler contents improves the strength and stiffness of the material, and decreases the characteristics ductility to brittleness. In thermal properties, there is no significant changes between the virgin LDPE and addition of wood contents (20% and 40%). Microstructure shows that the pure plastics has mixed and melt properly, in 20% wood filled polymer content the filler material is enriched in some places it leads to some minor changes in properties. In 40% content, the wood filler has enriched grains in particular place without mesh with polymer. There is more chance to create void, it may affect the material strength.

BALACHANDER, Kesavalu. Medienos polimerinių kompozitų mechaninių, šiluminių ir morfologinių savybių tyrimas. Magistro baigiamasis projektas / vadovas doc. dr. Paulius Griškevičius; Kauno technologijos universitetas, Mechanikos inžinerijos ir dizaino fakultetas.

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Reikšminiai žodžiai: LDPE, medienos polimeriniai kompozitai, DSC, stiprumas, kompozicija, medžiagos charakterizavimas

Kaunas, 2016. 63 p.

SANTRAUKA

Šiame tyrime, eglės pušies medienos miltai kartu su mažo tankio polietilenu yra naudojami kaip užpildo medžiaga norint paruošti kompozitus nepridedant jokių neorganinių užpildų. Atsižvelgiant į privalumus ir būtinybę saugoti aplinką, kainos efektą, mažą išlaikymą, gerą atsparumą oro sąlygoms ir aukštą matmenų stabilumą, medienos polimeriniai kompozitai yra naudojami ir jiems reikės daugiau patobulinimų ateityje. Šis tyrimas remiasi medienos polimerinių kompozitų apdorojimo sąlygų ir kompozicijų poveikiu, kuris buvo tirtas pagal tam tikras technines savybes, pavyzdžiui, mechanines, šilumines ir morfologines savybes.

Ruošiant pavyzdžius šiam tyrimui, medienos miltai ir LDPE (Low Density Polyethylene) *medžiaga buvo sumaišomos kartu ir ekstruduojamos 150°C temperatūroje bendro sukimosi šnekiniame ekstruderyje. Vėliau ekstruduotos medžiagos yra išlydomos į stiklinį vamzdelį naudojant gravimetrinę liejimo mašiną 130 ° C temperatūroje 3mm / min šildytuvo judėjimo greičiu, norint parengti pavyzdžius tyrimui.* Pagal *medžiagų charakterizaciją buvo atliekami tempimo, lenkimo, smūginio tąsumo“charpy“* ir DSC testai, *bandymai optiniu mikrostruktūrų mikroskopu, o jų rezultatai nubraižyti grafike ir palyginti su medienos medžiagų sudėtimi. Testavimui buvo naudojami 0%, 20% ir 40% kiekio medienos užpildai. Tačiau rezultatai pagal mechanines, šilumines ir morfologines savybes rodo, kad papildymas turinį medienos užpildas pagerina medžiagos stiprumą ir standumą, bet sumažina plastiškumą iki trapumo. Nagrinėjant šilumines savybes, nėra reikšmingų pokyčių tarp LDPE spaudimo ir pridėjus medienos turinį (20% ir 40%). Mikrostruktūra rodo, kad grynieji plastikai yra sumaišomi ir išlydomi tinkamai, 20% medžio kiekio užpildytų polimero užpildų medžiaga yra praturtinta, kai kuriose vietose ji veda prie nedidelių savybių pokyčių.* Pildant *40% kiekio, medienos užpildas praturtino grūdelius tam tikrose vietose be akučių su polimeru. Yra daugiau galimybių sukurti tuščią erdvę, bet tai gali paveikti medžiagos stiprumą.*

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INTRODUCTION

Wood Polymer composites are one of the major application for recycling, reuse, and a recovering a variety of byproducts from the industrial resources. Wood fibers are natural fibers and some more fibers are flax, hemp, cotton, jute, banana, ramie, sisal, and coir and date palm fibers. It has advantages like they are lighter than the fiber glass and talc [1]. In general, WPC are environmentally friendly and its usage is very significant [2]. These materials does not have any wear and it has very less abrasive for equipment. Main applications are in automotive industry (e.g. Dashpots), constructions, furniture and packaging [4]. During the composite preparation, the wood is hydrophilic in nature i.e. high surface tension, which is used to reduce its compatibility with the hydrophobic material i.e. low surface tension [3]. The combination of reinforced filler and matrix material are used to form composites in major applications.

In the WPC field, the chemical additives or agents are mixed with the filler and matrix material, they will improve the strength properties but the material turns into more non-degradable. In this research, the filler is Spruce pine wood flour and the matrix material is low density polyethylene (LDPE) is used to extrusion. We used the filler and matrix material composition without adding any chemical additives and also slight changes in processing methods.

OBJECTIVE

Main objective of this research is to study and compare the mechanical, thermal and morphological properties of wood polymer composites based on its compositions (0%, 20% and 40%) and processing conditions (wood flour drying, Sieving and extruding with LDPE). Strength, ductility and brittleness of the material is analyzed by performing the Tensile and bending test and fracture energy of the composite is obtained by performing the charpy test. Differential scanning calorimetry (DSC) test is used to analyze and compare the melting and crystallization temperature of 0%, 20% and 40% of wood filled polymer composites. Finally, the optical microscope is used to analyses the morphological properties (i.e. Color, appearance and grain structure) of the wood polymer composites. In this research study, all the above processing methods and testing methods are conducted at Kaunas university of Technology, Lithuania and University of Ljubljana, Slovenia.

1. LITERATURE REVIEW

Theoretical background

This literature review has different kinds of sections which relates the theoretical background of this Wood polymer composites. First part is an overview of plastics describing their properties and applications, as well as the detailed view of Low Density Polyethylene (LDPE). The second part provides an overview of Wood polymer composites and its related study. In third part, the principle and mechanism of WPC Extrusion process. Final section of the review, the theoretical background, and material characterization has been studied.

1.1 Polymers an Overview

To understand the Wood Polymer composites, first we have to know what the polymers is and how they behave. In this section, the general knowledge of polymers is given, from development and production through their applications, to the positive effect they have brought to our life.

1.1.1 What are polymers?

A *polymer* is a phenomenon which has a molecular structure made up of large number identical units linked together. Monomers are generally simple organic molecules containing a double bond or a minimum of two active functional groups acts as the driving force to add one monomer molecule to polymer molecule. This process of transformation of monomer molecules to a polymer molecule is known as polymerization [5]. Schematic presentation of ethylene polymerization is presented in Figure. 1.1

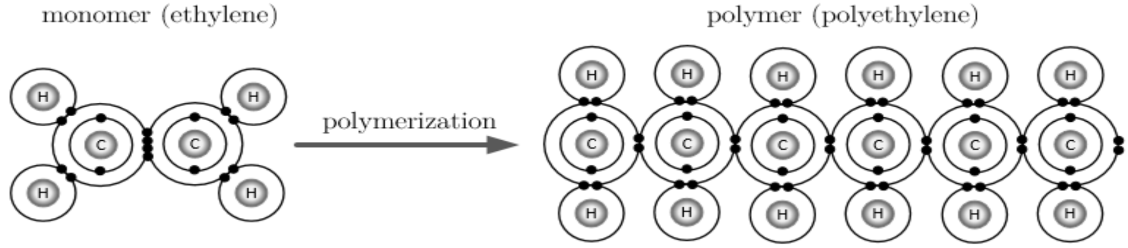


Figure 1.1 Schematic Presentation of ethylene Polymerization [5]

1.1.2 Polymer Classification

Polymers can be divided in many different ways; based on origin, polymerization mechanism, structure, thermal behavior, applications, etc.; in continuation only some general classifications are presented.

From the point of the structural shape of polymer molecules, polymer can be divided on; (i) linear, (ii) branched and (iii) cross linked [5]. Linear polymers have repeating units linked together in a continuous length, fig.1.2 a. when branches protrude from the main polymer chain at irregular intervals, the polymer is termed a branched polymer, Fig 1.2-b Example of cross-linked structure i.e. net formation is presented Figure.1.2-c.

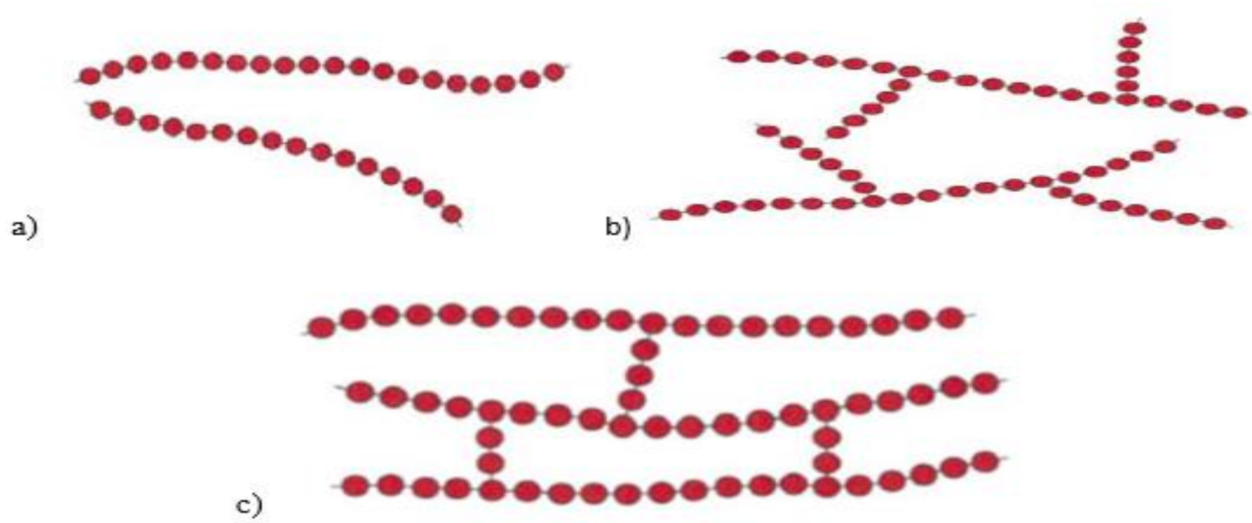


Figure 1.2 Schematic presentation of different types of Polymer structures:

a) Linear, b) branched polymer and c) Cross-linked structure [40]

For engineering purposes, the most useful classification of polymers is based on their thermal (thermal- mechanical) response. Under this scheme, the polymers are classified as thermoplastics or thermosets [3]. Thermoplastics materials can be melt and flow when they are heated and solidify as they cool. On subsequent reheating they melt and regain the ability of flow. This means they can be reprocessed and hence recycled by re-melting them. Thermoset materials are made up of cross-linked systems so the whole part or product is permanently set as one giant macromolecule with strong covalent bonds. Therefore, they cannot be reprocessed in the same way as thermoplastics, instead they tend to degrade and burn without melting. Highly dense molecular network also makes the material stiff and brittle. Several examples of thermoplastics and thermosets are presented in table 1.1 and 1.2.

Table 1.1 Most common thermoplastics types [3]

Material	**Abbreviation**	**Example Of Uses**
Acrylonitrile Butadiene Styrene	ABS	Housings, helmets.
Polypropylene	PP	Ropes, buckets, cups
Polyethylene	PE	Packaging, bags, pipes
Polyethylene terephthalate	PET	Drink bottles
Polystyrene	PS	Cups, plates, cutleries
Polyamide	PA	Gears, bearings, ropes.

Table 1.2 Most common types of thermosets [3]

Material	**Abbreviation**	**Example Of Uses**
Epoxy resin	ER	Bonding of other materials,
Polyester resin	PR	Bonding of other materials, adhesives.
Polyurethane	PU	Insulating foams, mattresses
Melamine formaldehyde	MF	Laminates for surfaces, tableware
Urea formaldehyde	UF	Electrical fittings, handles, adhesives

1.1.3 Low Density Polyethylene

There are several different plastic groups with specific properties for different applications. In that, mainly there are few high volume plastic families: Polyethylene including High density polyethylene (HDPE), Low Density Polyethylene (LDPE) and Linear Low density Polyethylene (LLDPE), Polypropylene (PP); Polyvinyl chloride (PVC); Polystyrene (solid PS and expandable PS); and polyethylene terephthalate (PET). All these five different plastic families are around 73% of all European plastic demand [6].

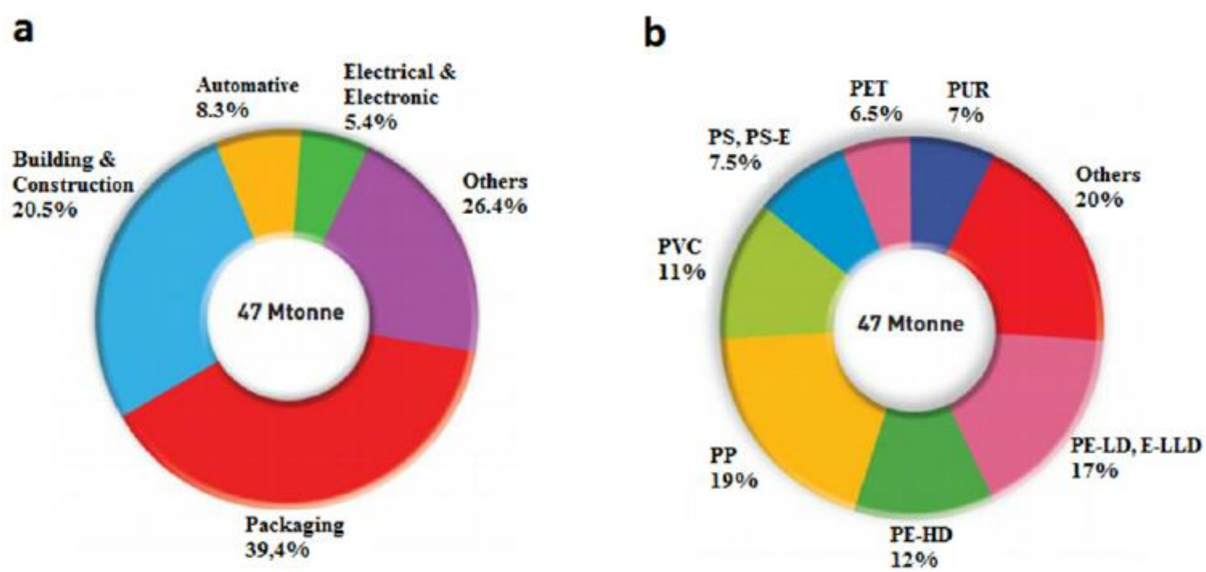


Figure 1.3 Europe plastic demands by resin type and its applications 2011 [6]

LDPE is manufactured by addition polymerization of ethylene under high pressure (103-345 MPa) and elevated temperature (200-350 ℃) in the presence of oxygen (0.03 – 0.1%) as free radical initiator [7]. LDPE has such good qualities like Flexibility, toughness, resistance to chemicals and weather, and low water absorption. It has very good flow behavior and environmental stress crack resistance. To manufacturing the films, LDPE are used as very extensively [6].

Some of the advantages of LDPE are low cost, impact resistance from -40 ℃ to 90 ℃, moisture resistance, Food grades available, readily processed by all thermoplastic methods, good chemical resistance.

LDPE which is soft, ductile, is additionally utilized for strong, elastic goods such as screw caps, lids and coatings. Some major applications of LDPE are carrying bags, bottles, toys, utensils, films and sacks [8].

