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Dedicated to My Beloved Parents

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Abstract

Now-a-days the world is trying to reduce the pressure on solid wood utilization and also the use of toxic materials in composite production. In this study an attempt was made to produce a wood- plastic composite board by using sawmill wastage of Mahagony wood and low density polyethylene (LDPE). In production of composite boards a lot of factors are interacting with each other and actual optimized level of parameters which give the better result are very difficult to find out for this kind of complex interaction of production variables. In this paper multi-response optimization process was used to optimize the process parameters of composite board production which are -mixing ratio, fire retardant (%) and pressing time (min). It was try to investigate the effect of these three process parameters in the mechanical and physical properties of the composite board. Afterwards, Box–Behnken design was performed as response surface methodology (RSM) with desirability functions to attain the optimal level of mixing ratio, fire retardant (%) and pressing time (min). Here the maximum MOE and MOR were found by the optimal conditions of mixing ratio 60:40, pressing time 9 minutes and 0 fire retardants percentage. The optimized MOR and MOE were found 13.129 N/mm² and 1781.0 N/mm² respectively. Finally, by using optimized levels of parameters a confirmation study was executed which showed well response to the predicted model.

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Chapter One: Introduction

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1.1 *Background of the Study*

The development of civilization from the primary stage to the present day's highly advanced technology has been evolved with dependence on wood because wood is such a material upon which human are fully depend from the cradle to the grave. Its availability in the universe and its easily usable quality has made wood an essential material for human survival (Panshin and De Zeeuw, 1980). It is said that– *we may use wood with intelligence, if we understand wood* (Wright, 1928).The greater the technological advances, the more diverse and sophisticated uses of wood have been found (Panshin and De Zeeuw, 1980). More and more the use of it increases the more and more scarcity of wood and degradation of biodiversity increases. Now-a-days, technological advances have made it more useful (Miller, 1999). Such a technology is making of composite products which is a useful alternative to solid wood in economic concern and also helps in conservation of scarce forest resources. Composite products such as ply wood, oriented standard board (OSB), hardboard, particle board, fiberboard, veneer board etc has created their demand both for interior and exterior construction and furniture manufacturing throughout the world (youngquist, 1999).

In early days, most of the marketable wood based composites were bonded with urea formaldehyde and phenol formaldehyde resin. Now emission of formaldehyde has become great concern to the environment from these types of composites (Lee *et al.*, 2002). For this reason, Wood-Plastic Composite (WPC) was introduced by the year 1970 in Italy to resolve this problem (Pritchard, 2004). Environmentally friendly alternatives to these wood- plastic materials include products that use polyethylene (Kuo *et al.*, 1998; Rahim, 2009). Different types of polyethylene are used in preparing WPC like- low-density polyethylene (LDPE), high density polyethylene (HDPE), and linear low-density polyethylene (LLDPE) etc. (Chen *et al.*, 2007). However studies on wood composites based on low-density polyethylene (LDPE) are very limited (Yamashita *et al.*, 1999). So in this study; LDPE is used as a binding material in a combination with wood flour to prepare the wood composite product to characterize its physical and mechanical properties.

Due to the wide range of polymer application, its flammability characteristic has attracted considerable attention. The combustion of polymers follows a cyclic pattern of preheating, decomposition, ignition, and combustion. In the presence of a flame source, the polymer becomes preheated. Boron based fire retardants for example combination of Borax ($Na_2B_4O_7.10H_2O$) and Boric acid (H_3BO_3) have showed better result in improved fire performance (Stark, *et al.*, 2010). In a study it has been found that, the composites with borax/boric acid had better dimensional stability and strength in the bending (Ayrilmis, et al., 2012). So it can be said that, the uses of fire retardant have an impact on the mechanical properties of wood. In this study, mixer of Borax and Boric acid is used as a parameter to find out the effect of using fire retardants on mechanical properties of wood.

Composite board has a homogenous structure and can be manufactured in different sizes, thickness, densities and grades for numerous uses, making it a desirable material with which to work (Ives, 2001). There are a lot of factors which have an influence on the board quality manufactured with LDPE like Mixing ratio, temperature, time, pressure etc (Atuanya, et al., 2011). In this study, the mixing ratio, pressing time, fire retardant (%) are used as the factors of production of wood-flour LDPE composite.

Recently, statistical experimental design has been used in manufacturing of wood composites in several studies (Jun, et al., 2008; Tabarsa et al., 2011; Azizi et al., 2011; Nirdosha, et al., 2011). But, use of any modern industrial optimization tools is very limited in characterizing the factors responsible to the quality production of wood composites. Which results in the waste of raw materials, reagents and increase in other productions oriented cost which can adversely affect forest resources and biodiversity (Islam et al., 2011).

Response surface methodology (RSM) is a collection of statistical and mathematical techniques useful for developing, improving and optimizing process that is dedicated to the evaluation of relations existing between a group of controlled experimental factors and the observed results of one or more selected criteria (Myers, 2002). In this study, an attempt was made to optimize the process parameters of wood composite board production by using multi-response optimization process. Box–Behnken design was performed as response surface methodology (RSM) to find out the optimum combination of mixing ratio, Pressing time and fire retardants that affect modulus of rapture (MOR) and modulus of elasticity (MOE) of particleboard production and physical properties of the boards.

1.2 Objective of the Study:

The objective of this study was to find out the best combination of parameters on the mechanical and physical properties of particleboard by using the multi-response optimization process. A motive of the study was to establish an understanding of processing and pressing techniques of composite manufacturing made from the LDPE and wood flour. This study also apprises to reduce the uses of solid timber by manufacturing of LDPE composite. An attempt was made to improve the quality of board by using LDPE as a binder and to reduce the emission of resin from resin based composites. It helps in ensuring the rational and optimum use of forest resource and other reagents by using the multi-response optimization process.

The study was undertaken with the following main objectives:

- To access the application of multi-response optimization process for the production of LDPE Wood flour composite board.

- To manufacture LDPE wood flour composites and determine the effects of processing parameters on physical and mechanical properties of composite board.

Chapter Two: Literature Review

Chapter Two: Literature Review

2.1 Wood Plastic Composites (WPC)

2.1.1 What is WPC?

Wood Plastic Composite (WPC) market share has been growing rapidly in the last few decades. The composites are typically used in the commercial residence decking. Other than that, they could be used as rails, floorings, window and door frames and automotive interior. For decades, traditional solid wood decking has always posed many problems for thousands of homeowners. In contrast, the demand for decking keeps increasing. Freedonia Group Inc estimated that by 2013, the decking in the United States would reach 3.6 billion lineal feet with 2.1% annual growth (Freedonia Group Inc, 2009).

Therefore, there was a need to invent a new material for the deck so that it could last longer, was more durable and at the same time had low maintenance cost. For those purposes, scientists and industrial representatives have conducted a lot of research in the past and they successfully developed a mix of thermoplastic and wood flour or fiber. They named the product Wood Plastic Composites (WPCs). The composite materials which are produced by combining thermoplastic polymers with natural fibers derived from wood and agricultural crops are called WPC (Wolcott and Smith 2004). A wood plastic composite, or WPC, is defined as a material that consists of wood fiber in a thermoplastic matrix, and the wood fiber must constitute greater than 50% of the mass of the composite (ASTM, 1995). WPCs are gaining in popularity for specialized applications because of several positive qualities that come from the combination of wood and plastic. These attributes include low maintenance requirements, high moisture resistance, and improved durability with respect to checking, splintering, decay, termites and marine organisms. Many of these WPCs can also be machined and installed in the field using conventional wood working tools.

2.1.2 Brief History of WPC

Historically, the idea of mixing wood fibers and thermoplastic to develop a more durable product was suggested by a number of researchers who realized that wood fibers could be a good replacement for inorganic fillers in thermoplastic such as glass, calcium carbonate and talc (Rowell, 2006). Its abundance, low cost and relatively lower weight made wood fibers a perfect material for fillers and reinforcements (Clemons, 2000). Furthermore, the environmental concern of using renewable materials was also a crucial factor that led to the development of the product (Rowell, 2006).

The first WPC was manufactured in 1983 by American Woodstock, an automotive interior company based in Sheboygan, Wisconsin. Polypropylene and wood flour, in the ratio of 1:1, were mixed and extruded into a flat sheet. The sheet was then formed into various shapes for automotive interior purposes. In 1990s, the development of WPC grew even more rapidly. Researchers and industrial representatives from both wood and plastic industries held a number of conferences such as the ones in Madison, Wisconsin in 1991 and Toronto, Ontario in 1992. The objectives were to share ideas and latest technology in Wood Plastic Composites. Furthermore, they would experiment on the best ratio between the wood fibers and plastics contents. In 1993, Anderson Corporation in Bayport, Minnesota manufactured wood plastic PVC for doors with 40% wood content. This then led to the future production of wood PVC window composites. Another company, Strandex Corp even patented its 70% wood fiber content composites that would be used as building components (Clemons, 2000).

Recently, consumers have started to use WPC in other products such as residential decks, tables, park benches, landscape timbers and floorings. Residential decking market is by far the biggest and fastest growing market for WPC, followed by window and door profiles (Clemons, 2000).

2.1.3 Advantages and Disadvantages of WPCs

Wood Plastic Composites combine the best features of wood and plastics (Tangram, 2002). In fact, the inclusion of wood fibers in the production process results in not only a lower production cost but also lower density, UV resistance and high specific strength product. Meanwhile, the thermoplastic part of the matrix acts as a barrier to natural degradation (Rude, 2007).

WPC is a very highly flexible product due to the extrusion process it undergoes. Therefore, it can be formed into almost any kind of shapes according to the needs. Moreover, unlike natural wood products which are prone to biodegradation, water absorption and weather changes, WPC is more durable and mold resistant, making it very suitable for outdoor applications. It is also more cost-effective compared to plastic products due to the low costs of raw materials. Some other benefits of WPC are listed below (Rude, 2007; Tangram, 2002; Anton, 2009):

- The raw materials include wood waste and recycled plastic, thus reducing the cost significantly.
- The price is competitive compared to other timber products.
- It is available in various types of designs, including wood grain designs and finishes.
- It is recyclable, making it environmentally friendly.
- The plastic part of the composite makes it resistant to changes of weather condition (humidity and temperature).
- It is UV resistant if UV stabilizers were added in the production process.
- It can last two to three times longer than solid wood products without changing its original appearance.
- It is easy and less expensive to maintain.
- It is safe due to the absence of splinters and chips.
- It is easy to install due to the Do-It-Yourself concept.
- The warranties offered which typically cover insect damages, splintering and splitting range from 10 to 20 years

In contrast, just like other products, WPC also has some drawbacks. Interfacial adhesion between the wood fibers and the thermoplastic is very limited and depends highly on the coupling agents which initialize the bond (Wolcott, 2010). The problem arises when the wood fiber which has polar nature does not interact well with the thermoplastic which is non-polar. The reasons behind this are the poor distribution of wood fiber, change in thermoplastic morphology and reactions between the lubricants and coupling agents (Rude, 2007). The drawbacks of WPC are mostly technical problems and thus can be improved by adopting better processes.

2.1.4 Production of WPCs

Manufacturing WPC is not a simple process. Many factors such as melting temperature, mixing process and raw material selection have to be considered to produce decent quality WPCs. One major concern with the production of WPC is the complexity of compounding two naturally different materials; woods and plastics (Tangram, 2002). Unfortunately, the compounding process is the most important step which determines the quality of the finish products. Insufficient compounding time would result in poor dispersion of plastic into wood particles and poor wetting of the wood fibers. Hence, low mechanical properties of the final products. On the other hand, over compounding will severely damage the fibers (Wolcott, 2010). Another concern is the selection of the raw materials as one type of material would perform better than the others, thus affecting the final quality.

Therefore, it is very crucial to have an excellent and well controlled production process. This segment will discuss in depth about the raw materials used as well as the production methods employed.

2.1.4.1 Raw Materials

Raw materials play an important part in the manufacturing process. In general, one needs three different materials to produce WPC; namely: woods, plastics and additives.

(i) Woods

Wood, in the process, acts as fillers or reinforcements due to its ability to increase the mechanical properties of the final product. Research has shown that wood fibers contribute to better stiffness and strength when compared to other types of fillers such as glass and talc. Another role of wood in the process is to lower the usage of polymer in the process (Wolcott, 2010). Not only is polymer expensive, but also can harm the environment. There are two kinds of wood products that could be used in the WPC production process; they are wood flour and wood fibers. Wood flour used usually range from 40 to 60 meshes in size. Wood fibers, on the other hand, are larger in size and this sometimes can be rather challenging to process. Moreover, he mentioned that production with longer fibers may require more advance compounding technology; the one that could evenly distribute the wood in the thermoplastics without damaging the fibers (Rowell, 2006). Therefore, using wood flour will be an ideal solution to the problem as it minimizes the fiber shrinkage issue.

Pine, maple and oak are predominantly used in the WPC manufacturing process. The species selection is influenced mainly by their availability rather than the mechanical properties (Rowell, 2006). Prof. Hansmann, in his article, "Flüssiges Holz", believed that rice husks, bamboo and cellulose could be the alternatives to wood flour (Hansmann, 2007).

(ii) Plastics

Another main components needed in the process is the polymer. Depending on its reaction to heat, there are two different kinds of polymers; namely: thermosets and thermoplastics. Thermosetting polymers would polymerize and gain stiffness as temperature increases whereas thermoplastic polymers would melt or soften under the same condition. Furthermore, polymers are divided into two categories based on their morphology; they are: amorphous polymer and crystalline polymer. While crystalline thermoplastic would melt under high temperature, the amorphous thermoplastics will only soften. However, at a certain point it will behave like a melting polymer (Wolcott, 2010).

Several kinds of polymers are available. Nonetheless, only a few could be employed in the WPC manufacturing process. Some of them are Polystyrene (PS), Polyvinyl Chloride (PVC), Low Density Polyethylene (LDPE), High Density Polyethylene (HDPE) and Polypropylene (PP). All those polymers have one property in common which is having melting or softening point lower than the thermal degradation temperature of wood (≈ 210°C). Polymers whose melting point is under 210°C fall under polyolephins category. Among those polymers, Polyethylene is the most common material that is used in the production process (Wolcott, 2010). Caulfield claimed that 83% of 685,000 tons of WPC consumed in North America is made of PE. Polypropylene (PP) comes second with 9% of total production while 7% of WPCs is made of PVC. The remaining 1% is produced out of other types of polymers (Caulfield, 2005). Thermoplastics are usually available in the form of pellets, powder, flakes or films as well as from some recycled products such as milk jugs, battery casing, and plastic shopping bags (Wolcott, 2010).

(iii) Additives

The major purposes of using additives are to improve the diffusion of the wood flour into the thermoplastics, advance the mechanical properties (MOE and MOR) and other miscellaneous purposes such as smooth the surface, protect against UV light and heat and reduce density.

The type of additive that is used the most is compatibilizers. It serves as glue and thus linking the woods and the thermoplastics together. As discussed in the previous section, wood is hydrophobic whereas thermoplastic is hydrophilic. Therefore, to be able to blend the two well, one needs a certain medium which is available in the form of Maleic Anhydride Grafted Polypropylene (MAPP) or Maleic Anhydride Grafted Polyethylene (MAPE). MAPP and MAPE have a chemical structure in which each end of the molecules can form a bond with either hydroxyl group of wood or melted thermoplastic. Another common type of additives is the colorant. It is usually used in the process to enhance the color of the finish products. Some colorants may even function as a UV protector. To ensure the consistent color between wood and plastic, colorants have to be added separately to each material before the extrusion process. Otherwise, the final products' surface will exhibit specks of lighter or darker color. WPC surface color tends to grow fainter over time due to exposure to UV light. Besides UV protector, pigments can also be added to minimize this phenomenon. Another solution to the problem is by co-extruding UV-stable layer over the WPC (Rowell, 2006).

To reduce the density of the final products, foaming agents may be dispersed to the thermoplastic material before the compounding process. The foam will generate bubbles which will then penetrate into the polymer matrix. A light-weight composite will be obtained after the polymers are melted and blended with the wood flour (Wolcott, 2010).

2.1.4.2 Production Method

As described briefly in the previous section, the most important step during the production process of WPC is the compounding process (also known as mixing or blending process). The purpose of this process is to disperse the wood flour into the melted thermoplastics. It becomes complicated due to the high physical energy (shear forces) required to melt the polymer. The blending process occurs in the blending equipment where the polymers are melted. Depending on the type of manufacturing process, there are two different kinds of blending equipment that one can use; they are: single screw extruder and twin screw extruder (refer to Figure 5 and 6 for detailed diagrams) (Wolcott, 2010).

Single screw extruder uses the screw to deliver the materials that are to be mixed. The screw itself is the parallel plates that wound about a shaft. The gap between the screw and the barrel is the area where the shearing forces are applied to the substance. The friction forces generated help the material to be pushed forward the barrel instead of rotating within the screw. Wolcott also noticed that the friction forces exerted are proportional to the screw diameter, angle, depth of the gap between screw and barrel and the rotation speed (Wolcott, 2010).

The twin screw extruder is normally used when single screw extruder could not achieve the desired degree of mixed products. In fact, the 'double screw' feature in the machine provides better mixing due to the intermeshing actions. Intermeshing action is defined as the displacement of mixed materials from one screw to another thus allowing better mixing. Twin screw extruder can further be divided into two categories depending on the direction of its rotation; namely: co-rotating screws and counter-rotating screws. While co-rotating screws offer great mixing and lower dwelling time, the counter-rotating screw can generate high pressure which could be crucial for certain processes (Wolcott, 2010). The compounding material, out of the extruders, can then either be shaped according to the needs or formed into pellets for further processing steps The extruder could produce finished or unfinished products in the rate of 3m/min (10ft/min) (Caulfield, 2005).

2.1.5 Product Design and Application of WPCs

One of the WPC characteristics is its flexibility. Due to the extrusion process, the compounding materials can be formed to almost any shape. Therefore, there is no certain standard of measurements for most of WPC applications. However, for the structural products such as wall profiles, there is a set of guideline of how thick the profiles should be; for exterior profiles, the normal thickness ranges between 4 – 6mm, whereas interior profiles will be about 2.5 – 3.5mm thick (Tangram, 2002). Some companies also combine the wood-filled thermoplastic with unfilled thermoplastic to gain specific attributes without changing the whole production process.

Decking industry is the largest consumer of WPC products. Their low maintenance, durability and mould resistance attributes are the main reasons that drag the homeowners away from traditional wooden decks and move to WPCs. Tangram Technology Ltd. stated that WPC products have been consistently in high demand for the last five years (since the year of 1997 considering the time the article was written) and they predicted that the trends will not change in the coming years. The growing market of WPC results in more products manufactured from it, especially those that are previously produced from wood products or plastics. Below are some applications of WPC in other products (Tangram, 2002):

- Door and window frames and components
- Exterior and interior wall profiles
- Docks and railings
- Stairs and hand rails
- Balustrade
- Floors
- Shelves
- Fence posts
- Garden furniture
- Office furniture
- Kitchen cabinets
- Sound proofing cladding

WPC's flexible design has become a very crucial factor in its fast growing market in North America and the world. In addition, its 1-step processing procedure and zero-waste process make it a very ideal solution to the production of other kinds of products such as door frames, rails and floors.

2.1.6 Marketability of WPCs

Currently, chemical treated lumbers are still inferior to WPC; about 80% of the building products are made of solid lumbers. However, based on its growth trends in the past, Caulfield predicted that the WPC market share will grow more rapidly in the future. This, in fact, had been proven by the increase of market share from 2% in 1997 to 8% in 2001. The increase in the market share was also helped by the excellent promotions of the benefits one can enjoy from WPCs (Caulfield, 2005). Previous section of this paper has mentioned that low maintenance cost, absence of chips and splinters and durability are few examples of WPCs advantages.

The advantages offered by WPCs also drive the customer to shift from previous products to WPC albeit the higher prices. Window application would be a perfect example in this case. Even though PVC window is cheaper than WPC window, customer would prefer the later due to better thermal stability, moisture resistance and stiffness. In places, such as European countries, where residential wood decking is not very common, WPCs are marketed in other types of products such as wooden profiles, door frames, furniture and automobile interior. In Japan, on the other hand, the future of WPC is very promising with the increasing demands on wooden decks, walls and floorings (Caulfield, 2005).

2.1.7 Properties of Wood- Plastic Composites

The quality of a product is generally determined by its properties and how well it performs under certain circumstances; good quality products must have excellent properties associated with them that differentiate them from the competitors. Depending on the type of the products, there are several different factors that will define the final properties.
In WPC, the final attributes will depend on (Rowell, 2006):

□ Volume fraction of wood

□ Processing temperature

□ Type of additives

□ Type of thermoplastic

While volume of wood will affect the mechanical properties of the final product, the processing temperature and the type of thermoplastic used will influence the adhesion between the substances. The additives, on the other hand, would add certain attributes depending on the needs.

2.1.7.1 Thermal Properties

Very little has been published about WPCs dependency on temperature while it is actually a significant characteristic which influences other properties such as mechanical properties. Schildmeyer conducted several tests where WPC panels were exposed to heat. Static tests were conducted at a temperature between 21°C (70°F) and 80°C (176°F). The goals of the experiment were to observe the changes in stress, strain and MOE of WPC under different temperatures and establish relationship between those properties and the temperatures. WPCs used during the experiment were composed of 58.8% 60-mesh pine flour, 33.8%

Polypropylene, 4% talc, 2.3% MAPP and 1% lubricant with respect to the final product mass (Schildmeyer, 2006).

Based upon the data obtained, Schildmeyer drew the following conclusions. The ultimate stress (σultimate) in tension and compression were both decreasing linearly with an increase in temperature. On the other hand, the maximum strain (εmax) in tension was slightly increasing when exposed to hotter environment; whereas the strain in compression remained relatively constant regardless the temperature. Furthermore, Schildmeyer also noticed that Modulus of Elasticity (E) was reduced in linear fashion in compression and in quadratic fashion in tension. WPCs have strong dependency on temperature since different thermal conditions may alter the mechanical properties of the material. Furthermore, the use of wood in the process could minimize the bad impacts of heat on thermoplastics since wood is thermally more stable than plastics (Schildmeyer, 2006).