1.2 Wood Polymer Composites

Wood Polymer composites are the polymerization of liquid monomers or oligomers already impregnated with wood. Wood polymer composites are generally the mixture of wood flour/particles filled with plastics and some chemical substances. It has greater dimensional stability, mechanical properties, less moisture than non-impregnated wood and greater resistance to chemical and biological degradation [4]. Wood polymer composites are becoming important structural materials in automotive, railway industry used as interior parts as well as in civil engineering [9].

In wood polymer composites, the composition of wood flour and polymers are greater in percentage than chemical additives. Some of the wood materials which are used for the WPC are Pine, Maple, oak with some typical particles sizes like 0.2 – 2.0 mm of wood content for the WPC [10]. Polymers like Polyethylene (PE) – (LDPE or HDPE), Polypropylene (PP), and Polyvinylchloride (PVC) are the most common plastics are used as composition of WPC [12]. These thermoplastic polymers are can be reclaimed, recycled, Virgin or blends. Main source of the WPC materials are either virgin or recycled materials.

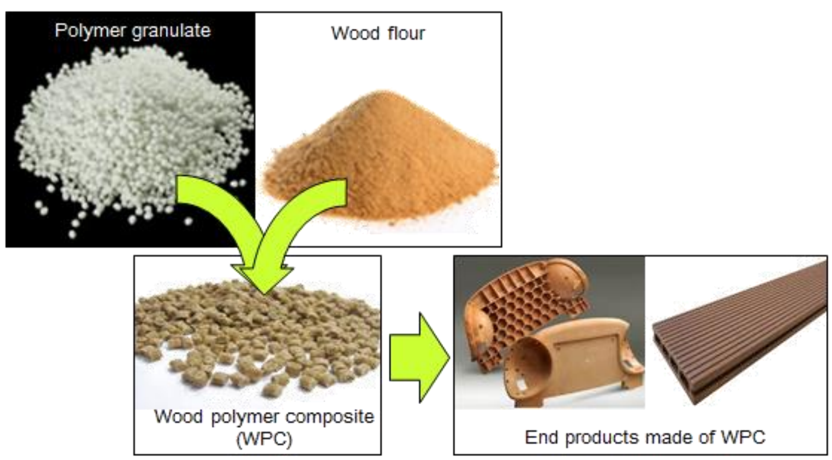


Figure 1.4 Schematic representation of Wood polymer composite (WPC)

The above figure describes the wood polymer composites, which the wood flour and polymer granules are mixed and fed by the extruder and the final product is prepared for the use.

1.2.1 Wood polymer composites: Advantages and disadvantages

WPC can be used both virgin and recycled materials which increasing the volumes, in wood the water absorption level is significantly high and it rupture wood products, due to this the addition of polymer materials which helps to resist the water absorption and provides more durability and strength to the material. Generally polymer materials are having good water absorption resistance. It is thermally stable than plastics, which absorb more heat and withstanding capacity is higher than plastics. When it compare to the appearance, the wood polymer composites are very slight variable than the wood, it dramatically reduces the usage of environmentally harmful paints, and sealers. Some of the demerits of the wood polymer composites are high costs, creep under load, thermal expansion and lower stiffness than wood [11, 12].

1.2.2 Uses

Majorly the WPC are used in the wide range of building and construction sector, which are having some applications like decking and boardwalks, cladding, exterior (Facias, Bargeboards) and interior building trim (doors, skirting boards) etc. Panels and trims in automotive industry, outdoor and indoor furniture, bathroom and kitchen cabinets in Furniture. Garden structures, playground equipment are also made by wood polymer composites [11].

1.3 Literature on WPC: Preparation, Processing and end results

In this study, R.N. Darie et al 2010 states that Low density polyethylene filled with 10, 20, and 30% of Oak wood flour which is melt processed by Brabender mixer method for the evaluation of properties exposed to ageing. For the test, the matrix material is virgin LDPE with filler is oak wood flour which is the remaining portion after the sawing operation. Wood flour has sieved and dried for 17 hours at 100°C. Sample is prepared with melt blended at 145°C then the system processed at 10 min at 60rpm. Later the samples are pre-melted for 5 min and sandwiched in carver press for 4 min and pressed at 145°C under a pressure of 200 bar to obtained specimen for mechanical test. Researcher has carried out two mechanical tests. Results which represented in tensile test shows that young's modulus of the composites increased with increasing the wood content in blends. Plastics provides more ductility while the wood flour exhibits brittle behaviour. In Charpy test, researcher analysed that decrease in impact strength especially at long exposure time. When the composites contains 30% of wood flour, the impact strength is decreased when compare to virgin LDPE [3].

In development of a new composite, the recycled polyethylene is filled with bagasse and wood flour. Mainly this combination of composites are used instead of wood parts in moist place to increase the endurance of the application. Researcher has stated that 40%, 30% and 20% of filler material combinations with matrix material for the preparation of specimens. Extrusion and Injection method is used for the preparation of specimen. First the filler and matrix is initially dried in an oven for 85°C for 20 hours and homogenized manually in a plastic container and later the pellets were placed in an oven for 20hrs at 80°C for drying before injection to produce the specimen. Aim of this research is to concentrate on the processing parameters, how the materials and specimen are prepared for the tests. For injection, the speed is 40mm/s with 50 bar of pump pressure and the injection temperature is 185°C is used.

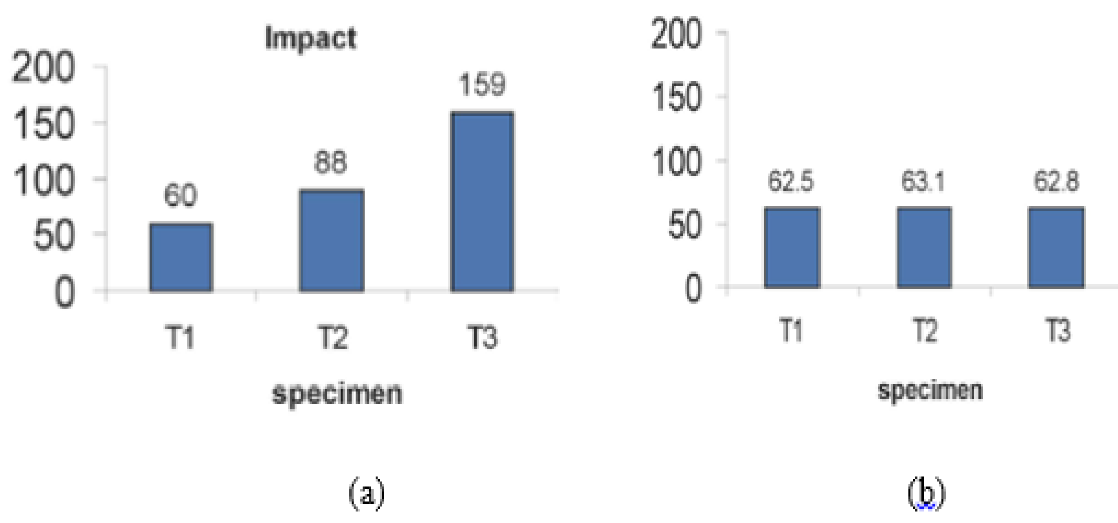


Figure 1.5 Test results of a) Impact strength (KJ/m^2) and b) Bending strength (N/mm^2) [2]

In results, S.S. Homami et al 2013,. Analysed and showed that ratio 20% of filler (T3) showed better performance in the impact test and when compared these polymer type property there is no significant difference between T1, T2 and T3. For pallet boards, LDPE was more suitable than recycled LDPE [2].

R.M.Government et al 2013, has discussed about the effect of particle size and filler content on ultimate tensile strength of date palm wood flour – recycled LDPE composites. Researcher's chosen 4, 8, 12 and 30% of wood flour content as a filler with different particle size like 150, 212, 250 and 300 µm. Both the wood flour and recycled LDPE was sundried to remove impurities and crushed or cut to small sizes. Injection moulding machine is used for the preparation of materials which is combination of Wt% of wood flour and recycled LDPE.

Tensile test is carried out experimentally and then compared the results with analytically using C-NIKBRAN software. Author expressed that both results are almost comparable and closed values [13].

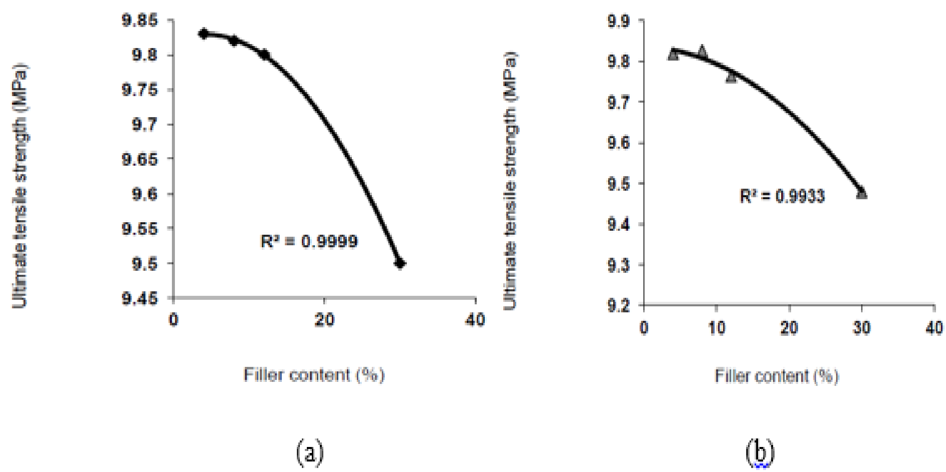


Figure 1.6 Test results of Ultimate tensile strength a) Experimental and b) Analytical model [13]

R.O. Medupin et al 2013, has investigated the mechanical properties of polymer matrix composites with addition of wood waste. Compression moulding techniques is used to fabricate the composites. Wood wastes ranging from 20%, 30%, 40%, 50% and 60% wt used for the composition, results which indicated that 40% of wood waste was the optimum reinforcement. Strength and stiffness of the composites improved when increased the amount of fillers, but impact strength decreased considerably. Researcher analysed that 60% of filler content absorbs more water and also in general the composites have high water absorption rate in first few hours compare to after sometime [14].

In this research work, Sailaja et al stated that LDPE with esterified wood pulp to prepare composites, for interfacial adhesion the LDPE grafted anhydride maleic acid is used. Addition of compatibilizer shows much difference in mechanical properties when 20% of wood loaded with composites shows that better ductility and higher stress values as compared to uncompatabilized composites. Here they states that when addition of wood till 40% as gains higher stress which shows more brittleness in composite. Author followed same method for thermal properties, it reveals that neat LDPE has single degradation at 465℃ with 70% wt. loss and 98% wt. loss at 485℃. For 20% wood loading uncompatabilized composites exhibits two stage degradation at 347℃ with 11% wt. loss and 480'C with 83% wt. loss. Similar trend in Compatabilized composite, but the wt. loss is higher (88%) owing to increased interactions between wood ph. and LDPE [15].

A.K. Mishra and A.S. Luyt has discussed about the influence of nano-silica mixed with LDPE through Sol-gel process. Researcher has focused on mechanical and thermal properties of LDPE, Wood flour/LDPE composites with absence and presence of Dicumyl Peroxide (DCP). Matrix Polymer is Low density Polyethylene with the addition of filler material is Pine wood flour with maximum particle size around 300µm and 1.05g cm^ -3 density. Material has prepared in the form of LDPE, wood flour and silica hybrid all mixed together in Brabender melt mixer at 160℃ for 15 mins at a speed of 30rpm. In results, the tensile modulus increased and tensile strength decreased when the addition of Wood flour continuously. Tensile modulus increased. Tensile modulus increased with WF content not with influence of presence of DCP. Thermal stability of matrix improved when DCP presence and at the meantime the crystallinity of LDPE is reduced by the Presence of Nano-silica [16].

Yaolin Zhang et al 2008, has investigated the effects of WF content, chemical agent content on tensile properties of wood fibre polyethylene composites. In this study, the polymer matrix is polyethylene and three different fillers are used. Black spruce fibre 1 and 2 with a steam pressure 15 bar and a cooling time 3 min and 6 min respectively. While jack pine fibre with steam pressure 7.5 bar and a cooling time 3 min. In each run 0.1% of antioxidants was added to present thermal oxidation during mixing. Composites were prepared with combination of polyethylene, WF, CA and antioxidant into a laboratory internal mixer. In results, the author states that addition of WF, a composite material changes its transition from ductile to brittle and also WPC stiffness increased. Young's modulus increased with increasing WF content, while WF type, content and CA content plays significant role in tensile stress at fractures [17].

In this research, the author states that LDPE/wood flour composite is mixed with degraded LDPE as compatibilizer to investigate its effects and properties. By melt mixing process the samples are prepared and then melt pressed for preparing the test specimen. SEM, thermal stability, mechanical and viscoelastic properties are analysed and the researcher found that the composite with compatibilizer is much better performance than uncompatabilized composite. In thermal stability, there is no significant improvement in degraded low density polyethylene treated composites. Because of this compatibilizer the mechanical and viscoelastic properties are influenced. This study carried out by S.S. Ndlovu et al. 2013 [18].

Xiang Li et al 2013 states that to increase the properties of wood polymer composites by reinforce the filler content bamboo charcoal with the matrix material LDPE. Bamboo charcoal is reinforced the wood plastic composites which is used to compare with WPC without bamboo charcoal. Researcher found that thermal properties, water resistance, tensile and flexural properties of this composition is better than the normal WPC. And also this composition has resist the water absorption on mechanical properties of composites [19].

For composites, some new types of fillers used with matrix material LDPE to analyse the effects of mechanical, thermal and electrical properties. In this study, author has suggested the grass coated with polypyrole as a filler for the modified organic fillers. This composition is prepared by melt mixing and then investigated with the influence of crosslinking. Results of mechanical testing subjected to the young's modulus where composites prepared with cross-linking is slightly decreased compared un-crosslinked grass. Matej Micusik et al 2006 states that presence of Polypyrole on grass surface has reduction of crosslinking of the LDPE matrix [20].

Mariam A. AlMaadeed et al 2013, states that the flexural strength and young's modulus of the composite are having composition of 10 to 70% of filler content shows greater strength and stiffness than the neat LDPE. In this paper, the date palm wood flour is used as the filler content with the LDPE matrix. While increase in filler content significantly increases property of the composite. Author mentioned that flexural strength of 17.8 MPa for composite filled with 70% content was two times greater than virgin plastic [21].

Four parameters are evaluated in this study, they are wood flour particle size, coupling agent dosage, lubricant content and the mass ratio of wood and recycled plastics. Where the compositions of the wood polymer composites were evaluated by the physical and mechanical properties in order to the parameters which is used. Researchers Shao-Yuan Leu et al 2014 states that the wood content limit has to maintain till the 50% to the composite for good properties, once it reaches the 50% or above the results are reduction in all physical and mechanical properties. Authors investigated the exact effects of wood polymer composites when changing the material composition. Reducing thickness swelling from water adsorption may also improves the mechanical performances of WPCs [22].

1.4 WPC Processing

In this section, the most widely used techniques for processing of Wood and thermoplastic materials are presented in the below topics. In this investigation, the most widely used technique or method to prepare the materials by Extrusion process, which is explained detailed in the further sections.

1.4.1 General overview of WPC processing methods

Generally, the processing methods are used to convert the raw materials into the desired product which is then used to the further operations. In WPC processing methods, majorly the processing methods are very similar to the thermoplastics methods. Even in these WPC, there is majority of the raw material is from thermoplastics and the remaining amount of material is wood flour/particles. Selection of technology depends on the type of the material, size and shape of the desired product, as well the properties.

Some of the widely used processing technique in polymer industries are extrusion, injection molding, and compression molding.

1.4.2 Extrusion

Extrusion is a process which is used to convert the raw material into the desired product with the constant melting temperatures in the system. Extruder has several parameters like drive unit, hopper which is used to pour the granulated materials into the barrel, inside the barrel the extruder screws are placed with the several heaters which is used to melt the material. Final portion of the extruder machine is the die which is used to extrudate the product with the desired size and shape. Then, using the pelletizer to cut the materials into different sizes.

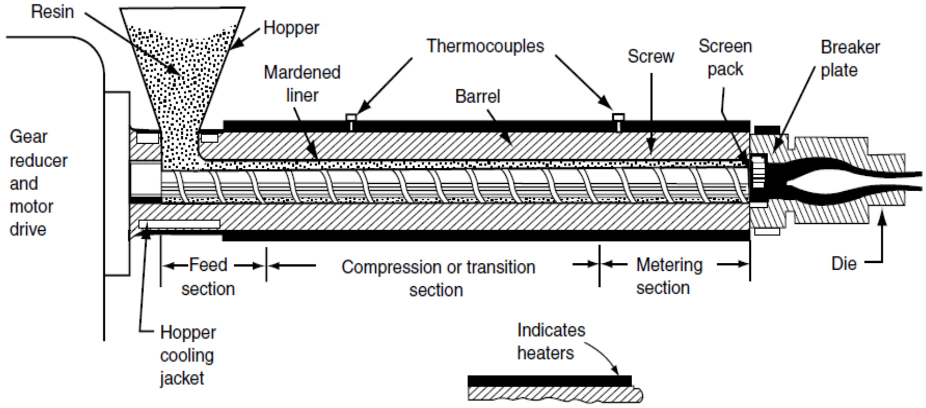


Figure 1.7 Schematic view of Extruder and its parameters [6]

In extruder process, material is feed into the barrel which is used to maintain the feeding area always cool to move the material and heated up further. In the cooling zone, the water is supplied inside the barrel and it recycled to the tank. Several heaters are used to melt the material at the elevated temperature and the rotating screws are used to mixing and moving the materials to the die. It is a continuous process, the melt exit the die with the desired shape of the final product. Extruders are capable to produce long length products which is later cut into the several sizes according to its usage. Some of the applications of extruder products are tubes, profiles, gaskets etc.; Extrusion process is explained in the section 1.4.4.

1.4.3 Injection molding

Another major processing method of WPC is injection molding, it is very similar to the extrusion process, as the material is fed into the hopper and it enter into the barrel, the rotating screw has pushed the melt with the elevated temperature. Once the melt reaches at the end of the barrel, the rotating screw rotates back and melt more material pushed forward to the end of barrel. After appropriate amount melt is reached, then the screw pushed the melt towards the mold cavity, before this operation the volume, pressure and stroke length of the screw has to be taken in account. Later the material in the cavity is exposed to additional pressure caused by the screw to compensate shrinkage because of cooling. Final stage of the injection molding process is to eject the product from the mold after the material solidifies.

For standard test samples, the injection speed is 80mm/sec, screw speed at 40rpm with 100 bar pressure were normally used in the Injection machine [23].

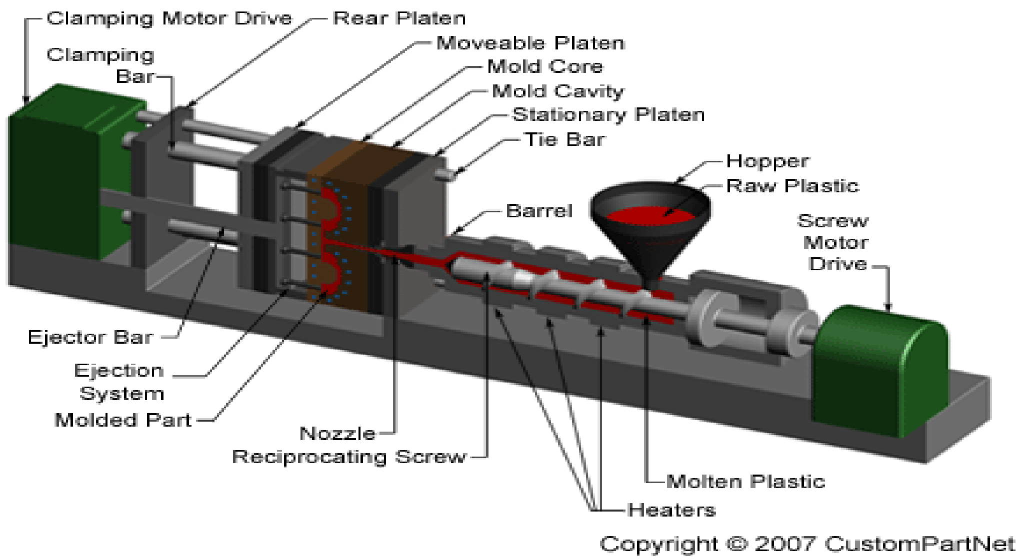


Figure 1.8 Schematic view of Injection molding machine and its parts [24]

Injection molding has very good advantages over the extrusion and other processing methods. It is capable to produce very complex things then these above methods. Some of the applications of Injection molding is toys, bottle lids. Tools etc.

1.4.4 WPC Extrusion

Wood polymer composites are manufactured in an industrial process for about 80 years. Plastics has two types of resins: one is thermosetting and the other is thermoplastics. Thermosetting is used in the early stages of production, later it changes to thermoplastics. It is most widely used for the industrial production of wood filled polymer material.

For WPC production, the thermoplastics are used both virgin and recycled materials. Wood polymer materials are manufactured by extruder process, there are some procedure to prepare the material properties for the processing operations. One is moisture content of wood and the other is pellets or powder of polymer. Wood is drying it before the extrusion by using the hot furnace. Moisture content of wood flour is accepted from 1-2%, in the worst case it will be 5-8% in the extrusion process. Pellets or powder are selecting according to the manufacturers need or industrial need. Powders are more suitable for extrusion process, because it can mix with wood flour more precisely but availability of powder is rare. But the pellets are available everywhere, so the transportation cost is saved more. In this case, pellets are widely used for the study.

Extruder has four types of processing systems, they are single screw extruder, Co-rotating twin screw, Counter-rotating twin screw and wood-truder. More detailed about these types are in the following paragraphs.

In Single screw extruder, it is simple extrusion system for processing WPC composites, which contains two sections. They are melting and metering, and vent section. For this process system, the polymer pellets and wood fibers are already mixed well in their composition.

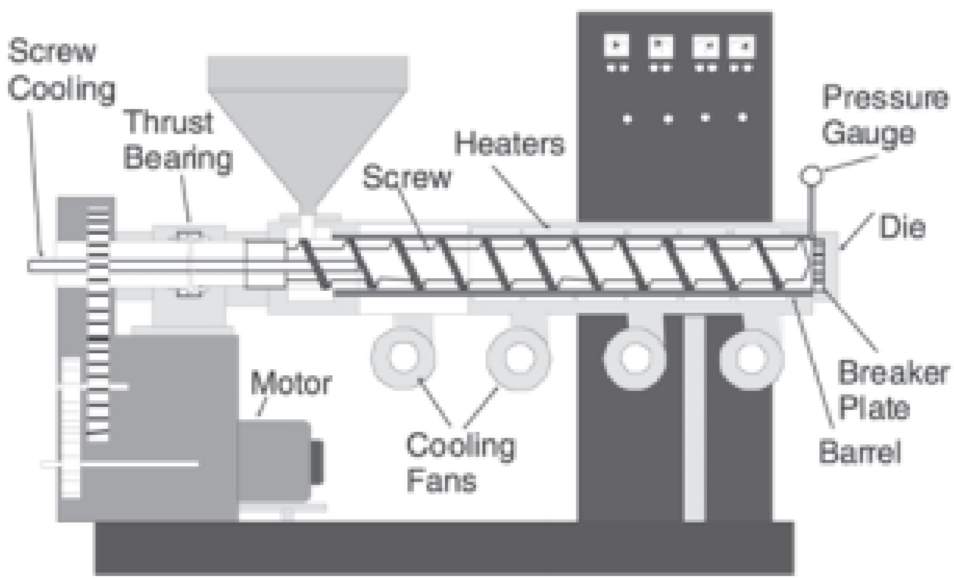


Figure 1.9 Extrusion Machine [25]

It won't mixing the material during the process, so it requires pre-compounded fiber filled polymer pellets. There is two process in the system one is barrel heat (ie. melting and metering) and other one is screw shear [26].

In Counter-rotating twin screw extrusion, the material flow is forced between two screws which resulting in high pressure at the nip and low pressure at the exit of nip. In co-rotating twin screw extrusion, the screw melt flow makes a pattern in a shape of X. This causes high and low pressure region. For this operation, the material is wood flour/fiber with 5 to 8 % of moisture content is valid.

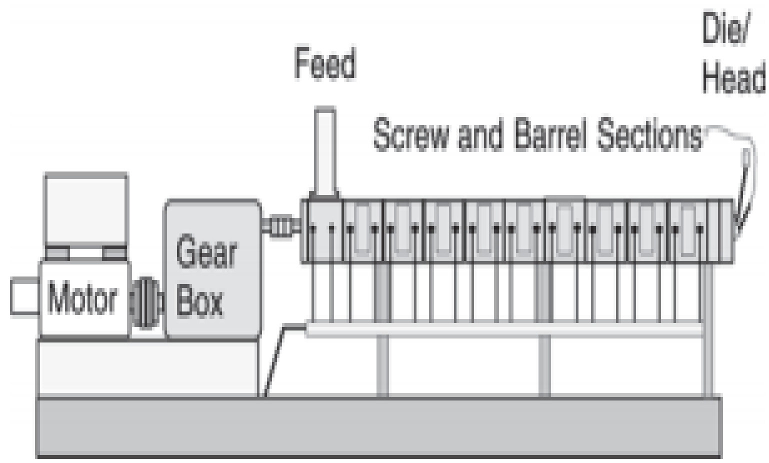


Figure 1.10 Co-rotating and counter rotating intermeshing screws [25]

Major advantage of this process is it has ability to accept the moisture wood content and it will dry and eliminate it during the high speed rpm of twin screw rotations. Some disadvantages are there is no screw cooling, it leads to burning some times and also it has very high speed rpm of screw rotation.

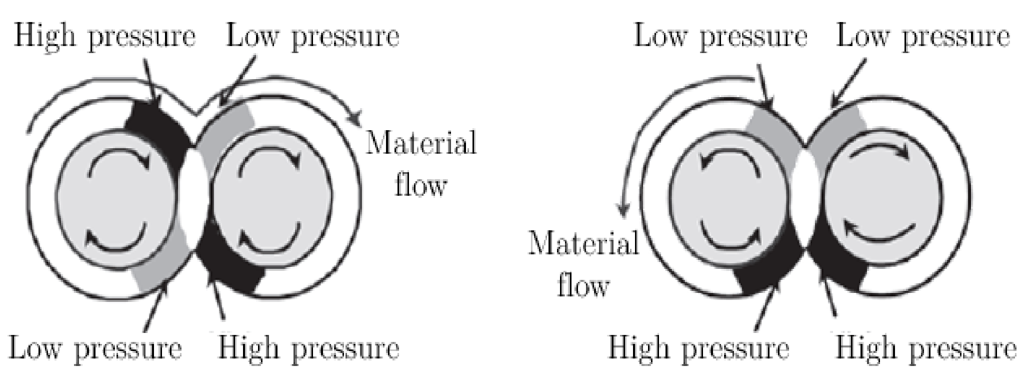


Figure 1.11 Screw rotations a) Co-rotating and b) Counter-rotating [25]

Screw design is one of the major parameter which is depended by the entire extrusion process. Screw design has having some configurations it can vary depending on the polymer, its sizes, requirements of screw is to feeding the solid material along the barrel, plastification, then conveying the melt, mixing, downstream feeding and pumping it to towards the die [25].

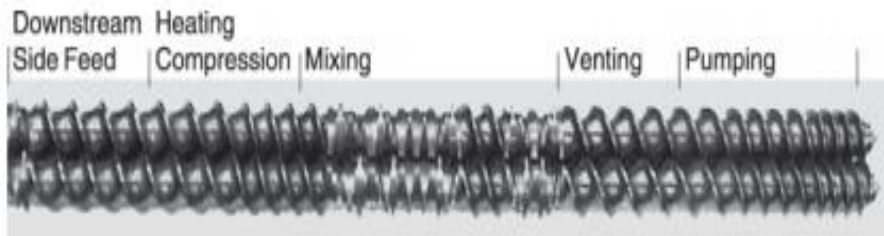


Figure 1.1 2 Twin Screw assembly [25]

Final section of the barrel, the die is attached which gives shape and desired size of the product. Die can be adjustable to compensate differences in rheology.

2. MATERIAL CHARACTERIZATION

2.1 Universal Testing Machines

In the method of testing the material characterization by the mechanical techniques which is used to test and compare the mechanical properties like strength, stiffness, toughness, ductility, young's modulus and elongation by the following test methods. Some of the mechanical test methods are tensile test, bending or flexural test, Impact test, Torsion test, Compression test etc. Mechanical test equipment's are almost used to test the standard specimens, which gives the strong material characteristics and it's very useful to predict the withstanding capacity of the materials. Strength is exposed when the material is going under tension or stretched. Similarly in the compression machine, when the material has compressed to show how much strength it will accept. A material which is strong and when one tries bend it, then it is called as bending or flexural test. If the material is twisted then it is called torsional test and if the material is hit by the hammer suddenly with the sharper end then it is referred has impact strength which is also called in technical testing machine name is Impact charpy tester. Elongation happens when the stress is applied to the material to break something. There are two important parameters that are used to measure during the deformation, which is ultimate and elastic elongation. In the below section the mechanical testing equipment's are explained detailed.

2.1.1 Tensile Testing

In general, the tensile testing machines are hydraulic or electromechanical. Each and every methods differs by which the load is applied. Variable speed electric motor and a gear reduction system which are the main specifications of electromechanical machine. Cross heads are moves up or down according the processing steps and test conditions. Testing machines consists some important parameters like speeds of testing, grips, load indicator, and extensometer. Parameters which are briefly explained further, tensile testing machines are capable of maintaining speeds of testing which shows in the below table,

2.1 Tensile testing machine

Grips are used to hold the test specimen in the machine, so that the specimen major axis coincides in the pull through directions of the centerline of the grip assembly. Clamping system does not involve any actions over the premature failure at the grips. Extensometer is used to calculate the test specimen length during the test which is relatively changes according to the applied force. This instrument is very accurately calculating the changes in length with 1% of relevant value [27].

In this testing machine, the tensile test and compression tests were performed. For tensile operation, the dimensions of the test specimen has been measured and the gauge length has been noted before the test began.