2.1.7.2 Mechanical Properties

Wolcott et al conducted few experiments to determine the effect of wood fibres addition on the tensile strength and tensile modulus of WPCs. Three different types of composites were used in the experiments; they were: PP-wood fibre, HDPE-wood fibre and PS-wood fibre (Wolcott, 2010). The results of the experiments can be seen in Figures 2.1 and 2.2 below –

Figure 2.1: The Changes of WPCs Tensile Strength with the Addition of Wood Fibre Content

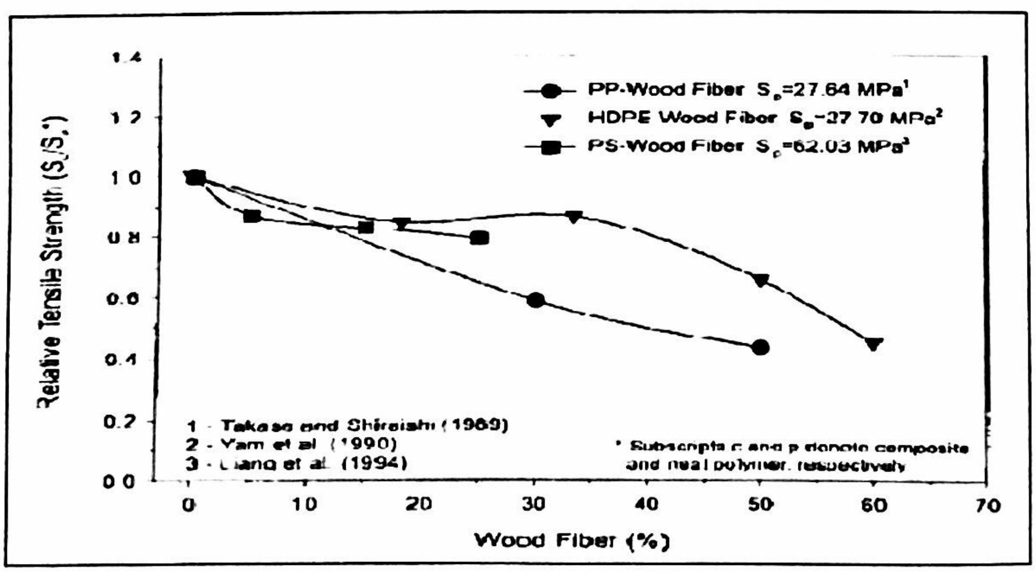
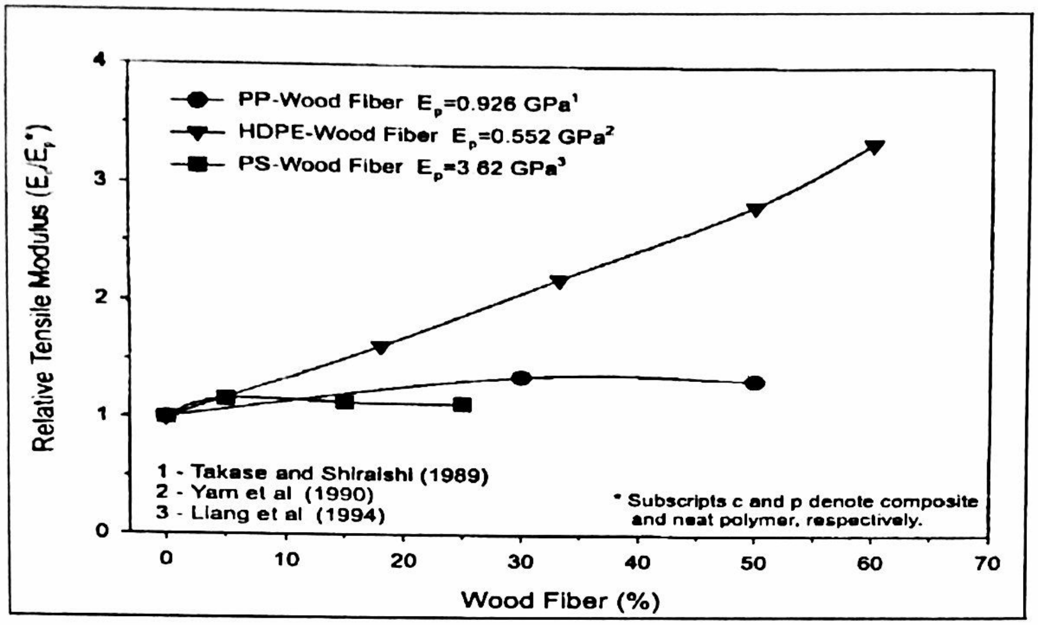


Figure 2.2: The Changes of WPCs Tensile Modulus with the Addition of Wood Fibre Content



In the diagrams above, tensile strengths of WPCs were decreasing with the addition of wood fibres. However, the tensile moduli were increasing. Figure 2.2 also shows that for PP- and PS-based composites, the tensile moduli reached their maximum values at certain points thus making further addition of wood ineffective. PP-based composites reached their maximum tensile modulus with 30% wood fibre content, while PS-based composites were optimal at 5% wood fibre content. Meanwhile, there seemed to be no maximum tensile modulus for HDPE-wood fibre matrix as the values kept rising with the addition of wood fibre (Wolcott, 2010).

Optimat Ltd and MERL Ltd also conducted similar experiments with further measurements on flexural strength and flexural modulus. Like tensile modulus, flexural strength of WPC would reach its maximum point before decreasing as wood content increased (Optimat, 2003).

2.1.8 Recycling in WPCs

It has been reported that majority of the Municipal Solid Waste (MSW) in the United States was waste wood, waste paper and waste plastics. In 1994, the amount of waste generated in the United States was 190 million tons and the number has kept increasing over the years (Winandy, 2004). These wastes would just be waste products if they were not used for other purposes. With the invention of Wood Plastic Composites whose main raw materials are those mentioned above, the wastes were able to re-enter the manufacturing process and be recycled to produce panels for construction purposes. This part of the thesis will discuss how WPC manufacturers obtain those raw materials.

2.1.8.1 Wood Based Material

As we have learned from the previous section, wood flour is the one of the main ingredients in the manufacturing process of WPCs. Usually; it comes from the post-industrial wood waste such as wood shavings, chips and sawdust. Other sources would be from the consumers; this includes wood pellets, building construction waste, cardboards and newspapers. Winandy also stated that recently wood pellet industry has been the major sources of wood flour with an estimated 44 percent of reclamation rate. Rice hulls could be an alternative to the wood flour. Nexwood has been reported to use about 9.1 million kg of rice hull in 2002 (Winandy, 2004).

2.1.8.2 Plastic Material

Similar to wood flour, the thermoplastics used in the production process are also recycled plastics. They typically come from recycled grocery bags and used pellet wrap. In 2004, the amount of thermoplastics used in the process of manufacturing WPCs was estimated to be 204 million kg; of which 95 percent of those amount were recycled products (Winandy, 2004).

2.1.9 Environmental Effect

The recycling activity also depends highly on how the WPCs are treated during their useful life. Most WPCs are used in the exterior application which in fact relates to degradation of the material performance and recycling potential. Winandy examined WPCs that had exhibited decay on their surface area and found that there existed mycelium cluster in the interface between the wood flour and thermoplastics the exterior; it would look like surface erosion. He also noted that WPC with higher wood content was more prone to this kind of decay compared to those which had less wood content (Winandy, 2004).

Another environmental issue would be UV-degradation. The surface of WPC would slowly degrade with continuous exposure to UV light. The oxygenated functional group in the thermoplastic could lead to further photooxidation when exposed to UV light. Moreover, Winandy also noted that photooxidation occurred more in the post-consumer polyolephins (the type of thermoplastic) than virgin polyolephins (Winandy, 2004).

2.2 Basic Information about Low Density Polyethylene (LDPE)

Plastics are synthetic substances produced by chemical reactions. They are polymers that consist of monomers linked together by chemical bonds. Among polymers, polyethylene is a thermoplastic polymer produced by monomers of ethylene. Polyethylene was discovered by British chemists in 1933. It contains the chemical elements carbon and hydrogen. Polyethylene is classified into several different categories such as HDPE, LDPE, LLDPE, etc. Low density polyethylene (LDPE) is a thermoplastic made from petroleum. LDPE materials are strong, light-weight and durable (Pramila & Ramesh, 2011).

Low density polyethylene (LDPE) is a polyethylene with a density < 940 $kg/m^3$and melting point 120°C, produced by a high pressure process, and is therefore often referred to as high pressure polyethylene (István, 2010). LDPE is chemically very similar to high density polyethylene (HDPE), but is more flexible and less dense. LDPE is slightly waxy and stretches well (ronz.org).

Because of its versatility (large range of density, molecular weight (MW) and MW distribution, and chemical inertness), LDPE remains a popular plastic in use today (Plastic Europe, 2011). Low density polyethylene (LDPE) represents the majority of thermoplastics currently used as food packaging materials. Since the production and consumption of these polymers is incessantly increasing (Tajeddin, 2009). They are widely used for manufacturing various containers, dispensing bottles, plastic bags and various molded laboratory wares (Pramila & Ramesh, 2011). Low density polyethylene (LDPE) is by far the most common polymer film material (Cleverley, 1979).

Properties of LDPE:

- Light-weight
- Good impact resistance
- Extremely flexible
- Easily cleaned
- Thermoforming performance
- Meets food handling guidelines

- No moisture absorption
- Chemical- and corrosion-resistant

Table 2.1: Properties of Low Density Polyethylene

Property	Test Method	Units	LDPE
Physical			
Density	ASTM D-792	lbs/ft^3	57.39
Water Absorption	ASTM D-570	%	slight
Mechanical			
Yield Point	ASTM D-638	psi	1,363
Tensile Break	ASTM D-638	psi	1,943
Elongation at Break	ASTM D-638	%	515
Tensile Modulus	ASTM D-638	psi	41,615
Flexural Modulus	ASTM D-790	psi	28,565
Flexural Strength	ASTM D-790	psi	1,175
Izod Impact	ASTM D-4020	ft-lbs/in	No Break
Tensile Impact	DIN 53448	ft-lbs/in^2	401
Hardness	ASTM D-2240	Shore D	55
Thermal			
Melt Point	ASTM D-3417	•F	230
Heat Deflection	ASTM D-668		
264 psi		•F	98
66 psi		•F	110
Electrical			
Volume Resistivity	ASTM D-257	ohm-cm	>10^15
Surface Resistivity	ASTM D-257	ohm/square	>10^15

(Source: LDPE, Plastic International)

2.3. LDPE - Wood Composites

In 1500BC, the natural fibres were used for reinforcement (Chawla 1987). To produce bricks with improved mechanical properties the ancient Egyptians used straw as reinforcements in bricks (Smith and Li, 2000). In the 20th century, increasingly composites became an attractive material type (Hancox 2001). The specific advantages offered by natural fibres like, low cost, low density, high toughness, acceptable specific strength, enhanced energy recovery, recyclability, etc, (Chauhan et al., 2000; Debapriya 2004; Jacob et al., 2006). Polymer composites were used to manufacture by thermosetting for about three decades (Hancox 2001). The products are non-recyclable, non-reparable, and cannot be re-formed, reshaped, or remoulded. Then Thermoplastic polymers have drawn attention due to their advantages over thermosets.

Now a days, there creates a widespread concern of using non-biodegradable plastics. They are a harmful to the environment as they can not be recycled, and cause potential harm by the release of CO2. Wood like other natural fiber sources is readily available and biodegradable, hence, it can reduce the non-biodegradable fraction in the composites and this has beneficial consequences on the environment.

Sisal, Jute, flax, hemp, henequen, and others have been used to make composites (Fung 2003a; Keller 2003; Garkhail et al. 2000; Herrar-Franco et al. 1997). In a study it has been found that, the mechanical properties are enhanced at fibre loading of 10 - 30%, and then starts deteriorating afterwards in jute fibre reinforced HDPE composites (Mohanty et al. 2006).