2.1.2 Bending Test

Bending test method is used to determine the behavior of materials subjected to the loading at any point and also known as transverse beam test. When the load is applied over the material, upper surface is in compression mode and the bottom surface is in tension or expansion. Bending test has some important test method like three point bending and four point bending test to perform the small samples and large samples respectively.

Principle of Operation

In general, the bending test equipment consist of two anvils support at the end to carry the specimen over its surface. Indenter is used to move downwards to apply the bending force and moves upward move to release the stress in the specimen after it breaks. Forces are applied at two different positions according to the type of method for 3 point bending the force is applied at the centre of the specimen, similarly for 4 point bending test, the force is applied at a defined distance either side of the center. Anvils are adjustable supports which is used to change the span freely according the dimension of the specimens used. In the end, the specimen deflection can be measured by crosshead displacement or by extensometer [28].

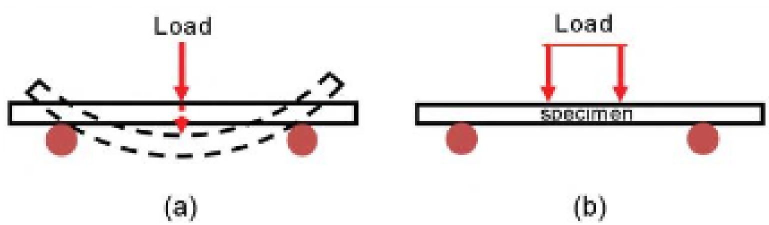


Figure 2.2 Bending methods a) Three Point bending and b) Four point bending [29]

Bending test produces tensile stress in the upper surface of the specimen and compression stress on the bottom surface, this creates a shear stress along the midline. In this test, mostly polymers are performed in 3 – Point bending test, while in 4 – Point bending tests the material like wood, steels, and composites are mostly used to test. Brittle materials like Ceramic or concrete is also tested on 3 point and also in 4 point bending method [30]. In results, the bending strength and modulus are required to analyses by the collected data with the appropriate plotted graphs.

2.2 Charpy Testing

Charpy test is one of the impact test with IZOD, the specimens placed horizontally for the operation refers charpy and the vertical sample specimens are used in IZOD impact test. For impact test, the charpy tester machine is used with 1 joule hammer arm. Samples are cut with 80mm for the test. Needle in the charpy scale has calibrate it before use. Needle position has changed after every specimen test. It should be adjust to measure the proper value of the energy absorbed during the specimen fracture. Fracture energy and impact energy is to be calculated.

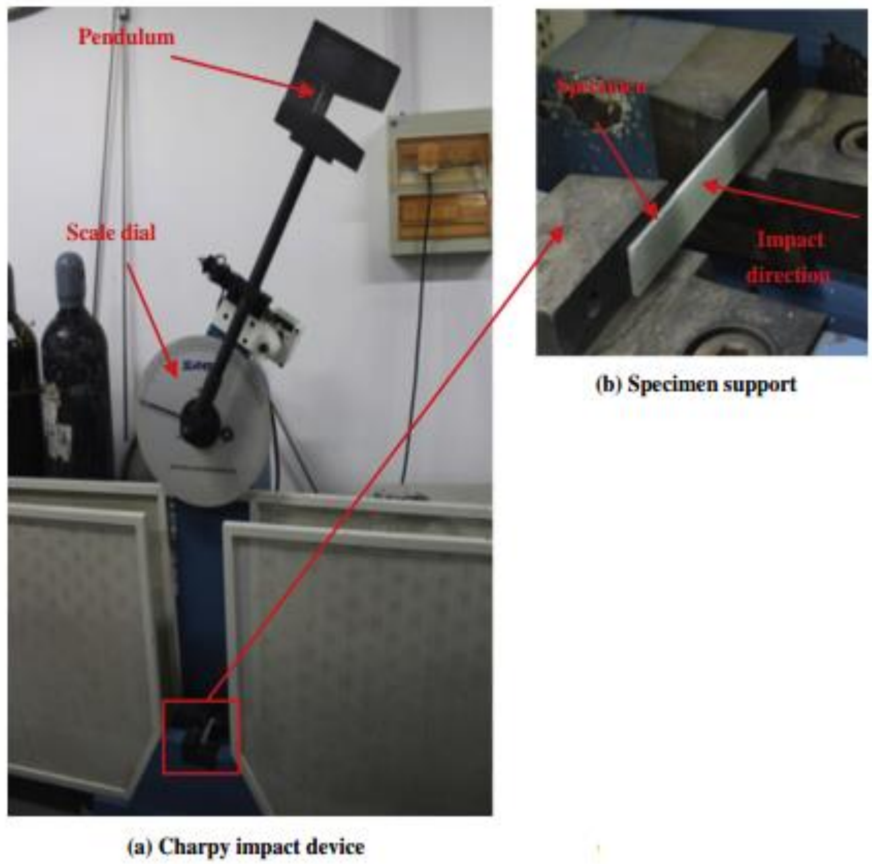


Figure 2.3 Charpy impact testing equipment [31]

Charpy test is the important material testing machine which is used to determine the material behavior at various working conditions [32]. Using Charpy tester machine, the impact strength, impact energy and fracture energy is tested with the movement of the arm/hammer. Hammers are from 1 J, 2 J, 5J, 10J, 25 J etc., which is used to test specimens like plastic, steel, aluminium etc.,

2.3 Differential Scanning Calorimetry (DSC)

As defined by Höhneet al (2003) in their book, Differential Scanning Calorimetry (DSC) is the measurement of the change of the difference in the heat flow rate to the sample and to a reference sample which is controlled by the temperature program. In other words, quantity of heat released or absorbed by substance when it undergoes chemical or physical change determined by the technique named as thermos analytical and also in condition it is monitored against time and temperature. Such changes alter the internal energy of the substance which is at constant pressure is known as enthalpy. When enthalpy increases, then the process is named as endothermic (glass transition, melting, evaporation) at the same time when the enthalpy decreases or lower, then it is called as exothermic (crystallization, decomposition) [33].

The apparatus test chamber used in DSC power – compensation device may consist of two small and separate furnaces that are controlled independently by existing heating program. Each of them contains its own temperature sensor, and a heater. Each furnace contains one pan, in that sample is filled in one and other one should be empty (reference).

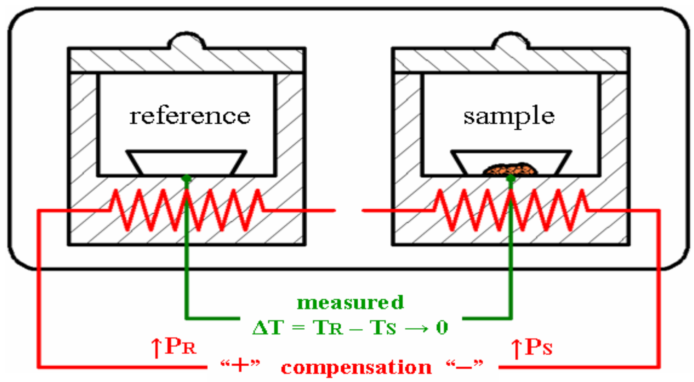


Figure 2.4 DSC scheme of Power transfer to reference and Sample [33]

P_R - Thermal power of Specimen furnace

P_S - Thermal power of reference furnace

T_R - Temperature of reference material

T_S - Temperature of specimen material

A control circuit supplies the same heating power to both furnaces in order to change their mean temperature according to the Pre-set temperature – time Program. In the case of ideal thermal symmetry, the temperature of both furnaces is the same. In theoretically, the symmetry is disturbed and a temperature difference between the furnaces result is happens when a sample reaction (exothermic or endothermic) occurs. This temperature difference is at the same time the measured signal and the input signal of a second proportional control circuit, which tries to compensate it by applying an additional heating power either to the sample or to the reference [34]. Ideally ΔT = 0 should be provided all the time. The difference in applied thermal power ΔP is equal to the change in heat flux ΔQ.

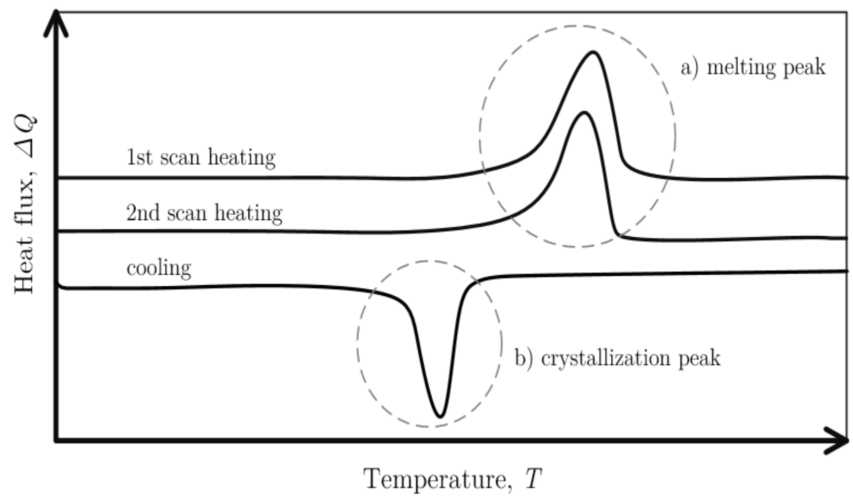


Figure 2.5 Schematic view of DSC curve for temperature [34]

This endothermic transition is represented by a peak in the heart flux, where the position of the top of the peak is considered as melting temperature, while from the area below this peak, the melting enthalpy can be determined. A similar approach is also valid for the crystallization, then it has exothermic transition. In DSC, the crystalline temperature represents the cooling curve in which the crystallinity promotes the heat resistance, hardness and rigidity. Normally the crystalline temperature has decrease in temperature in increased crosslinking when compared to uncompatabilized polymer composite used [35].

2.4 Optical Microscopy

Microscopy is a technical field of using microscopes for investigation of objects (or their elements) too small to be seen with the naked eye. Mostly morphology investigations are performed − to explore the object’s structure and also shape, size, position, relationship of its elements either on surface or inside.

There is a variety of microscopic techniques. However, the basic principles of all kinds of microscopy are nearly the same and can be clearly explained by the example of optical microscopy, which involves visible light transmitted through or reflected from the sample. Maximum magnification provided by optical microscope is 1500x. Theoretically, it is possible to create system with higher magnification. However, the main idea of microscopy is not to get as high magnification as possible, but to define the smallest elements of the structure, i.e. to use the highest resolution. The last is limited due to the wave properties of light (diffraction) and for optical microscope is approximately 0.2 μm.

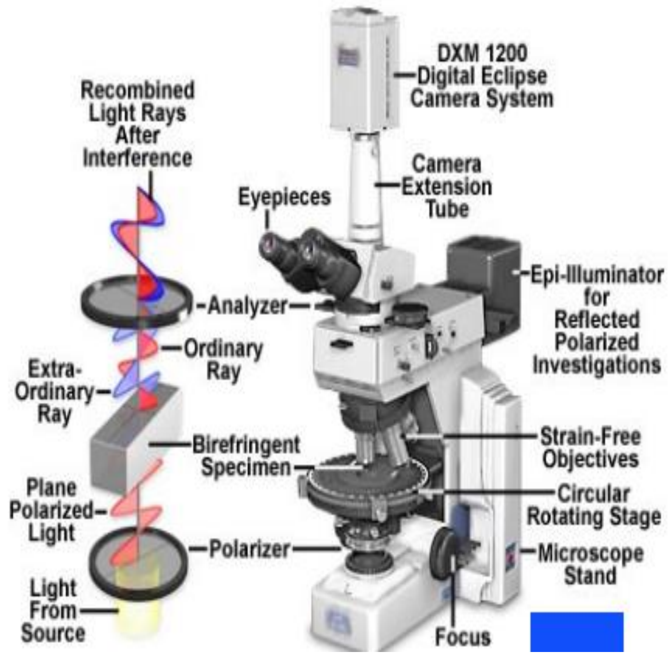


Figure 2.6 3D view of Optical Microscope [36]

The object structure can be investigated only if its elements reflect or absorb the light in different ways or differ from each other by refractive index [36]. This properties cause the difference in phase and amplitude of the light waves which went through different parts of object and so have effect on the image contrast. That is why the used contrast method should be chosen according to the properties of the object.

Polymers are examined by both transmitted- and reflected-light microscopy. It is worth to note that pure polymers hardly absorb any light, therefore bright field transmitted light contrast method is not very useful for them. On the other hand, polymers develop partially crystalline or amorphous structures, which are considered optically anisotropic and so, can be revealed by polarized transmitted light. For this reason polarization is the most common contrast method for the examination of polymers.

3. EXPERIMENTAL WORK

3.1 Materials for Preparing Samples

Filler material (Wood Flour) – Mixture of Spruce Pine
Polymer (Plastics) – Low Density Polyethylene (LDPE)

3.1.1 Wood Filler

For the preparation of Wood polymer composites (WPC), the filler material is Spruce Pine wood flour. These wood flour is remaining portion of the wood after sawing operation, it is collected from the local small scale industry. Wood flour has dried for 3 hours at 100 °C in hot furnace oven before extrusion process.

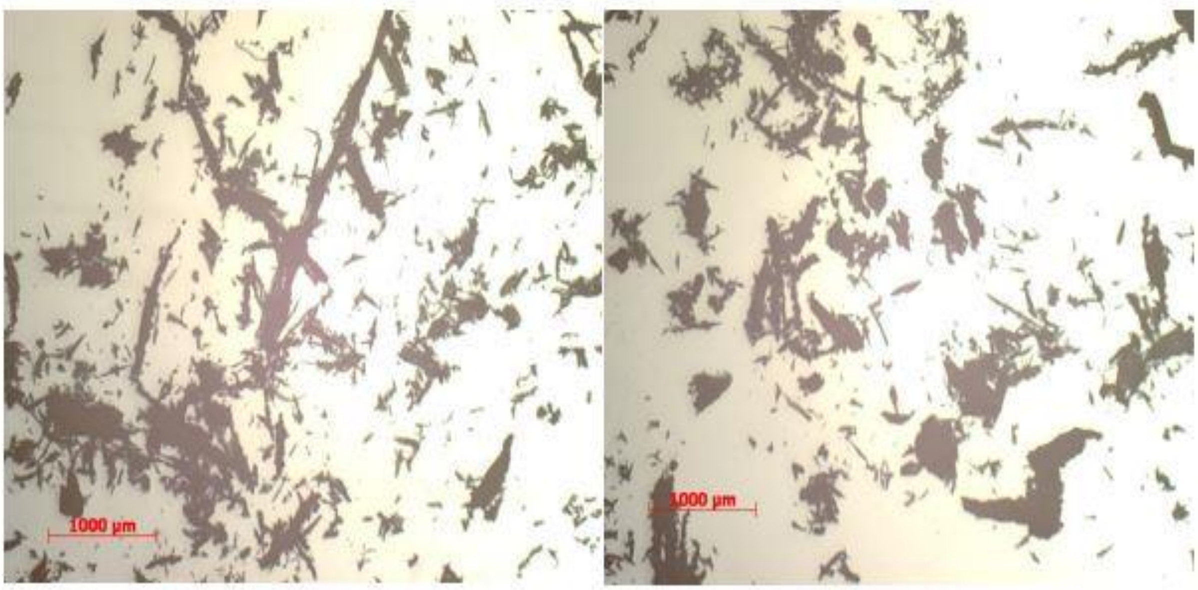


Figure 3.1 Microstructural view of Spruce Pine wood particle

Some of the wood particles are taken as a sample to check their size and structure in microscopy which is shown in the above figure 3.1.