In another study, it has been found that in untreated, alkaline, and silane treated flax fibre when used to reinforce HDPE the mechanical properties improved with increasing fibre content. The silane treated flax fibres showing the best mechanical properties. This is result of better interfacial adhesion (Anup 2008). It has been reported that flax reinforced composites have not significantly increased the tensile strength of composites because of poor adhesion between fibre and matrix (Oksman et al. 2003; Santos and Pezzin 2003). Improved mechanical properties of fibre reinforced composites have been alluded to improved bonding between the fibre and the matrix (Mohanty et al. 2001; Bledzki and Gassan 1999).

Due to its melting temperature PE remains useful as composite matrix. Thus, natural fibres which degrade at a higher temperature could be used. For polymers with very high melting point, the fibre can experience degradation during compounding. This may adversely affect the mechanical properties of the fibre, and also the composite (Van de Velde and Kiekens 2001).

Flammability characteristic of polymer application has been attracted considerable attention due to the wide range. The combustion of polymers follows a cyclic pattern of preheating, decomposition, ignition, and combustion. In the presence of a flame source, the polymer becomes preheated. It then loses structural integrity and decomposes releasing combustible hydrocarbon gases into the atmosphere. The gases at the appropriate temperature ignite, and then combustion takes place releasing heat. The heat continues the cycle: preheating, decomposing, igniting, and finally combusting the polymer. Since the whole process is time dependent, the flammability properties of the polymer can therefore be expressed as a function of time. The presence of wood in a polymer composite can reduce the flammability property of the polymer because the amount of available hydrocarbon is reduced. Flammability properties of wood polymer composites have received limited attention (Stark et al. 2010).

However studies on wood particleboard composites based on low density polyethylene (LDPE) are very limited (Yamashita et al., 1999). In a study, it has been observed that in wood flour- low density polyethylene board the highest modulus of elasticity was obtained from board produced at 180°C, 50/50 wood particles/LDPE. The higher modulus of rupture of 20.31 N/mm2 and MOE of 1363 N/mm2 was obtained from board produced 140°C, 60/40 wt% wood particles/RLDPE boards.

In another study, Kola Nitida wood fibres were used to reinforce Low Density Polyethylene and improved mechanical and flame retardancy properties were obtained. LDPE/KN wood fibre composite showed improved tensile properties. Yield strength, yield strain, and Young's Modulus increased from 0 – 20 wt%. Results also show values increasing with increasing fibre loading while hardness values decreased with increasing fibre loading. Flammability retardancy tests show that flame resistance of LDPE/KN composites was increased by the presence of Kola Nitida wood fibres.

In a research work focuses on the effect *Alstonia boonei* would have on both the mechanical and flame retardancy properties of Low Density Polyethylene (LDPE). Hence, the mechanical and flammability properties of LDPE and various African plants remain open to research. Reinforcing LDPE with AB wood fibres improves both mechanical and flammability properties
of the composite. Tensile propertiessuch as yield strength, Young's Modulus and yield strain increased from 0 – 20 of wt%. However, the value of hardness decreased with increasing fibre content. The general improvement in the mechanical properties of the composite could mean proper compatibility of *Alstonia boonei* wood fibres and LDPE matrix.

2.4 Response Surface Methodology

As an important subject in the statistical design of experiments, the *Response Surface Methodology* (*RSM*) is a collection of mathematical and statistical techniques useful for the

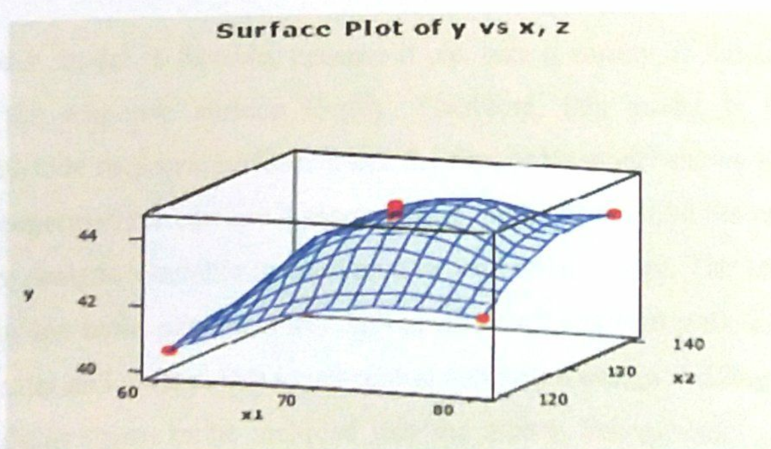
modeling and analysis of problems in which a response of interest is influenced by several variables and the objective is to optimize this response (Montgomery 2005).
It can be expressed as

$$y = f(x1, x2) + e$$

The variables x_1 and x_2 are independent variables where the response y depends on them. The dependent variable y is a function of x_1, x_2, and the experimental error term, denoted as e. Response Surface Methods are designs and models for working with continuous treatments when finding the optima or describing the response is the goal (Oehlert 2000).

The first goal for Response Surface Method is to find the optimum response. When there is more than one response then it is important to find the compromise optimum that does not optimize only one response (Oehlert 2000). When there are constraints on the design data, then the experimental design has to meet requirements of the constraints. The second goal is to understand how the response changes in a given direction by adjusting the design variables. In general, the response surface can be visualized graphically. The graph is helpful to see the shape of a response surface; hills, valleys, and ridge lines. Hence, the function $f(x_1, x_2)$ can be plotted versus the levels of x_1 and x_2 as shown as Figure 2.3.

Figure 2.3: 3d Graph of Response Surface Methodology



In order to understand the surface of a response, graphs are helpful tools. But, when there are more than two independent variables, graphs are difficult or almost impossible to use to illustrate the response surface, since it is beyond 3-dimension. For this reason, response surface models are essential for analyzing the unknown function.

The relationship between the response variable y and independent variables is usually unknown. In general, the low-order polynomial model is used to describe the response surface f. A polynomial model is usually a sufficient approximation in a small region of the response surface. Therefore, depend ing on the approximation of unknown function f, either first-order or second-order models are employed. the approximated function f is a first-order model when the response is a linear function of independent variables. A first-order model with N experimental runs carrying out on q design variables and a single response y can be expressed as follows:

$$y_i = \beta_0 + \beta_1 x_{i1} + \beta_2 x_{i2} + \ldots\ldots\ldots + \beta_q x_{iq} + \varepsilon_i \qquad (i = 1, 2, \ldots\ldots, N)$$

The response y is a function of the design variables $x_1, x_2, \ldots, x_q$, denoted as f, plus the experimental error. A first-order model is a *multiple-regression* model and the b_j 's are regression coefficients.

When there is a curvature in the response surface the first-order model is insufficient. A second-order model is useful in approximating a portion of the true response surface with parabolic curvature. The second-order model includes all the terms in the first-order model, plus all quadratic terms like

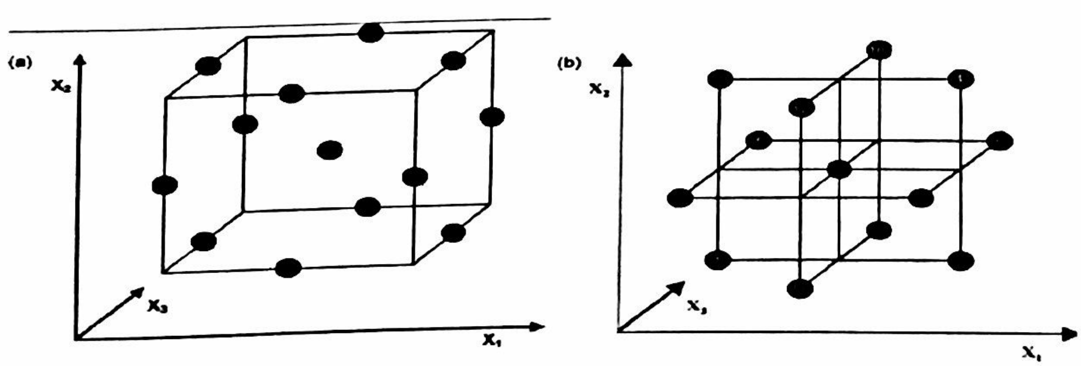
The second-order model is flexible, because it can take a variety of functional forms and approximates the response surface locally. Therefore, this model is usually a good estimation of the true response surface. When the first-order model shows a significant lack of fit, then an experimenter can use a second-order model to describe the response surface. There are many designs available to conduct a second-order design. The central composite design is one of the most popular ones. An experimenter can start with $2q$ factorial point, and then add center and axial points to get central composite design. Adding the axial points will allow quadratic terms to be included into the model. Second-order model describes quadratic surfaces, and this kind of surface can take many shapes. Therefore, response surface can represent maximum, minimum, ridge or saddle point. Contour plot is a helpful visualization of the surface when the factors are no more than three. When there are more than three design variables, it is almost impossible to visualize the surface. For that reason, in order to locate the optimum value, one can find the stationary point. Once the stationary point is located, either an experimenter can draw a conclusion about the result or continue in further studying of the surface.

2.5 Box Behnken Design

The Box-Behnken is a good design for response surface methodology because it permits: (i) estimation of the parameters of the quadratic model; (ii) building of sequential designs; (iii) detection of lack of fit of the model; and (iv) use of blocks. A comparison between the Box-Behnken design and other response surface designs (central composite, Doehlert matrix and three-level full factorial design) has demonstrated that the Box-Behnken design and Doehlert matrix are slightly more efficient than the central composite design but much more efficient than the three-level full factorial designs.

Box and Behnken suggested how to select points from the three-level factorial arrangement, which allows the efficient estimation of the first- and second order coefficients of the mathematical model. Box-Behnken designs (BBD) are a class of rotatable or nearly rotatable second-order designs based on three-level incomplete factorial designs. For three factors its graphical representation can be seen in two forms (a) A cube that consists of the central point and the middle points of the edges, as can be and (b) the cube for BBD and three interlocking 2^2 factorial designs.

Figure 2.4: (a) The Cube for BBD (b) Three Interlocking 2^2 Factorial Designs



The number of experiments (N) required for the development of BBD is defined as $N=2k$ $(k-1)$ factors and C_0 is the number of central points). For comparison, the number of experiments for a central composite design is $N=2k+2k+C_0$.

A comparison between the BBD and other response surface designs (central composite, Doehlert matrix and three-level full factorial design) has demonstrated that the BBD and Doehlert matrix are slightly more efficient than the central composite design but much more efficient than the three-level full factorial designs where the efficiency of one experimental design is defined as the number of coefficients in the estimated model divided by the number of experiments.

Another advantage of the BBD is that it does not contain combinations for which all factors are simultaneously at their highest or lowest levels. So these designs are useful in avoiding experiments performed under extreme conditions, for which unsatisfactory results might occur. Conversely, they are not indicated for situations in which we would like to know the responses at the extremes, that is, at the vertices of the cube. BBD for four and five factors can be arranged in orthogonal blocks. Because of block orthogonality, the second-order model can be augmented to include block effects without affecting the parameter estimates, that is, the effects themselves are orthogonal to the block effects. This orthogonal blocking is a desirable property when the experiments have to be arranged in blocks and the block effects are likely to be large.