Table 3.1 Specification of wood [37]

Common name	Spruce Pine
Average dried weight	525 kg/m^3
Elastic Modulus	9.69 GPa
Crushing strength	39.0 MPa

3.1.2 LDPE

For Polymer material, Virgin LDPE is used in this research. Virgin LDPE – Commercial product OKITEN® 245 S by DIOKI was used for the preparation of material for Wood Polymer composites. It is intended for extrusion of thin blown film having high slip properties, exhibits high transparency, and very good mechanical properties. This OKITEN® 245 S LDPE material is extruded in temperature range of 145 °C to 170 °C. Some of the properties of Virgin LDPE is mentioned in the below Table 3.2 with its ISO standards and units [37].

Table 3.2 Properties of Low density polyethylene (LDPE) [38]

Property	**ISO Standard**	**Value**	**Unit**
Density	1183	0.924	g/cm^3
Melt Flow Rate	1133	2.3	g/10 min
Tensile strength at yield	527/2	11	MPa
Tensile strength at break	527/2	14	MPa
Elongation at break	527/2	535	%
Hardness, shore	868	47	Scale D
Melting point (DSC - air)	11357	114	°C
Haze	14782	4	%
Friction coefficient: Static/dynamic	8295	≤0.11	-

3.2 Preparation of WPC

For the purpose of preparation of wood-polymer mixture, first wood flour and raw polymer granulate were premixed. Both components were, in proper proportion, fed into a container and manually stirred. The stirring was followed by the process of preparation of wood polymer composites was done by extrusion process. For this purpose, twin-screw extruder, PolyLab PTW 16/40 OS, produced by Thermo Scientific (Germany) was used, detail (a) on Figure 4.2. Extruder had installed horizontal rod die (type 557 - 3235) and a nozzle with diameter of 4mm, (b) in Figure 3.2. The arrangement of the co-rotating extrusion screws used for the process of mixing and moving the material is presented in Figure 3.3.

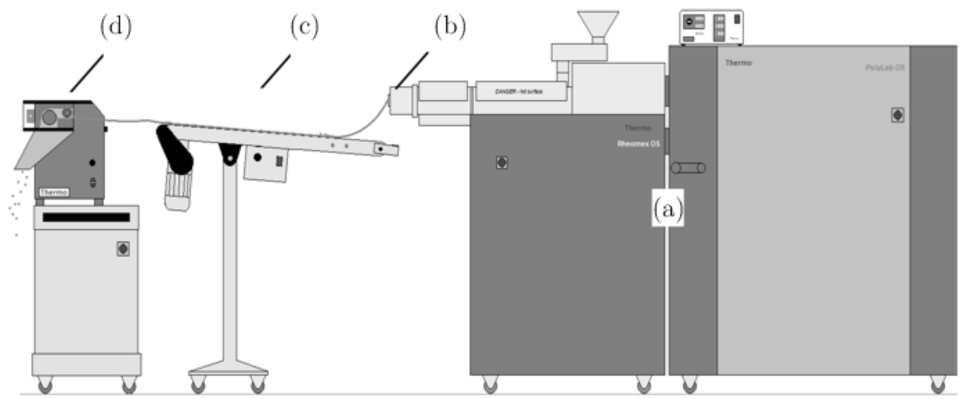


Figure 3.2 Configuration of equipment during preparation of WPC

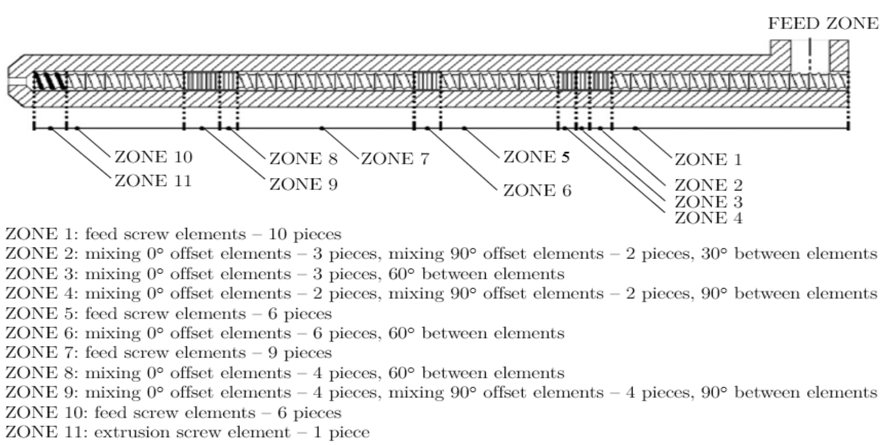


Figure 3.3 Used configuration of extruder screws

Material was extruded at screw rotation of 130 min-1, and processing temperature of 150 °C. The same temperature was set along the extruder barrel. After material was extruded, it was (c) firstly transported by the conveyor, (d) afterwards pelletized using a Thermo Haake pelletizer (type 557-2685), are presented in Figure 3.2.

After the extruded process, the pellets were cut from 2 to 4 mm size for the preparation of the samples shown in figure 3.4.

Figure 3.4 Pellets of WPC

3.3 Sample Preparation

Gravimetrical Casting

For the material characterization of wood polymer composites, the samples are prepared by gravimetrical casting machine it has parameter like Heaters, motor, glass sticks, and rotating screw for moving the heaters up and down direction. Cylindrical shape specimen with 6mm diameter and 180mm length for 130 °C at 3 mm/min. Gravimetric casting machine is a non- commercial apparatus which is the most suitable method for assuring isotropy of samples.

Heater is fixed in the moving plate and it connected to the rotating screw. Heaters are in the shape of hollow cylinder was positioned around the glass tube and moved from bottom to top with melt the material for 130 °C at 3 mm/min. Granules are filled in the glass tube, when the heaters moved up, in the meantime granules are filled upto 20 to 30 mm above the surface of the heater. Two piston rods contains loading mass of 600g each in the top for the compression force to push granulates melts to avoid vacuum in the specimen. After the melt process, the glass tubes are allowed to cool normally for 10-15 mins to get good specimen.

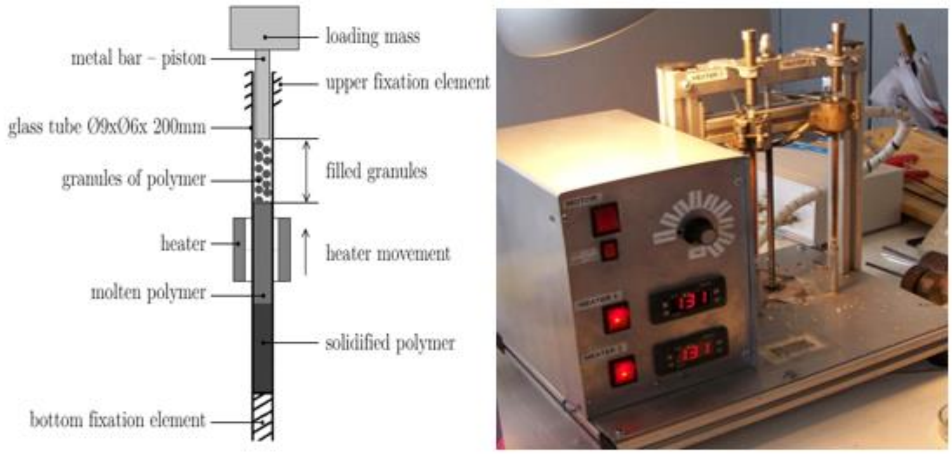


Figure 3.5 Gravimetrical casting machine and its parameters

Two specimen samples were prepared during one cycle of operation. Specimens are removed from the apparatus for experimental test. Most of the specimens are having void in the edges, so it is very important to remove the edges and cut the specimen for standard dimensions. Glass tubes specifications are 200mm long and 6mm diameter with high temperature withstanding capacity. Same Procedure followed for preparing the 0%, 20% and 40% sample specimens with constant temperature at 130 ℃.

3.4 Specimens for Testing

Sample specimens are prepared according to the weight 0%, 20%, and 40% of wood flour content mixed with the Virgin LDPE for the material characterization techniques like Tension, Bending, Impact charpy, Optical microscope and DSC. For Tensile test, the specimens are cut for 160 mm length including holding position, 80 mm long for bending test and charpy impact test and 8 ± 0.2 mg of sample packed in small pan and small portion of sample tested in microscope.

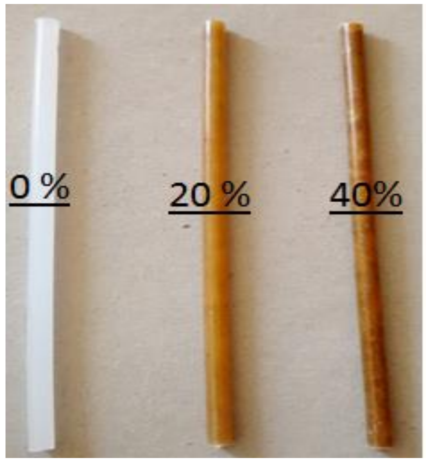


Figure 3.6 Sample specimen for Testing

3.5 Tensile test

In order to perform the tension test, the standard testing machine Model H25kT, Tinius Olsen ltd., England was used. In this machine, specially using HT40 grips to test high strength flexible materials with testing speed range between 0.001mm to 1000 mm and horizon software to collect data and stored in the database for long time. In our case, the specimens are cylindrical in shape. According to this condition the grips/holder are having cylindrical configuration in their edges to support the specimen for accurate test performance.

Figure 3.7 Tensile test machine during testing

For tensile test, specimen dimension is selected in the form of 160mm long and 6mm in diameter. Gauge length is 70mm, in the both end of grips 45mm each is used to hold the specimen. Testing speed is selected around 25mm/min for the 0%, 20%, and 40% specimen. Objective of the tensile test is to obtained the tensile strength, young's modulus and maximum stress at the fracture point and breaking point and it is plotted in graph to compare all results of 0%, 20% and 40% wt. wood flour-polymer matrix specimen.

For testing, six specimens in each type of %WF content specimen to get standard and accurate values of the stress and strains. For results, engineering stress and engineering strain is calculated by the formula.

$$Stress, \sigma = \frac{F}{A}, (\frac{N}{mm^2})$$

Where, F – Force (N)

A – Cross sectional Area

$$\text{Cross Sectional Area}, A = \frac{\pi d^2}{4}\ mm^2$$

Where, r – radius (mm) (cross sectional diameter of specimen (d/2)

$$Strain, \varepsilon = \frac{\Delta l}{l_0}, \left(\frac{mm}{mm}\right)$$

Where, l_0 – Original length (gauge length) (mm)

Δl – Change in length (mm)

Graph is plotted between Engineering Strain (m/m) vs Engineering Stress (MPa) for the comparative study of the property of material.

3.6 Bending Test

In order to perform the bending test, the standard testing machine Model H25kT, Tinius Olsen ltd., England was used. In this machine, specially using HT40 grips to test high strength flexible materials with testing speed range between 0.001mm to 1000 mm and horizon software to collect data and stored in the database for long time. In our case, the specimens are cylindrical in shape. Specimens are prepared as per standard dimension of 64 mm gauge length and 6mm in diameter, total length of the specimen is

80mm which the remaining part is placed over the anvil support. Cross head movement testing speed is set to 10mm/min for the WPC specimens. All the data's including Position, stress, load, area and time are stored in the database of Horizon software.

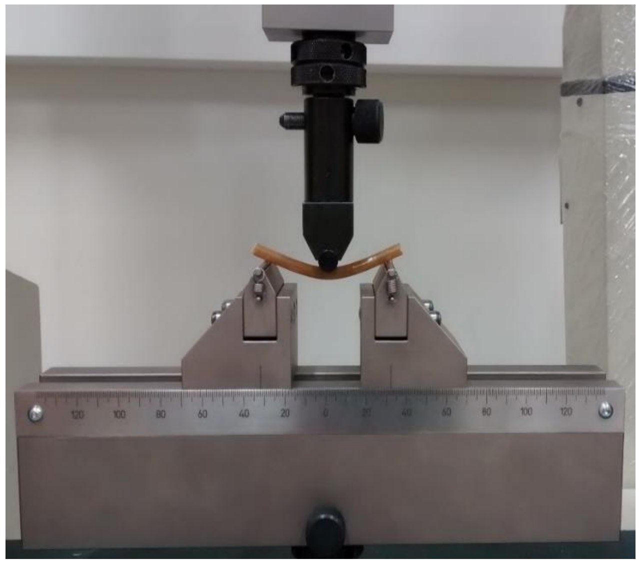


Figure 3.8 Three Point Bending Test on Specimen

For bending test, the test samples of 0%, 20% and 40% of wood filled polymer composites are tested under the loading conditions with 10mm/min of testing speed and the gauge length is 64 mm. Bending stress and bending strain is calculated by using the formula. Before testing, the test specifications, specimen details, speed, crosshead distance and type of material will be entered in the software. And also the details about the end result data like Position, load, time and area are selected to display. Later the files are export to the excel format to plot the graph for the flexural strain vs flexural stress and the flexural modulus are required to calculate for the comparison the specimen property.

For calculating the bending stress, displacement for the cylindrical specimens, the below mentioned formulas are used,

$\text{Bending Strength}, \sigma = \left(\frac{PL}{4Z}\right),$

$\text{Dispacement}, \Delta = \left(\frac{PL^3}{48EI}\right),$

Where,

P = Force (load) in N,

L = Length of beam in mm,

r = radius of specimen in mm,

E = Flexural modulus

Z = Section modulus

I = Moment of inertia

3.7 Impact Charpy test

For impact test, the charpy tester machine is used with 1 joule hammer arm. Samples are cut with 80mm for the test. Needle in the charpy scale has calibrate it before use. Needle position has changed after every specimen test. It should be adjust to measure the proper value of the energy absorbed during the specimen fracture. Fracture energy and impact energy is to be calculated. Dimension of the Specimen is 80mm in length and 6mm in diameter.

Model no. KM-0.5, 3 N. Russian branded charpy impact testing equipment with 2 joules hammer arm used to test the virgin LDPE specimens.

Figure 3.9 Impact Charpy Testing Machine

3.8 Differential Scanning calorimetry

Differential scanning calorimetry (DSC) was used to analyses the thermal properties of the materials like Melting temperature (T_m), Crystallization temperature (T_c) and heat fusion (H_f) and degree of crystallinity (X_{cr}). DSC tests were performed by DSC7 instrument which is produced by Perkin Elmer, United States of America. For measurements of this thermal properties are according to the standard ISO 11357. In our research work, the mass of the specimens was 8.0 ± 0.1 mg.