2.6 Derringer's Desirability Function

Desirability function is a popular and established technique for the simultaneous determination of optimum settings of input variables that can determine optimum performance levels for one or more responses. Recently, several articles are reported in the application of desirability function various fields of biological science. Harrington (1965) for specifying the relationship between predicted responses on a dependent variable and the desirability of the responses. The desirability procedure involves two steps: (1) finding the levels of the independent variables that simultaneously produce the most desirable predicted responses on the dependent variables and (2) maximize the overall desirability with respect to the controllable factors. Therefore, the desirability functions are used in order to obtain qualitative and quantitative responses by the simple and quick transformation of different responses to one measurement. The general approach of the desirability function is to first the response into an individual desirability function (d_i) that varies from 0 to 1 (lowest desirability to highest desirability). The individual desirability scores for the predicted values for each dependent variable are then combined into overall desirability function D, by6 computing their mean of different (d_i) values.

$$D = (d1 \times d2 \times d3 \times \ldots\ldots \times d_n)^{1/n} = (\prod_{i=1}^{n} di)^{1/n}$$

Where d_i indicates the desirability of the response and n is the number of responses in the measure. If any of the responses beyond the desirability, then overall function turned into zero.

It can be extended to

$$D = [d_1^{v1} \times d_2^{v2} \times \ldots\ldots\ldots \times d_n^{1/n}]^{1/n}$$

$$0 \leq v_i \leq 1 \ (I = 1, 2, \ldots\ldots, n), \ \sum_{i=1}^{n} vi = 1$$

Where (d_i) indicate the desirability of the response y_i (i=1, 2, 3,……, n) and vi represents the importance of responses. So, maximum over all desirability function D depends on the v_i (importance) value.

For simultaneous optimization each response must have a low and high value assigned to each goal. The meanings of the goal parameters are:

Maximum:

$d_i = 0$ if response < low value.

$0 \leq d_i \leq 1$ as response varies from low to high.

di = 1 if response > high value

Minimum:

$d_i = 1$ if response < low value.

$1 \leq d_i \leq 0$ as response varies form low to high.

$d_i = 0$ if response > high value.

Target:

$d_i = 1$ if response < low value.

$0 \leq d_i \leq 1$ as response varies from low to target.

$1 \geq d_i \geq 0$ as response varies from target to high.

$d_i = 0$ if response > high value.

Range:

$d_i = 0$ if response < low value.

$d_i = 1$ if response varies from low to high.

$d_i = 0$ if response > high value.

The d_i for "in range" are included in the product of he desirability function "D", but are not counted in determining "n":

$$\mathbf{D = (\pi\ d_i)^{1/n}}$$

If the goal is none, the response will not be used for the optimization.

Chapter Three: Materials and Methods

Chapter Three: Materials and Methods

3.1 Materials and Equipments

3.1.1 Collection of Raw Materials

Saw dust of Mahagony (*Swietenia mahagoni*) was collected from the local sawmill of Khulna, Bangladesh and LDPE (Low density polyethylene) was collected from the local market of Dhaka, Bangladesh. Boric acid and Borax which are used as fire retardants in the manufacturing process is also obtained from the local market of Khulna, Bangladesh.

3.1.2 Chipper

A Chipper machine of Forestry and Wood Technology laboratory was used to make powder form of the pallets of the LDPE by inputting the materials five to six times into the chipper. The rpm of the Chipper motor was 1420.

3.1.3 Mesh

The Wood flour and plastic powder are screened through a 35 mesh (500 micron or .5mm).

3.1.4 Digital Balance

A digital balance was used to measure the weight of all the raw materials, i.e. (plastic and wood flour) as well as the board samples.

3.1.5 Oven

A Ventilated Oven (Electrically heated Thermoplastic blast dry box, Model: DGH-910-ISA, Serial: 5054, SAFNA, Germany) was used to determine and control the moisture content (%) of the raw materials. The indicator outside the oven indicated the inside temperature.

3.1.6 Hot Press

A multi- daylight hot press is used to manufacture of the boards which is digital and electrically heated.

3.1.7 Slide Caliper

An Electronic Digital Caliper (Accuracy: ± 0.02mm/0.001IN; < 100mm and ± 0.03m/ 0.001N;. 100-200 mm; 5°-40°C, UK) was used to measure the thickness of the board and test sample.

3.1.8 Universal Testing Machine (UTM)

An Analogue Hydraulic Universal Testing Machine (UTM) (model: UTN-100, Serial-11/98-2443, maximum capacity- 100000 kgf, Fuel Instrument and Engineers Pvt. Ltd. Yadrav-416145, Maharastra, India) of Khulna University of Engineering and Technology (KUET) was used to determine the mechanical properties of the boards.

3.2 Manufacturing of Composite Boards

3.2.1 Manufacturing Process

The manufacturing procedure of the board is given under this section.

3.2.1.1 Experimental Design

Three levels of Wood flour and to LDPE mixing ratios (70:30, 60:40, 50:50) and fire retardants (Boric acid +Borax) were used as 0%, 3% and 6% of the total weight of raw materials to manufacture composite boards. Also three level of pressing time are used (7, 9 & 11 min) in the manufacturing process of boards in the Wood Technology laboratory of Forestry and Wood Technology Discipline, Khulna University. The Temperature and pressure were fixed into 150°c and 4Mpa respectively. The experimental design variables are designed by using Box- Behnken design of RSM (Response Surface Method).

Box-Behnken design allows establishment a statistical relationship between experimental variables and responses. Table 3.1 shows three levels (low, medium and high) of three factors (mixing ratio, pressing time and fire retardant %). The Box- Behnken design works with only three levels coded -1, 0, +1 for low, medium and high values respectively of each factors. Box- Behnken design avoids extreme treatment condition (all high values). The applied Box- Behnken was consisted of 15 experiments which are shown in table 3.2

Table 3.1 Factors with Range, Level and Level of Manufacturing of Composite Boards

Factors	Range and Level		
	Low(-1)	Medium(0)	High(+1)
Mixing ratio	1 (70:30)	2 (60:40)	3 (50:50)
Pressing time (min)	7	9	11
Fire Retardant (%)	0	3	6

Table 3.2 Variables Studied and Design Used in the Manufacture of the Composite Board

Run	Mixing ratio	Pressing Time (min)	Fire Retardant (%)
1	1.00 (70:30)	9	6.00
2	2.00 (60:40)	9	3.00
3	1.00 (70:30)	9	0.00
4	2.00 (60:40)	9	3.00
5	1.00 (70:30)	11	3.00
6	2.00 (60:40)	9	3.00
7	2.00 (60:40)	11	6.00
8	2.00 (60:40)	11	0.00
9	3.00 (50:50)	11	3.00
10	2.00 (60:40)	7	6.00
11	3.00 (50:50)	9	6.00
12	3.00 (50:50)	7	3.00
13	1.00 (70:30)	7	3.00
14	3.00 (50:50)	9	0.00
15	2.00 (60:40)	7	0.00

3.2.1.2 Preparation of Raw Materials

The saw dust collected from saw mills of Mahagony Species was screened by the mesh. The LDPE was reduced its size to powder form by using grinder machine. The boric Acid and Borax were also reduced to powder form by grinder.

3.2.1.3 Drying of Processed Raw Materials

After processing the saw dust was kept in an electrically heated lab scale oven at 103±2 ºC to dry that ad remove the moisture content properly.

3.2.1.4 Mat Formation

The mats used for this study were formed manually after mixing and blending all the dried materials in appropriate proportions on an aluminum plate. The dimension of the boards is 20 cm × 19 cm. The average mat thickness of each type of board was six times of the targeted board thickness (6 mm).

3.2.1.5 Hot Pressing

After Mat formation, a steel sheet was placed on the mat. At the same time the hot press was switched on to raised the temperature. When the temperature was raised to the required temperature then the mat was pressed by the automated multi daylight hydraulic compression hot press. After inserting the mat in to the hot press, the pressure was raised up to 4 MPa. Then each board was pressed by maintaining the temperature and pressing time according to the research design. Each type of board was retained another 10 min under pressure.

3.2.2 Finishing of Composite Boards

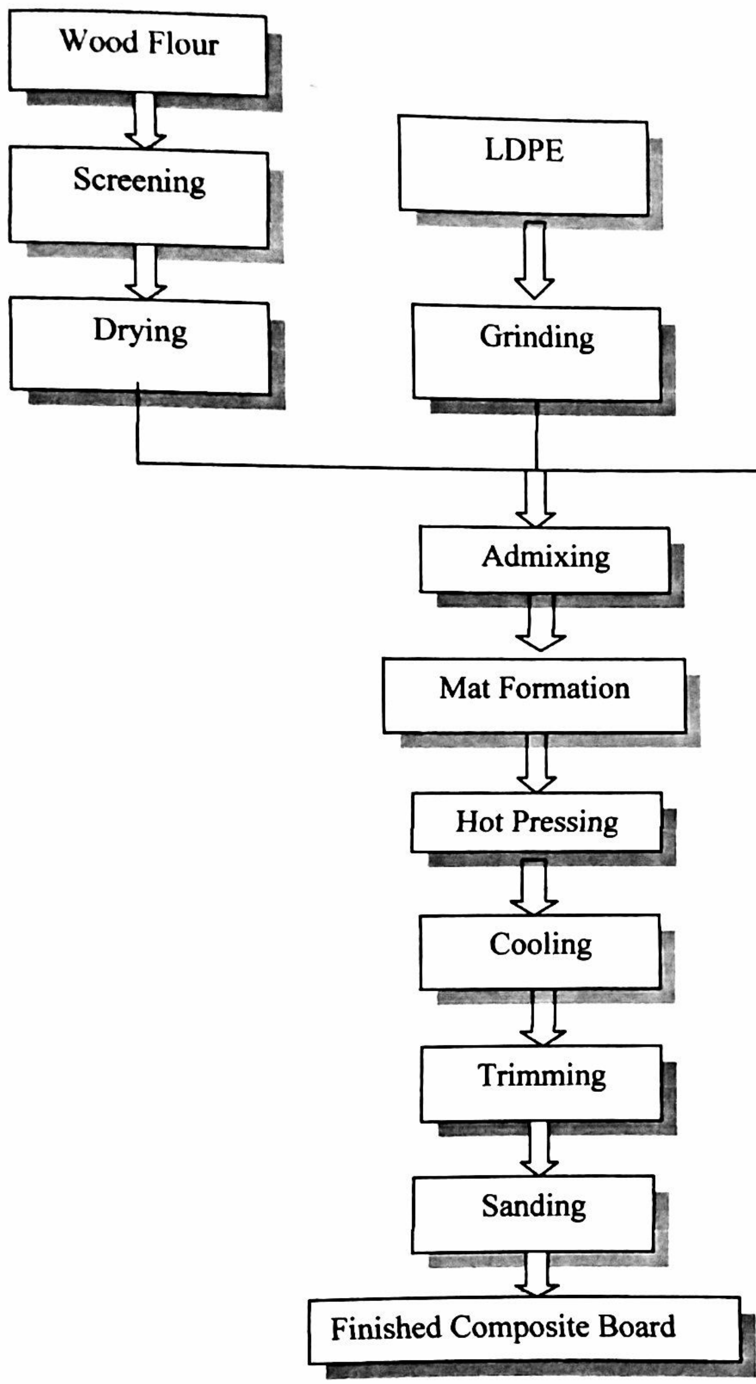
3.2.2.1 Trimming

After producing each type of board separately, these were trimmed at edges with the one-man handsaw. The boards were trimmed to 18cm×20 cm dimension.