Each Sample were weighed at 8.0 ± 0.1 mg and then packed in the small pan and sealed it by the puncher. Later for the operation, the work sample pans are placed in the DSC7 instrument and it is covered by the aluminium cover. Meanwhile the reference spot (without sample) also covered by aluminium cover. Using the software, working conditions of DSC the program is monitored and set according to the specifications of sample materials and time and temperature range for the operations. All the specimen measurements are performed under nitrogen atmosphere. Rate of heating and cooling process are carried out at 20 °C/min. All tests are following in a same procedure, (i.e.) after the sample placed in the pan, the experiment is about to carry before that the DSC is calibrated and set the working conditions steps, (i.e). First holding the sample at 0 °C for 5 mins, then heating it from 0 °C to 170 °C, again holding at 170 °C for 5 mins, then cooling it from 170 °C to 0 °C, once again holding it for 5 mins at 0 °C, for getting the accurate measurements of heating stage, the sample again heating from 0 °C to 170 °C to complete the process.

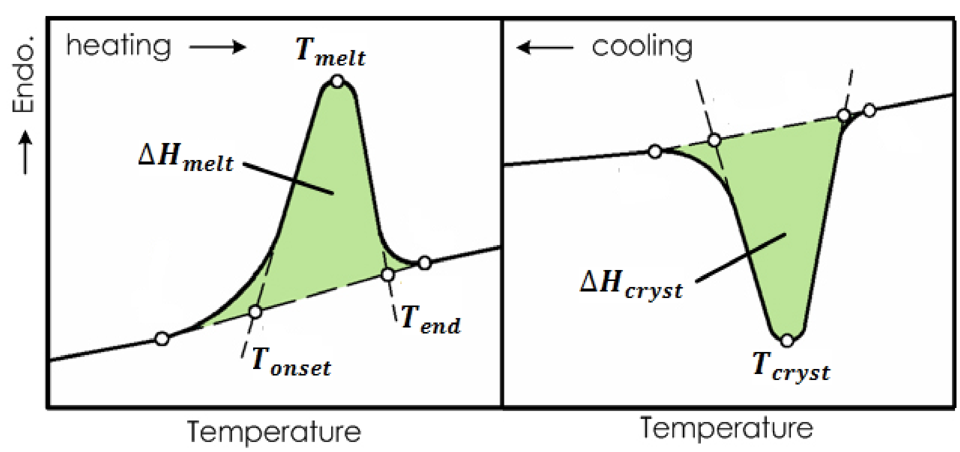


Figure 3.10 Schematic view of thermal properties and its parameters [39]

Tm – represents the melting temperature, which is the heating flow reaches the maximum value during the melting peak, Tc – represents the crystallization temperature, which is the heating flow reaches the minimum value during the crystallization peak [39].

3.9 Optical Microscope

Used conditions

The morphology of all WPC samples was studied with an optical microscope Axioscop 2 MAT (Carl Zeiss, Germany), appointed with polarized light equipment. Slices with nominal thickness 10 μm were cut from each 0%, 20% and 40% WPC sample and observed with polarized transmitted light under magnification of 100x, 200x and 500x at room temperature. The analyser was installed perpendicularly to polarizer. All the images were captured by AxioCam HRc digital camera with resolution 1300×1030 pixels.

These microstructures are used to identify the crystallization and the clear view of interaction between the wood and LDPE in the sample. During this microscopy test, the samples structure is captured with colour image and also this sample can use for some other microscopy test. Samples from different composition like 0%, 20% and 40% are used to test at the room temperature with different magnification. All those structures are used to compare each other to analyse the crystal structure.

4. RESULTS AND DISCUSSION

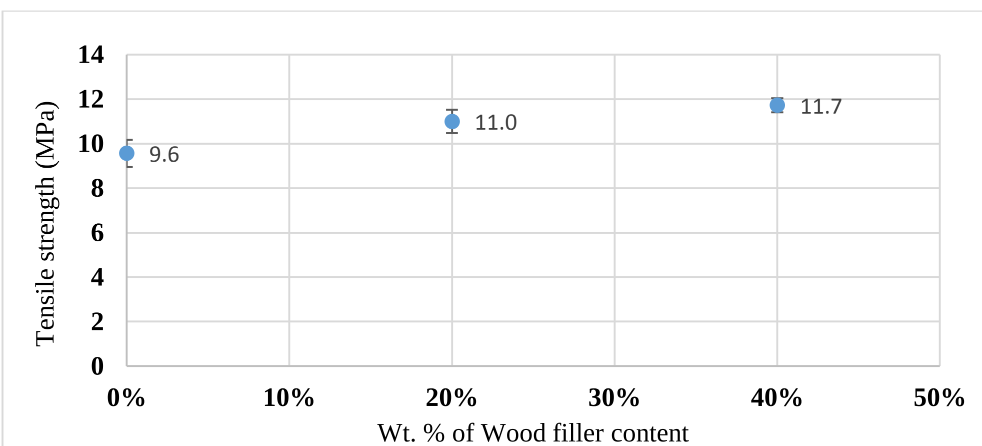
4.1 Mechanical Properties

`To evaluate the mechanical properties of the Wood filled polymer matrix composites, we can observe using the figure/graph 1 to compare the maximum tensile strength, bending strength, and fracture energy of the LDPE polymer compared with wood filled polymer composites without any addition of additives or chemical agents. Tensile test, bending test and impact charpy test are conducted to examine the material properties and its strength.

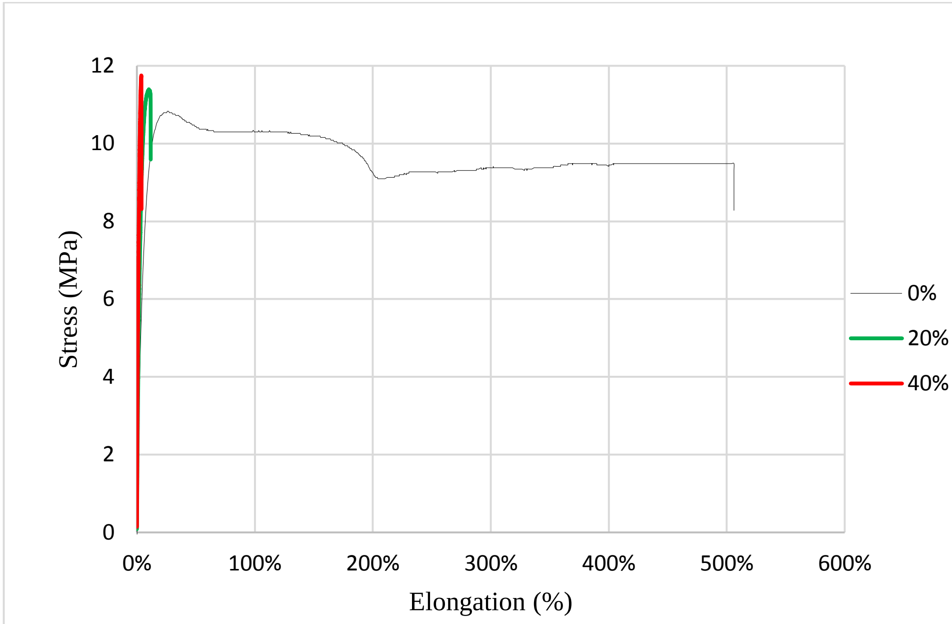
4.1.1 Tensile Test

For tensile test, the experiment is carried out using Tinius Olsen H25Kt machine at the room temperature with crosshead speed at 25mm/min, the dimension of the specimen is 70mm length and 6mm diameter. The wood polymer composite is tested and the results are compared using the graph.

Graph 4.1 reveals that the mechanical properties of WPC, when increase in wood filler content with plastic shows increase in tensile strength compared to the neat LDPE (polymer). And also it shows that slight difference in strength between the 0%, 20% and 40% of Wood filled Polymer composites.

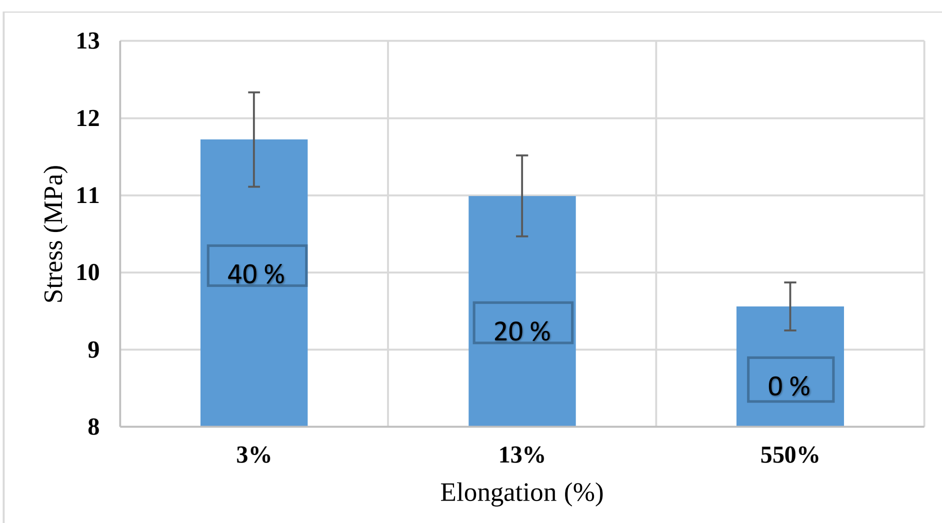


Graph 4.1 Tensile strength (MPa) vs Weight of wood content (%)



Graph 4.2 Tensile Stress (MPa) vs Elongation (%) curve for specimens a) 0%, b) 20%, c) 40%

Graph 4.3 reveals that the ductility of the plastic is much more than the wood filled polymer composites. 40% WPC Specimen has more brittleness than the 20% WPC. When the tensile force is applied, the deformation of 20% and 40% of wood filled composites are having minimum value than the 0% (Pure LDPE/polymer). Addition of wood particles leads to increase in strength and decrease in characteristics of the material.

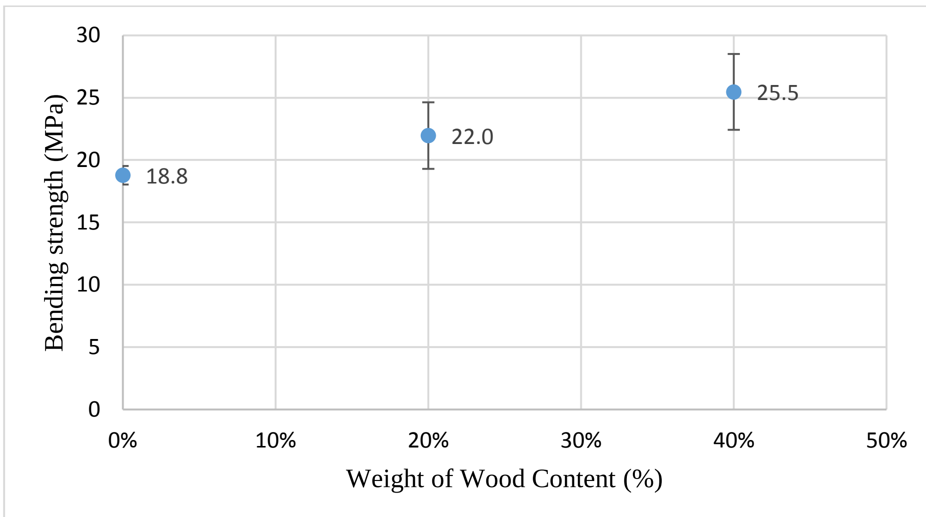


Graph 4.3 Stress (MPa) vs Elongation (%) diagram for Wood/LDPE composites

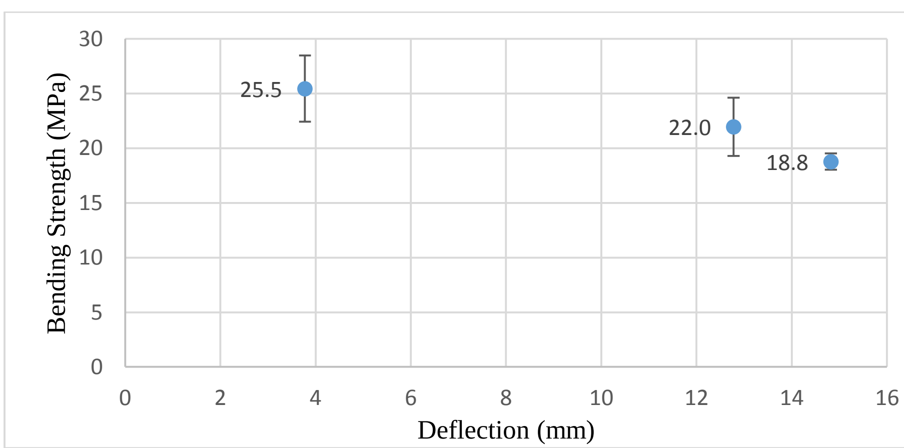
For the above two results, we used the student t distribution because number of specimens are small. 95% confidence level is used to find the Student t distribution, which is used to show better comparable results.

4.1.2 Bending test

For bending test, the experiment is carried out using Tinius Olsen H25Kt machine at the room temperature with crosshead speed at 10mm/min, the dimension of the specimen is 80mm length and 6mm diameter. The wood polymer composite is tested and the results are compared using the graph.

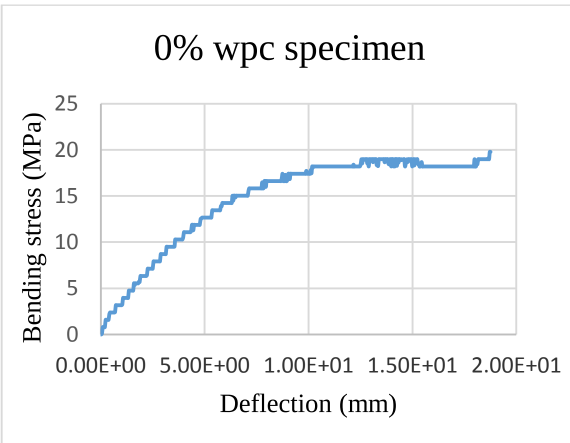


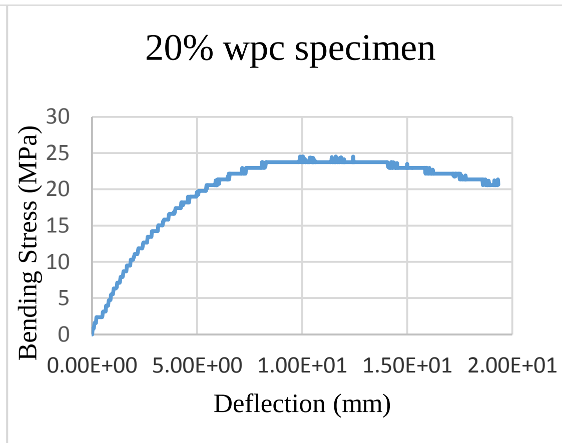
Graph 4.4 Bending strength (MPa) vs weight of Wood content (%)



Graph 4.5. Bending strength (MPa) vs Deflection (mm) for 3 Point bend test

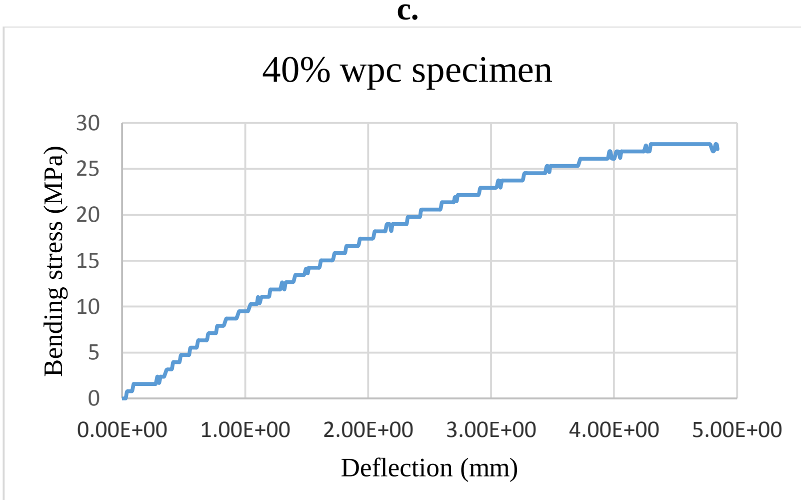
From above graph 4.4, reveals that the increasing in wood filler content to the polymer composite shows that increase in bending strength of the material when adding 20% of wood content, when adding 40% of wood filler content strength increases much more because of addition of wood. There is no very large difference between the LDPE and 20% of WPC because of addition of wood content ratio is very less compare to plastics. There is significant difference between the LDPE and 40% of WPC, because in this case the wood filler content is almost equal to the plastic content. So, there is a chance of less adhesion when we are not adding the chemical composition to the WPC. For the comparison of results, the confidence level 95% is used in Student t distribution. Below graphs shows the detailed view of all specimens when testing in the 3-point bending test method.





a. **b.**

c.