3.2.2.2 Sanding

The trimmed boards were sanded with 80 grade sand paper with rough sanding machine.

Chart 3.1: Flow Diagram of composite board manufacturing process



3.3 Laboratory Test

Physical and mechanical properties were tested in the Laboratory of Forestry and Wood technology Discipline, Khulna University, Khulna and Department of civil Engineering, Khulna University of Engineering and Technology, Khulna. The Properties were tested according to the procedures defined in the American Society for Testing of Materials (ASTM) standard.

3.3.1 Preparation of Test Sample

For the test of the mechanical properties the samples are prepared into 20 cm × 5 cm and for the physical properties test the samples are prepared to 5 cm× 5 cm dimention.

3.3.2 Mechanical Properties testing

3.3.2.1 Modulus of Rupture (MOE)

The modulus of elasticity (MOE) was calculated from the following equation-

$$MOE = \frac{P'L^3}{4\Delta bd^3}$$ .... Equation 1. (Desch and Dinwoodie, 1996)

Where,

MOE is the modulus of elasticity in N/mm^2

P' is the load in N at the limit of proportionality

L is the span length in mm

Δ is the deflection in mm at the limit of proportionality

b is the width of sample in mm

d is the thickness/depth of sample in mm

3.3.2.2 Modulus of Elasticity (MOR)

The MOR was calculated from the following equation-

$$MOR = \frac{3PL}{2bd^2}$$ .... Equation 2. (Desch and Dinwoodie, 1996)

Where,

MOR is the modulus of rupture in N/mm^2

P= Load in N

L= Span length in mm

b= width of test sample in mm

d= Thickness of test sample in mm

3.3.3. Physical Properties Testing

3.3.3.1 Density

Densities of treated and untreated specimens were determined on the oven-dry (103 ± 2° C) basis using the following equation with in the Wood Technology Laboratory of FWT Discipline of Khulna University, Khulna.

The formula is given below-

$$\rho = \frac{m}{v}$$ .... Equation 3. (Desch and Dinwoodie, 1996)

Where, ρ = Density in gm/cm^3

m = Mass of the sample in gm and

v = Volume in cm^3.

3.3.3.2 Moisture Content

The moisture content was determined, from the differences in weights before and after the sample has been drying in the oven for 24 hour in 102±3°C. Initial and final constant weight of the samples was measured by electric balance. It was calculated by the following formula-

$$MC\ (\%) = \frac{m_{\text{int}} - m_{od}}{m_{od}} \times 100$$ .... Equation 4. (Desch and Dinwoodie, 1996).

Where,

MC = Moisture content (%)

m_{int} = Initial mass of the sample (gm)

m_{0d} = Oven-dry mass of the sample (gm)

3.3.3.3 Water Absorption

Water absorption is defined as the difference in weight before and after immersion in water for 24 hour and expressed in percentage. The water absorption was calculated by the following formula-

$$A_w = \frac{m_2 - m_1}{m_1} \times 100$$ .... Equation 5. (Youngquist *et. al.*, 1997).

Where,

A_w = Water absorption (%)

m_2 = The weight of the sample after (24 hr.) immersion in water (gm)

m_1 = The weight of the sample before immersion in water (gm)

3.3.3.4 Thickness Swelling

Thickness Swelling is defined as the difference in weight before and after immersion in water for 24 hour and expressed in percentage. Thickness swelling was calculated by the following formula-

$$G_t = \frac{t_2 - t_1}{t_1} \times 100$$ .... Equation 6. (Youngquist *et. al.*, 1997).

Where,

G_t = Thickness swelling (%)

t_2 = Thickness of sample after immersion (24 hr.) in water (mm)

t_1 = Thickness of sample before immersion in water (mm).

3.3.3.5 Linear Expansion

Linear Expansion was measured by the digital calipers from the difference in length of the sample before and after 24 hours immersion in water.

It was calculated by the following formula-

Linear Expansion (%) = $\frac{l_2 - l_1}{l_1} \times 100$ .....Equation 7. (Youngquist *et. al.*,1997).

Where,

l_1 = length before immersion in water in mm

l_2 = length after immersion in water in mm

3.4 Analysis of Data

Statistical software STATISTICA was used for the design of the experiment. A second order polynomial model is used to fit the response to the independent variables is shown below-

$$Y=\beta_0 + \sum \beta_1 X_1 + \sum \beta_{11} X_1^2 + \sum \beta_{12} X_1 X_2 \text{ .... Equation 8.}$$

Where Y is the response (MOR), β_0 is the intercept and β_1, β_{11}, β_{12} are the co- efficient of parameters for linear, squared and interaction effects respectively.

Chapter Four: Results and Discussions

Chapter Four: Results and Discussions

4.1 Mechaical Properties

The processing variables of the experiment (Mixing ratio, pressing time and fire retardant percentages) have significant effect on the mechanical properties of boards. In this study the interaction effects of different factors on bending properties of board particularly on modulous of ruture (MOR) and modulus of elasticity (MOE) were executed by using the Box- Behnken design.

4.1.1 Modulus of Rupture (MOR)

4.1.1.1 Adequacy of the Model for MOR

The sufficiency of the model was evaluated through Analysis of varience (ANOVA) and observed vs. predicted plot for MOR. The adequacy of the model was evaluated through ANOVA (Table 4.1)

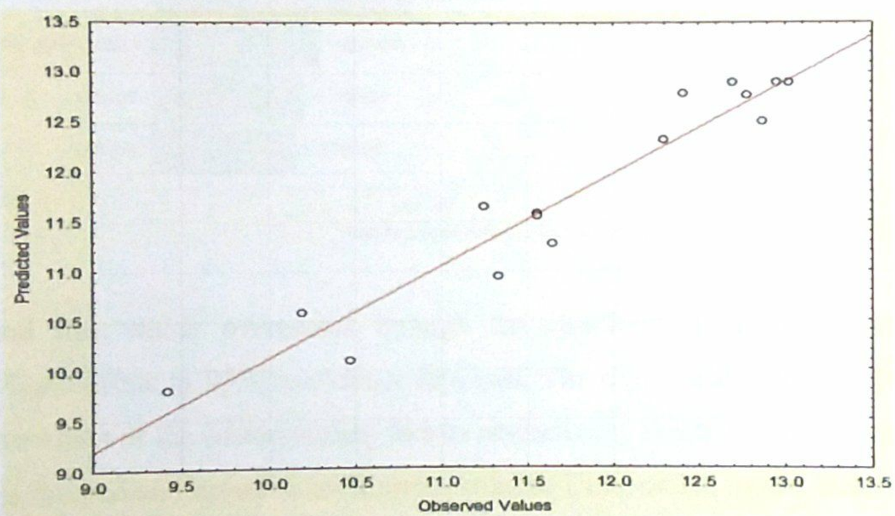
Table 4.1 ANOVA for Response Surface Quadratic Model for MOR

Source	Sum of Squares	df	Mean Square	F- Value	Prob > F	Remarks
(1)Mixing Ratio(L)	1.91101	1	1.911013	67.5269	0.014488	Significant
Mixing Ratio(Q)	5.19577	1	5.195775	183.5963	0.005403	Significant
(2)Pressing time(L)	0.48511	1	0.485113	17.1418	0.053683	
Pressing time(Q)	5.96705	1	5.967052	210.8499	0.004709	Significant
(3)Fire retardant(L)	0.09245	1	0.092450	3.2668	0.212433	
Fire retardant(Q)	0.44267	1	0.442667	15.6420	0.058388	
1L by 2L	0.04202	1	0.042025	1.4850	0.347230	
1L by 3L	0.05760	1	0.057600	2.0353	0.289804	
2L by 3L	1.82250	1	1.822500	64.3993	0.015176	Significant
Lack of Fit	1.16408	3	0.388025	13.7111	0.068739	**Insignificant**
Pure Error	0.05660	2	0.028300			
Total SS	17.01536	14				
R^2=0.9282; Adjusted R^2=0.7991; L indicated linear effect; Q indicated quadratic effect						

In ANOVA for MOR lack of fit (LOF) test was conducted. The lack of fit (LOF) is the variation of the data around the fitted model. LOF is a Special test of adequacy of data to fit with the model, because of the additional higher – order terms are removed from the error. Three center points are used for calculating the error. If the model does not fit the data well, this will be significant.

In this Study with regards to MOR, The LOF is not Significant relative to the poor error, which indicating good response to the model. The model regression co- efficient of determination of (R^2) of 0.9282 for MOR is reasonable agreement with the experimental results, indicating 92.82% of the variability can be revealed by the model and are left with 7.18% residual variability for MOR.

Figure 4.1: Observed Vs. Predicted Plot for MOR



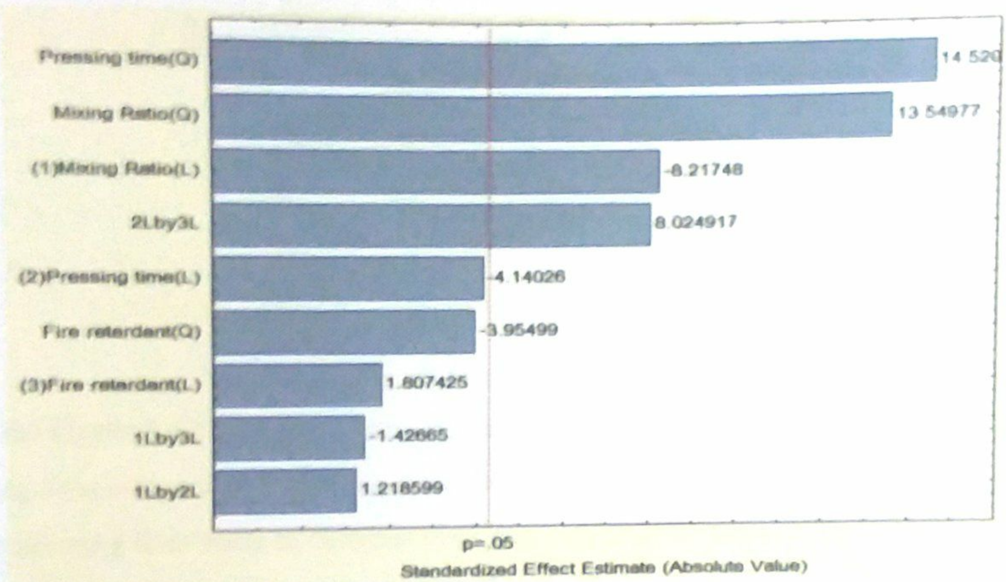
In addition to LOF test, the model was again evaluated by the observed vs. predicted plot (Fig: 4.2). The points of all predicted and actual responses fell in 45° lines indicating also good response to the model. From the above Statistical results it may be inferred that the Box–Behnken design was adequate to predict the MOR of board within the range of variables studied. The final predicted process model in terms of actual significant factors for MOR equation:

MOR= -11.5534 + 3.9150* mixing ratio – 1.1863* mixing ratio' +5.1575*pressing time - 0.3178*pressing time^2 -1.1275*fire retardant -1.47*fire retardant' +0.1125* pressing time * fire retardant.

4.1.1.2 Effects of Different Factors on MOR

The actual factors which have significant effect on MOR of boards are shown before in ANOVA table 4.1 and also represented by standardized Pareto chat developed by the software shown in figure 4.3.

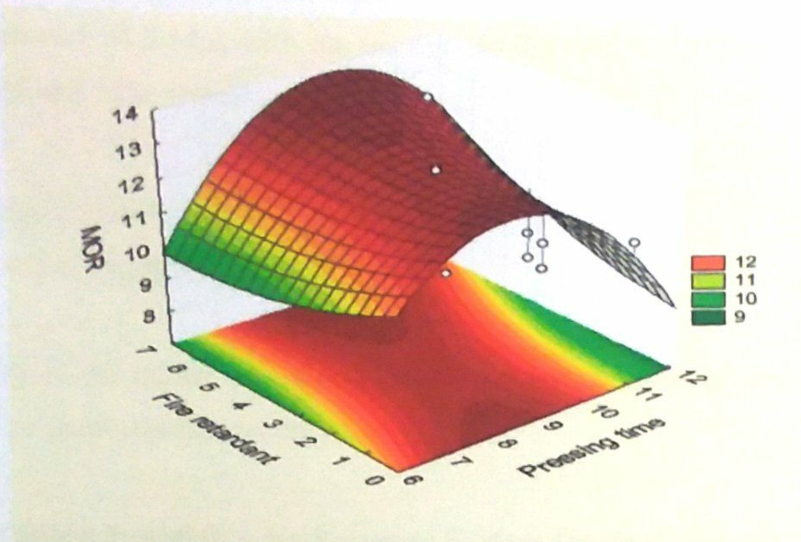
Figure 4.2: Standardized Pareto Chart for MOR



The vertical line which overpasses through the standardized factors determines the statistical Significance at 95% confidence intervals. The sign + and – reflects the positive and negative effect of the corresponding factors respectively. Positive co- efficient indicates the MOR is favored and negative coefficients indicate Unfavorable by the factors and their interactions.

It has been observed from ANOVA and Standardized Pareto Chart for MOR of board that the quadratic effect of pressing time and quadratic effect of mixing ratio has positive significant effect on MOR and linear effect of mixing ratio have an negative significant effect on MOR. It has been also observed that, the interaction between linear effect of pressing time and fire retardant (%) on MOR has positive significant effect on MOR. The positive interaction effect of Pressing time and fire retardant (%) on MOR is presented in the figure 4.4.

Figure 4.3: 3d Graph Showing the Effect of Pressing Time and Fire Retardant % on MOR



From the Figure 4.4 it has been observed that MOR attained the highest value 12 N/mm²for pressing time ranges 8 to 9 minutes and a fire retardant (%) ranges from 2 to 3 percentages. As the pressing time starts to decrease from 8 to 6 minutes or increase from 9 to 12 minutes and as the fire retardant % starts to starts to decrease below 2 percent and increase from 3 to 7 percentages the MOR starts to decreases sharply.

The MOR ranged from 16.25 to 20.31 N/mm^2.However boards produced at 140°C, pressure 4 MPa, pressing time 10 minute and at 60/40 wood particles/LDPE have higher MOR of 20.31 N/mm^2 (Atuanya *et.al*, 2011). In anther study we have found that in preparation of board with LDPE the mechanical properties of board (tensile properties) increased with increasing wood fibre content from 0-20 wt%. Young's Modulus and yield strength values showed a rather progressive increase after initial decrease of wood fiber content percentage (Obidiegwu and Nwosu, 2012).

In a study it has been found that, when the amount of coupling agent 0%, hot pressing time 10min, hot pressing temperature 180°C, hot pressing pressure 3.0 MPa in WPC production with low density the resultant WPC had a modulus of ruputre (MOR) value of 21MPa (Zhang etal., 2013).

In another study where fire retardants are used it was found that The MOR of the uncoupled (without Polypropylene) specimens decreased with increasing the FR content The uncoupled control specimens had the highest MOR, Here we have found 68.8 MPa, 64.2 MPa, 62.5 Mpa and 58.5 Mpa with the increase in fire retardant (Borax and Boric acid) control, 4%, 8% and 12%. (Nadir *et.al,* 2012).

4.1.2 Modulus of Elasticity (MOE)

4.1.2.1 Adequacy of the Model for MOE

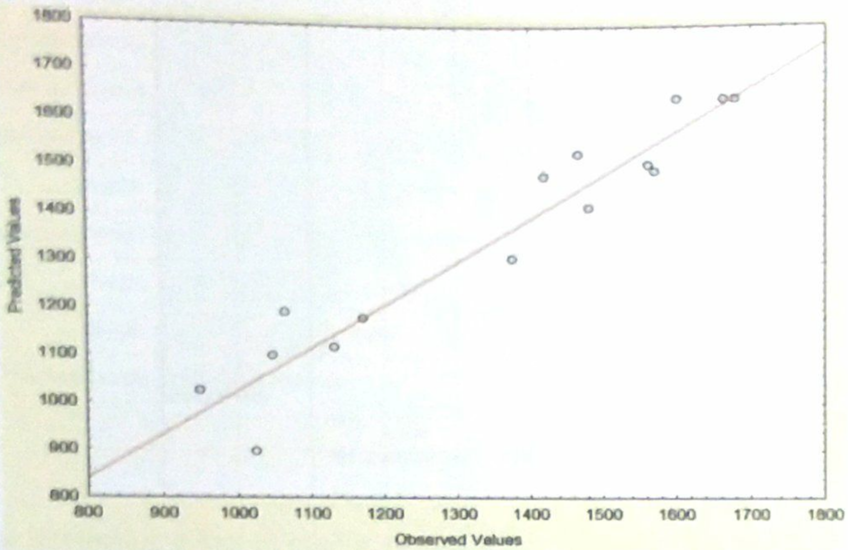
The sufficiency of the model was also evaluated through Analysis of varience (ANOVA) and observed vs. predicted plot for MOE.

Table 4.2: ANOVA for Response Surface Quadratic Model for MOE

Source	Sum of Squares	df	Mean Square	F- Value	Prob > F	Remarks
(1)Mixing Ratio(L)	63674.5	1	63674.5	37.3545	0.025741	Significant
Mixing Ratio(Q)	263082.9	1	263082.9	154.3370	0.006417	Significant
(2)Pressing time(L)	27599.6	1	27599.6	16.1912	0.056572	
Pressing time(Q)	296011.1	1	296011.1	173.6543	0.005709	Significant
(3)Fire retardant(L)	169836.7	1	169836.7	99.6343	0.009888	Significant
Fire retardant(Q)	653.4	1	653.4	0.3833	0.598966	
1L by 2L	11275.3	1	11275.3	6.6146	0.123737	
1L by 3L	45746.8	1	45746.8	26.8373	0.035300	Significant
2L by 3L	8703.0	1	8703.0	5.1056	0.152337	
Lack of Fit	66764.9	3	22255.0	13.0558	0.071981	**Insignificant**
Pure Error	3409.2	2	1704.6			
Total SS	918765.2	14				
R^2=0.9236; Adjusted R^2=0.7861; L indicated linear effect; Q indicated quadratic effect						

In this Study with regards to MOE, The LOF is not Significant relative to the poor error, which indicating good response to the model. The model regression co- efficient of determination of (R^2) of 0.9236 for MOE is reasonable agreement with the experimental results, indicating 92.36 % of the variability can be revealed by the model and are left with 7.64 % residual variability for MOE.

Figure 4.4: Observed Vs. Predicted Plot for MOE



In addition to LOF test, the model for MOE was again evaluated by the observed vs. predicted plot. The points of all predicted and actual responses fell in 45° lines indicating also good response to the model.

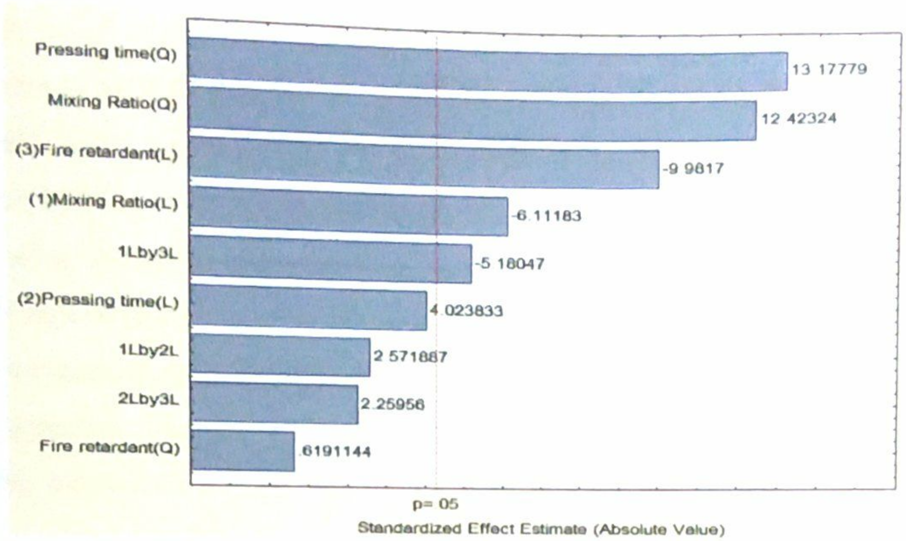
From the above Statistical results it may be inferred that the Box-Bhenken design was adequate to predict the board strength (MOE) within the range of variable studied. The final predicted process model in terms of actual significant factors for MOE equation.

MOE= -4632.44 + 846.53* mixing ratio – 266.93* mixing ratio^2 +1227.094*pressing time -70.785*pressing time^2 - 38.372 *fire retardant -1.47*fire retardant^2 -35.647 * mixing ratio * fire retardant.

4.1.2.2 Effects of Different Factors on MOE

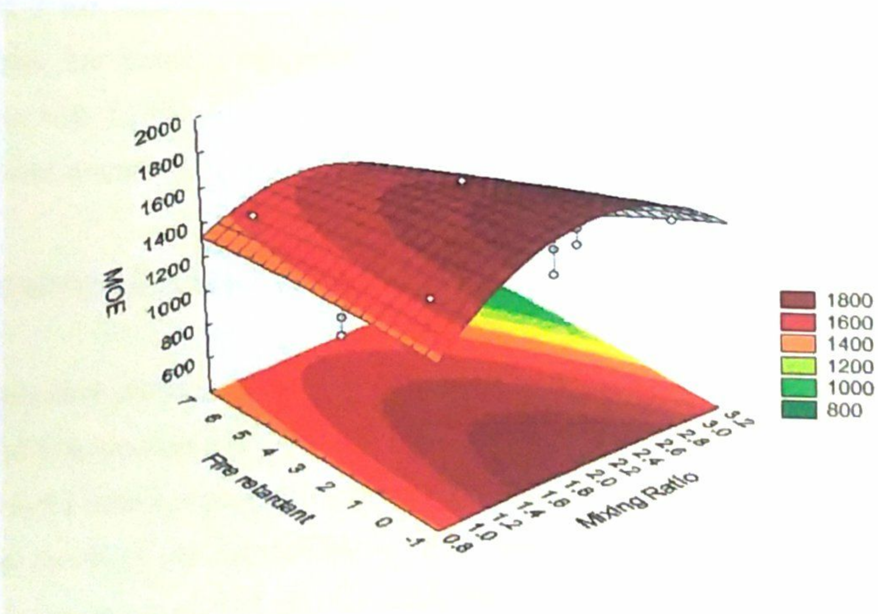
It has been observed from ANOVA and Standardized Pareto Chart for MOE of board that the quadratic effect of pressing time and quadratic effect of mixing ratio has positive significant effect on MOE and linear effect of fire retardant (%) and mixing ratio have an negative significant effect on MOE. It has been also observed that, the interaction between linear effect of mixing ratio and fire retardant (%) on MOE has negative significant effect on MOE.