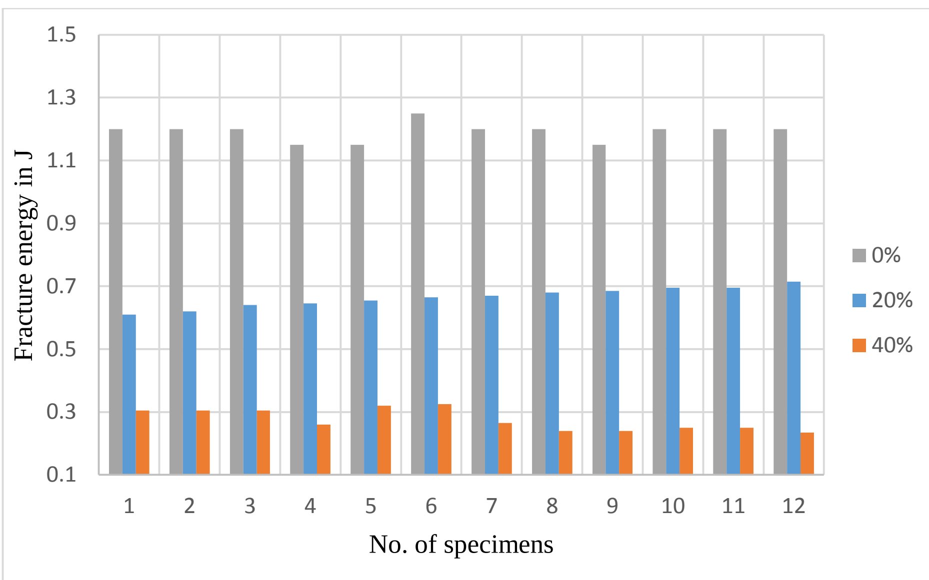


Graph 4.6 Bending stress (MPa) vs Deflection (mm) curve for specimen's a. 0%, b. 20%, and c. 40%

4.1.3 Impact Charpy test

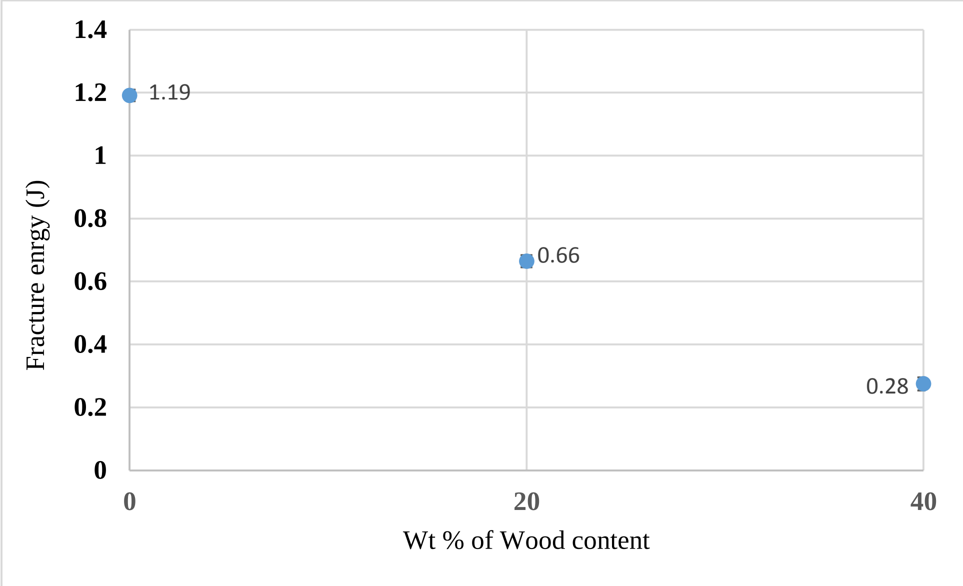
In this method, the test specimen is chosen for the dimension of 80mm in length and 6mm in diameter. Hammer (arm) of the charpy machine has having an arm capacity of 1 joule for this experiment. Fracture energy is calculated from this experiment with the 12 sample specimens. Graph 4.6 shows that nearly all specimens are fractured at similar range of energy. Plastics are having more toughness, so it is difficult to break the LDPE material. In 1 joule arm charpy tester, the 20% and 40% of wood filler polymer composites are used to test the fracture energy.

There is slight difference in fracture energy between the several sample specimens which is tested. In chart Graph 4.6, the blue lines represents the 20% of wood filler content polymer composite which absorbs more energy while hammer hitting the specimen to break. Other hand, the line which represents the 40% of wood filler content polymer composite has absorbs less energy to fracture.



Graph 4.7 Fracture energy (J) vs Specimens

In graph 4.7, the plot shows the wood filler content vs Fracture energy, according to the results the results that the fracture energy decreased rapidly as a wood content increased to the wood polymer composites. Due to uneven composition of wood and polymer in the specimen, the property may change due to wood particles enriched in particular area. By chance, this also makes the lower fracture energy in high wood content particle.



Graph 4.8 Fracture energy (J) vs Weight of Wood filler content (%)

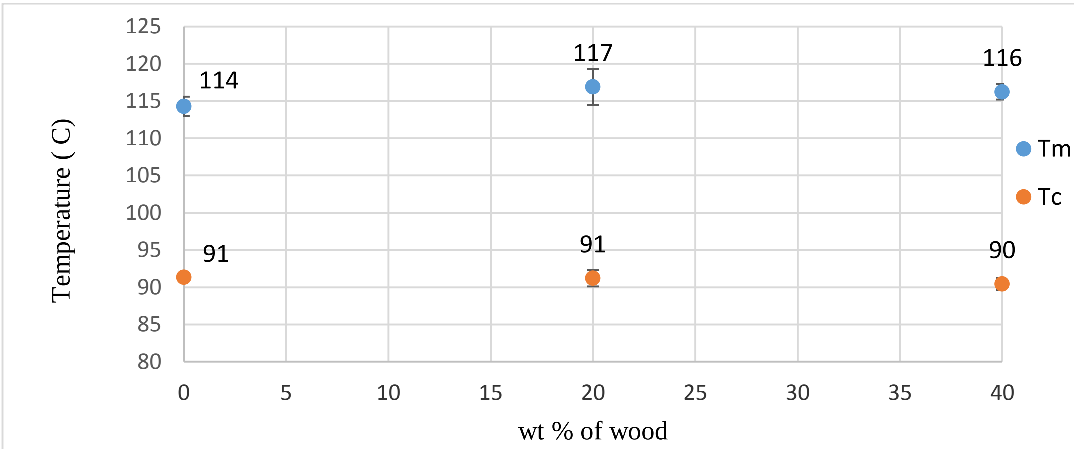
4.2 Thermal Properties

Thermal properties are used to analyses the thermal stability of the material and also by measuring the specimen with the heating and cooling temperatures, checking the glass transition materials. Differential scanning calorimetry is used to measure the thermal properties of the wood polymer composites.

4.2.1 Differential Scanning Calorimetry (DSC)

DSC is used to investigate the thermal properties like melting temperature (Tm) and crystallization temperature (Tc). In this research study, the sample is tested by using temperature program which is already programmed before starting to the experiment. WPC are having slight differences in their properties when adding the wood content to the composites.

In graph 4.8 shows that the melting temperature of the material reaches high when addition 20% of filler contents to the composites. But there is no significant difference between the other two contents (0% and 40%). Similar results shows for the crystallization temperature of the material. Here also the 20% of wood filled polymer composites shows little higher temperature value than other wood content polymer matrix. In this study, the crystallization temperature also shows very common temperature value, may be if we had any chemical agents to the composition, then the crystallization rate will increase.



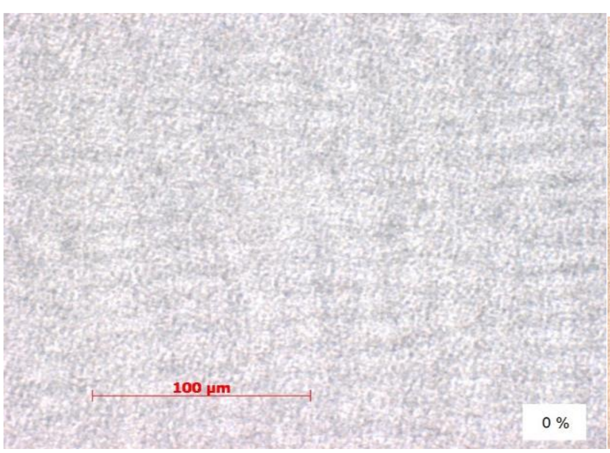
Graph 4.9 Melting (Tm) and Crystallization (Tc) peak temperature vs Weight of wood (%)

4.3 Morphological Properties

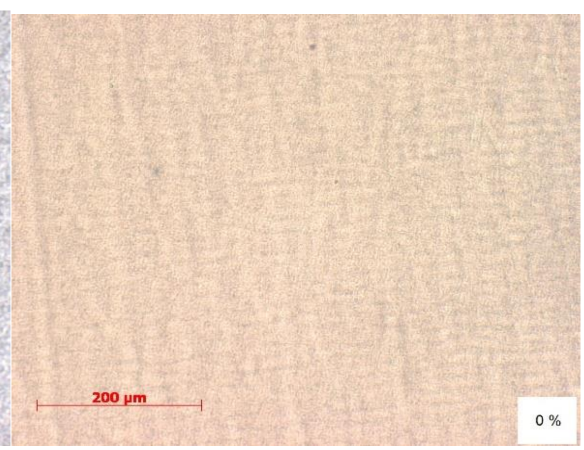
4.3.1 Optical Microscope

In this study, the Specimens of 0%, 20%, and 40% of WPC are tested under the microscope with polarized light to find the grain structures and diffusion of wood polymer composites.

0% of wood filled WPC



a.



b.

c.

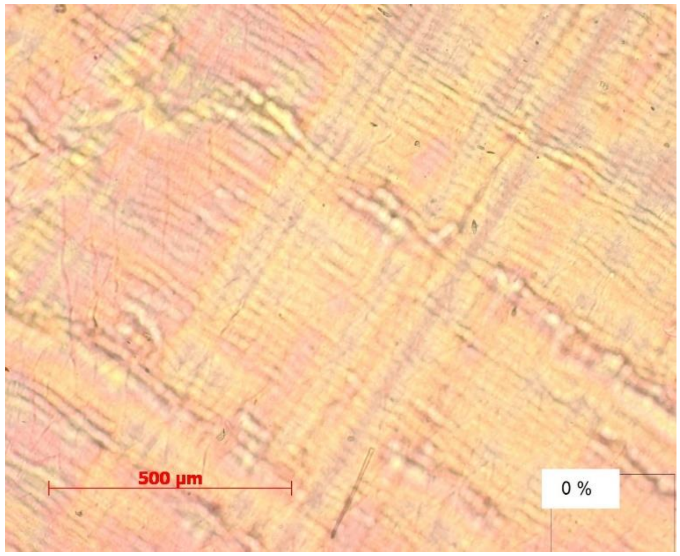
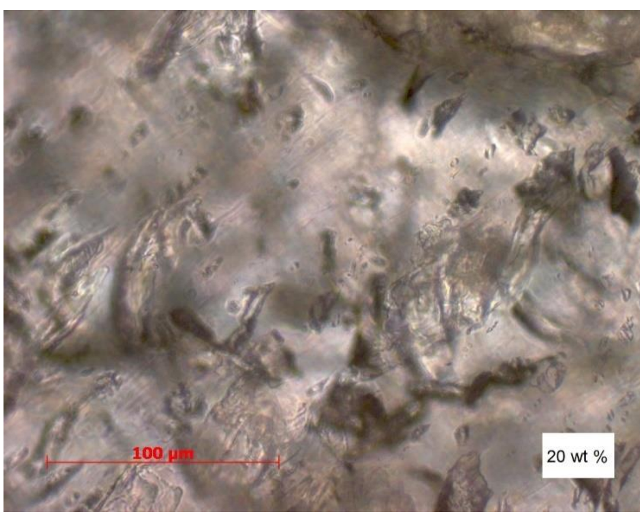


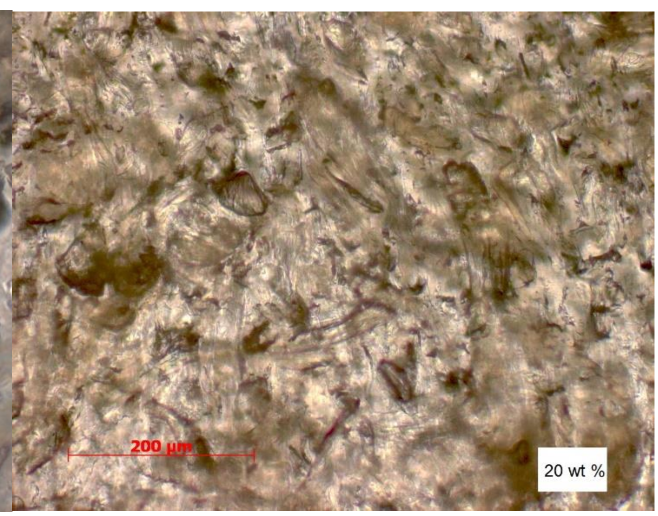
Figure 4.1 Microstructure image of 0% wood filled WPC, a) 100x, b) 200x, c) 500x

From above Figure 4.1, the microstructure has shown that LDPE grains are having neat grain structures, especially in the magnification 100x and 200x it shows that the LDPE is melt properly and the specimen has having good smooth surface.

20% of wood filled WPC



a.



b.

c.

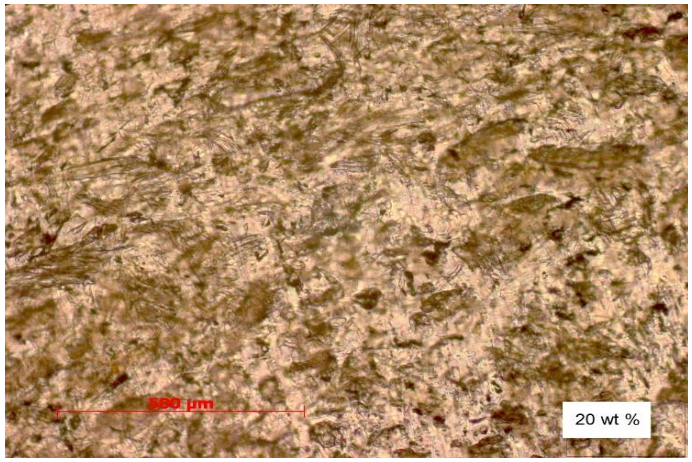
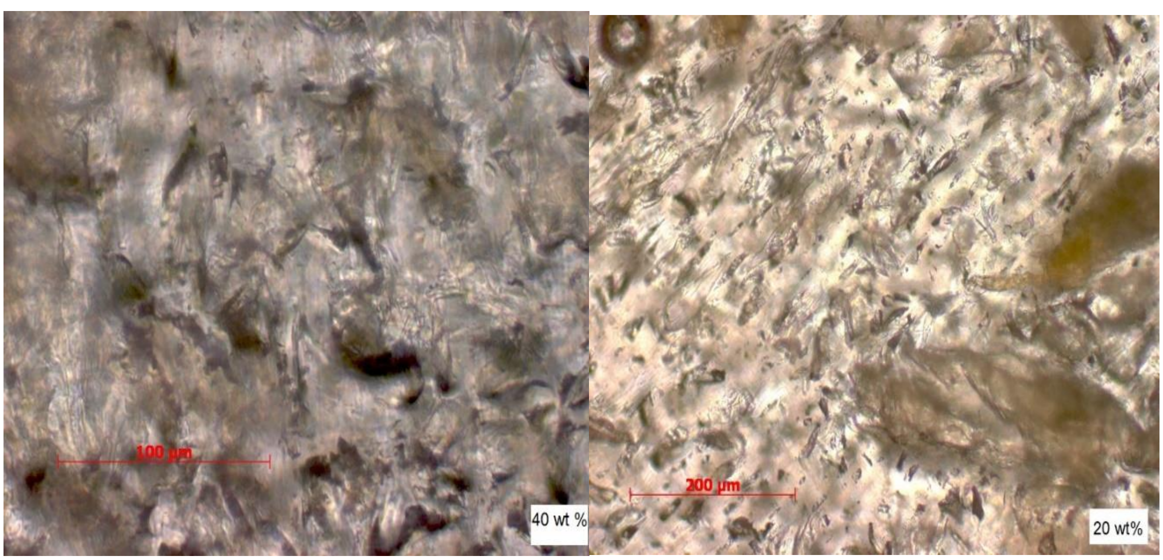


Figure 4.2 Microstructure image of 20% wood filled WPC, a) 100x, b) 200x, c) 500x

In the figure 4.2, it shows the microstructural image of 40% wood filled WPC. Magnification 100x shows that the wood particles are formed like grain in some portion, and also clearly visible that more LDPE has appeared when we see this structure closely. But the specimen has turned bit darker in color than 0% (LDPE) wood filled WPC. Forming of grains leads some places have void and the diffusion is not happened perfectly. Chances of fracture increases due to this composition.

40% of wood filled WPC



a. **b.**

c.

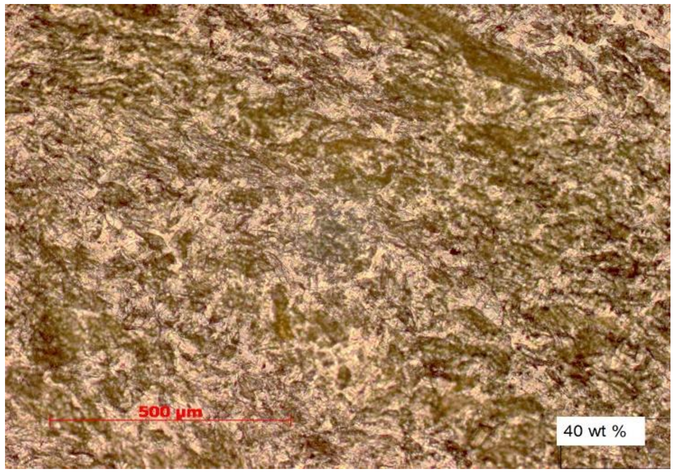


Figure 4.3 Microstructure image of 20% wood filled WPC, a) 100x, b) 200x, c) 500x

From figure 4.3, the microstructures reveals that when addition of wood contents to the composite, the grains forms and it not diffused properly. This is will create void and makes the specimen weaker because of the poor interfacial adhesion between the components. Sample has easily occurs fracture due to the non-diffused grains in the structure. Material has looks in much darker in color than other 20% and 0% of WPC.

CONCLUSIONS

The main objective of this research was to study and compare the mechanical, thermal and morphological properties of WPC on its compositions and processing conditions. The composition of Spruce pine wood flour and the low density polyethylene has mixed in the ratio of 0%, 20% and 40%. Processing conditions are wood flour drying, sieving and extruding with LDPE in twin screw co-rotating extrusion machine at 150˚C and finally sample prepared by gravimetric casting machine at 130 ˚C.

The following results is obtained by performing the mechanical tests, thermal and morphological tests for analyzing the effects of wood polymer composites.

- **Tensile test** showed that the increasing in wood content increases the tensile strength and brittleness. Overall wood content of 0-40% shows the total tensile strength increased by 18 % (9.6-11.7 MPa). Increase in wood content from 0-20% shows 13% (9.6-11.0 MPa) increase in tensile strength, while addition of more wood content (20-40%) it shows 6 % (11.0-11.7 MPa). Virgin LDPE composite is having more ductility (550%) than 20% WPC (13%) and 40% WPC (3%), therefore the increasing in wood content of composite decreases in ductility and increases in brittleness of the material.

- **In bending test**, the specimen contains 20% of wood the bending strength increases 18% (18.8-22 MPa) with increasing in wood flour. When there is more wood content (20-40%) the bending strength increases 10% (22-25.5 MPa). Bending strength increases with increasing in wood flour content to the composite.

- **In charpy test**, the virgin LDPE absorbs more energy (1.19J) to fracture the composite when the hammer hits. Similarly, 20% and 40% of WPC absorbs less energy (i.e. 0.66 J and 0.28 J respectively) to break the sample when compared to 0% of WPC. Increase in wood flour decreases the toughness of the wood polymer composites, because in the intermolecular structure the presence of more wood grains leads to fracture of the specimen and no proper interaction or mixing between the filler and matrix content.

- **In DSC test**, there is no significant changes in melting temperature (1.67% - 114 to 117˚C) and crystallization temperature (1.02% - 90 to 91˚C) between the compositions of wood polymer composites.

- **In Microscope**, the structural image reveals that in 20% WPC the grains formed smaller in size in few places, but in 40% WPC the presence of voids due to poor interaction between matrix and filler material, some wood particles stick together, this leads to increase in brittleness to fracture the material easily and 40% looks much darker in color.

Therefore, the above results are compared to the wood polymer composites with addition of chemical additives or agents, this will be essential for environmental safety, less weight and reduction of cost in adding both raw material and chemical agents to the composite. As per literature, maintaining wood content at 60% or 50% or even less produced the best mechanical properties. Wood content above 50% or 60% resulted in reduction in all mechanical properties.

REFERENCE

1. ALMAADEED, M.A. et al. Improved mechanical properties of recycled linear low-density polyethylene composites filled with date palm wood powder. In *Materials & Design*. 2014. Vol. 58, p. 209–216.
2. HOMAMI, S.S. et al. Preparation of Wood Plastic Composite from Polyethylene and Bagasse. In *Middle-East Journal of Scientific Research*. 2013. Vol. 14, no. 4, p. 453–455.
3. DARIE, R.N. et al. Evaluation of properties of LDPE/oak wood composites exposed to artificial ageing. In *Cellulose Chemical Technology*. 2011. Vol. 45, no. 1-2, p. 127–135.
4. IZEKOR, D.N. et al. Effects of geometric particle sizes of wood flour on strength and dimensional properties of wood plastic composites. In *Journal of Applied and Natural Science*. 2013. Vol. 5, no. 1, p. 194–199. .
5. D.E.HUDGIN *Handbook of Plastic technology*. 2007
6. CHANDA, M. - ROY, S.K. *Plastic technology handbook*. . 3rd and 4th edition. Ed. Clemson; CRC press, 2006.
7. LISICINS, M. et al. Conversion of Polymer and Perforated Metallic Residues into New Value-added Composite Building Materials. In *Energy Procedia*. 2015. Vol. 72, p. 148–155.
8. Market study: Polyethylene-LDPE Ceresana Research, 2010. Prieiga per internetą: <http://www.ceresana.com/upload/Marktstudien/brochueren/Ceresana_Brochure_Market-Study_Polyethylene-LDPE_2nd_ed.pdf>.
9. STOLF, D.O. - LAHR, F.A.R. Wood-polymer composite: physical and mechanical properties of some wood species impregnated with styrene and methyl methacrylate. In *Materials Research*. 2004. Vol. 7, no. 4, p. 611–617.
10. ROWELL, R.M. *Handbook of Wood Chemistry and Wood Composites*,. . Second Edition. Ed. [s.l.]: CRC Press, 2012.
11. ED SUTTIE, Briefing note for Forestry Commission: An update on Wood Plastic Composites (WPC). In *BRE*. 2007.
12. Compounding and extrusion of wood plastic composites, Coperion K-Tron, 2014. <http://www.ktron.com/industries_served/plastics/woodplastic.cfm>.

13. GOVERNMENT, R.M. et al. Modelling and Statistical Analysis of Ultimate Tensile Strength LDPE/Date Palm Wood Flour Composites. In *INTERNATIONAL JOURNAL OF MULTIDISCIPLINARY SCIENCES AND ENGINEERING*. 2013. VOL. 4, NO. 9.
14. MEDUPIN, R.O. - ABUBAKRE, O.K. Effect of Wood Fibre Characteristics on the Properties of Wood Polymer Composites. In *Journal of Multidisciplinary Engineering Science and Technology (JMEST)*. 2015. Vol. 2, no. 1.
15. SAILAJA, R.R.N. - DEEPTHI, M.V. Mechanical and thermal properties of Compatibilized composites of LDPE and esterified unbleached wood pulp. In *Polymer Composites*. 2011. Vol. 32, no. 2, p. 199–209.
16. MISHRA, A.K. - LUYT, A.S. Effect of sol–gel derived nano-silica and organic peroxide on the thermal and mechanical properties of low-density polyethylene/wood flour composites. In *Polymer Degradation and Stability*. 2008. Vol. 93, no. 1, p. 1–8. .
17. ZHANG, Y. et al. Effects of wood fibre content and coupling agent content on tensile properties of wood fibre polyethylene composites. In *Holz als Roh- und Werkstoff*. 2008. Vol. 66, no. 4, p. 267–274.
18. NDLOVU, S.S. et al. LDPE–wood composites utilizing degraded LDPE as compatibilizer. In *Composites Part A: Applied Science and Manufacturing*. 2013. Vol. 51, p. 80–88.
19. LI, X. et al. The utilization of bamboo charcoal enhances wood plastic composites with excellent mechanical and thermal properties. In *Materials & Design*. 2014. Vol. 53, p. 419–424.
20. MIČUŠÍK, M. et al. Effect of crosslinking on the properties of composites based on LDPE and conducting organic filler. In *European Polymer Journal*. 2006. Vol. 42, no. 10, p. 2379–2388. .
21. ALMAADEED, M.A. et al. Mechanical, sorption and adhesive properties of composites based on low density polyethylene filled with date palm wood powder. In *Materials & Design*. 2014. Vol. 53, p. 29–37.
22. LEU, S.-Y.et al. Optimized material composition to improve the physical and mechanical properties of extruded wood–plastic composites (WPCs). In *Construction and Building Materials*. 2012. Vol. 29, p. 120–127. .
23. FATIH MENGELOGLU - KADIR KARAKUS Mechanical properties of injection molded foamed wheat straw filled HDPE bio composites the effects of filler loading and coupling agent content. In *Bio Resources*. 2012.

24. Injection moulding for plastics<//lerablog.org/business/industry/what-are-the-environmental-effects-of-plastic- injection- moulding/>.
25. HAROLD F. GILES et al. *The Definitive Processing Guide and Handbook*. . [s.l.]: William Andrew, 2005. ISBN 978-0-8155-1473-2.
26. GARDNER, D.J. - MURDOCK, D. Extrusion of wood plastic composites. In *University of Maine, Orono ME* [interaktyvus]. 2010. [žiūrėta 2016-05-30].
27. DAVIS, J.R. *Tensile testing* [interaktyvus]. . [s.l.]: ASM international, 2004.
28. RAGER, A. et al. Numerical Analysis of the Three Point Bend Impact Test for Polymers. In *International Journal of Fracture*. 2005. Vol. 135, no. 1-4, p. 199–215. .
29. MUSTAFA AKAY *Introduction to polymer science and technology*. . [s.l.]: ventus Publishing APS, 2012. ISBN 978-87-403-0087-1.
30. Instron about flexure test: Illinois Tool Works <http://www.instron.us/en-us/our-company/library/test-types/flexure-test>.
31. LI, D. et al. Charpy impact properties and failure mechanism of 3D MWK composites at room and cryogenic temperatures. In *Cryogenics*. 2014. Vol. 62, p. 37–47. .
32. TÓTH, L. et al. Historical background and development of the Charpy test. In *European Structural Integrity Society*. 2002. Vol. 30, p. 3–19. .
33. HÖHNE, D.G.W.H. et al. *Differential Scanning Calorimetry*. . 2nd revised and enlarged edition. Ed. [s.l.]: Springer-Verlag Berlin Heidelberg New York, 2003. ISBN 978-3-642-05593-5.
34. KUBYSHKINA, G. et al. The Influence of Different Sterilization Techniques on the Time-Dependent Behaviour of Polyamides. In *Journal of Biomaterials and Nano biotechnology*. 2011. Vol. 02, no. 04, p. 361–368. .
35. KAZEMI NAJAFI, S. Use of recycled plastics in wood plastic composites – A review. In *Waste Management*. 2013. Vol. 33, no. 9, p. 1898–1905. .
36. ZININ, P. - MISRA, A. Advanced Techniques in Geophysics and Material science: HIGP, University of Hawaii, Honolulu, USA.
37. The Wood Database: <http://www.wood-database.com/lumber identification/softwoods/spruce-pine/>.
38. Properties of Virgin Low density Polyethylene" OKITEN 245 S by Dioki Croatia. <http://www.globichem.com/pdf/polyethyene-pe/ldpe/LDPE%20-%20245S.pdf>.

39. JOAMIN, G.-G.et al. Time-Dependent Properties of Multimodal Polyoxymethylene Based Binder for Powder Injection Moulding. In *Journal of Solid Mechanics and Materials Engineering*. 2012. Vol. 6, no. 6, p. 419–430.
40. Polymer Structures. In *Introduction to Materials Science*. University Tennessee, Dept. of Materials Science and Engineering.