Figure 4.5: Standardized Pareto Chart for MOE



The negative interaction effect of mixing ratio and fire retardant (%) on MOE is presented in the figure below-

Figure 4.6: 3d Graph Showing the Effect of Pressing Time and Fire Retardant % on MOE



From the Above Figure it has been observed that MOE attained the highest value 1400 N/mm^2 for mixing ratio ranges from 1.5 to 2.5 and a fire retardant (%) ranges from 2 to 4 percentages. As the mixing ratio starts to decrease from 1.5 to 1 or increase from 2.5 to 3 and as the fire retardant (%) starts to decrease from 2 to 0 percentage and starts to increase from 4 to 7 percent the MOE starts to decreases sharply.

In a Study it has been observed that there is an increase in modulus of elasticity with increasing pressing pressure, pressing time and decreasing the amount of LDPE addition for boards. The MOE of board in this study ranges from 1035 N/mm^2 to 1617 N/mm^2. The highest value of MOE is found in the board which is manufactured under the condition of mixing ratio 70:30, pressing time 13 minute, 180 °C temp and 5Mpa of pressure. The addition of LDPE to the wood particles increases the stiffness of the composite boards (Atuanya *et.al*, 2011). In anther study we have found that in preparation of board with LDPE the mechanical properties of board (tensile properties) increased with increasing wood fibre content from 0-20 wt%. Young's Modulus and yield strength values showed a rather progressive increase after initial decrease of wood fiber content percentage (Obidiegwu and Nwosu, 2012).

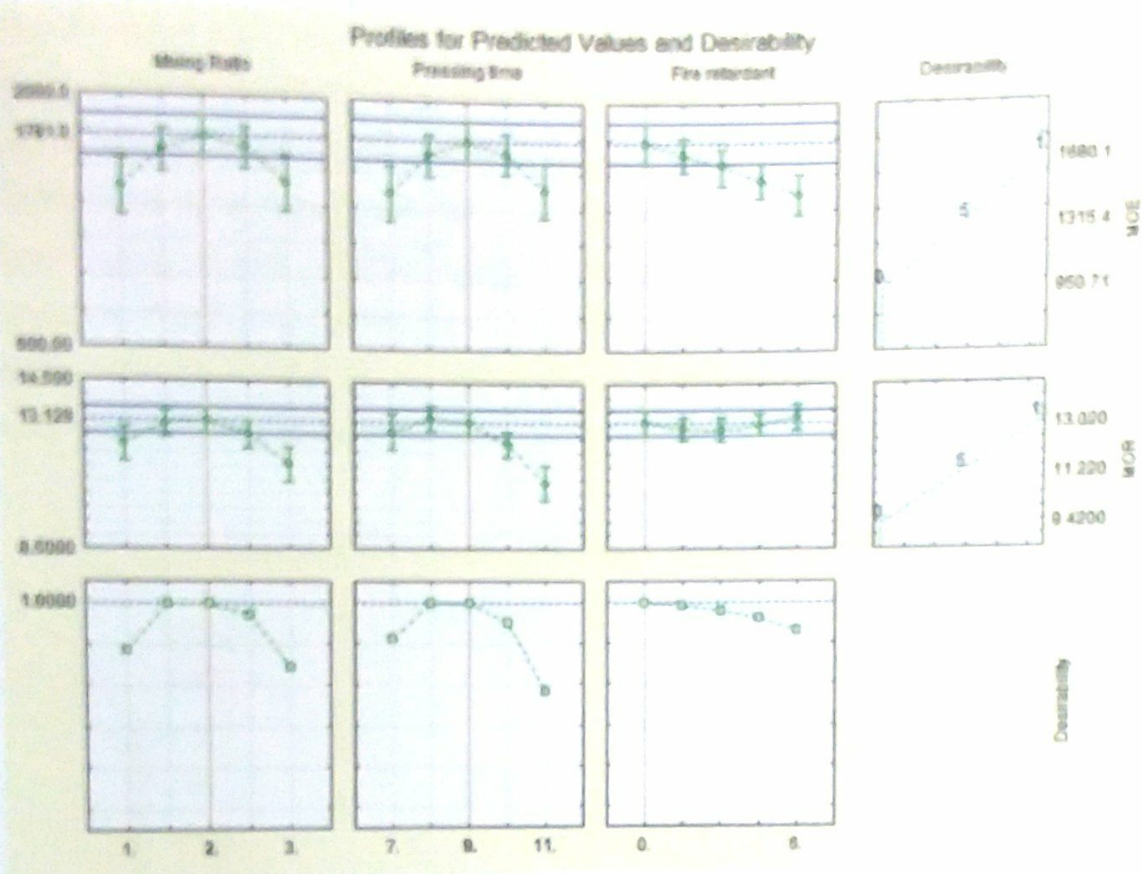
In another study where fire retardants are used as a coupling agent it was found that the MOE of the coupled specimens significantly improved with increasing the FR content. Here we have found 9085MPa, 9376MPa, 9557Mpa with the increase in fire retardant (Borax and Boric acid) 4%, 8% and 12% (Nadir *et.al,* 2012).

In a study it has been observed that when he amount of coupling agent 0%, hot pressing time 10min, hot pressing temperature 180°C hot pressing pressure 3.0 MPa, in WPC production with LDPE the modulus of elasticity (MOE) was found of 1482 MPa and internal bond strength (IB) of 1.20MPa (Zhang etal., 2013).

4.1.3 Optimization by Using Derringer's Desirability Function

The optimization process was done by selecting software profile and desirability option. In this desirability function a prediction profile for a dependent variable consists of a series of graphs, one for each independent variable, of the predicted values for the dependent variable at different levels of one independent variable, holding the levels of the other independent variables constant at specified values, called current values. If appropriate current values for the independent variables have been selected, inspecting the prediction profile can show which levels of the predictor variables produce the most desirable predicted response on the dependent variable.

Figure 4.7: Desirability Profile for Optimization of process Parameters for MOE



In this study the response MOR was optimized by desirability function. The best optimized conditions were found to be mixing ratio in the level of 2, pressing time 9 minutes and fire retardants at o percentage that also optimized 13.129 N/mm² and 1781.0 N/mm² for MOE (figure:4.8). In this study the best treatment condition were found: Mixing ratio 60:40 at level 3, pressing time 9 minutes and fire retardant (%) at 3%.As can be seen the best treatment conditions obtained from the experiment were very closed to the optimized condition mentioned before in the paragraph. Finally, by using optimized levels of parameters a confirmation study was executed which showed well response to the predicted model.

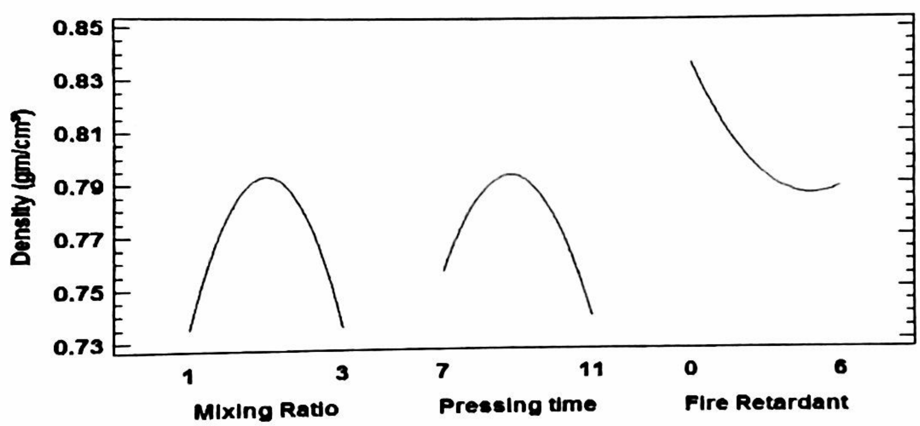
In a study the Optimum level of parameters of WPC production was coupling agent 0%, hot pressing time 10min, hot pressing temperature 180°C, hot pressing pressure 3.0 MPa, the mass ratio of alcohol consumption to plastic 3% (Zhang etal., 2013) which is almost similar to findings of this paper.

4.2 Physical Properties

4.2.1 Density

From the figure 4.8, it has been observed that, the Mixing ratio has a remarkable effect on density. It increases with the increase of plastic and highest at the level 2 means when the ratio of wood flour and LDPE is 60:40 and then it decreases with the further increase in LDPE. Again it has been found that density is rising with the rising of pressing time. It shows highest density at approximately 9 minute pressing. Then it decreases with the further increase in pressing time. The fire retardant % has an irregular effect on density, the highest value shows when the fire retardant is 0% then sharply fall with the increase in the percentage. It density slightly rises when the fire retardant % increase from 5 to 6 percent.

Figure 4.8: Effects of Production Parameters on Density

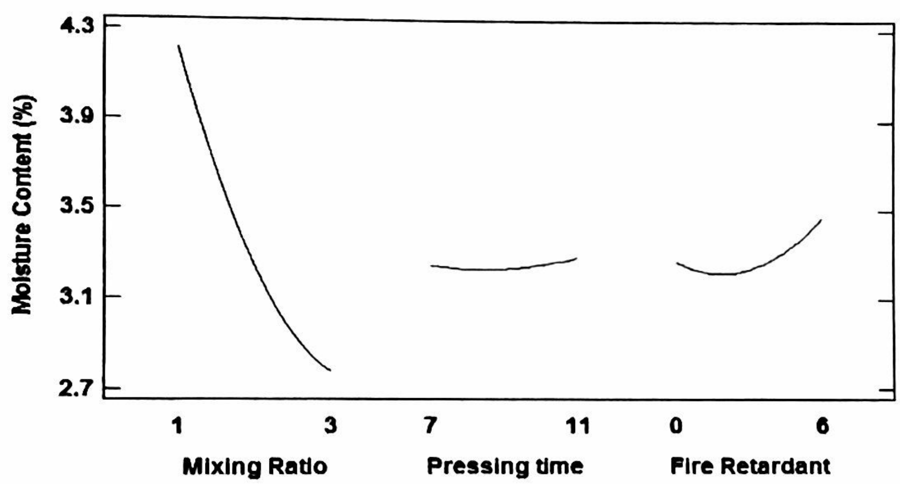


In a study, it has been found that, all WPC panels had a constant density of about 800 kg/m^3, the porosity contents of the WPCs containing fire retardant were higher than those of the WPCs without fire retardant at 50 wt % of the Wood flour. The porosity contents of the WPC panels increased with increasing volume percentage of the fire retardant. This was mainly attributed to different densities of the fire retardant (Ayrilmis etal. 2011). It also have been found that the density increases 1.05 gm/cm^3 to 1.15 gm/cm^3 with the decrease in plastic content (Yuan Leu, etal., 2011). In another study it has been found that the density of WPC increase 1050 to 1060 kg/m^3 with the increase in fire retardant % (Boric Acid+ Borax)% 4 to 8 Which was 1030 at 0% level and then decrease to 1050 at the level of 12 % of fire retardant (Ayrilmis etal. 2012). This is quite similar to the result found in this study.

4.2.2 Moisture Content %

From the figure 4.9, it has been observed that the Mixing ratio has a remarkable effect on moisture content percentage. It decreases with the increase of plastic and highest at the level 1 means when the ratio of wood flour and LDPE is 70:30. Again it has been found that pressing time has such no remarkable effect on Moisture content %. It remains almost same in increasing of pressing time. The fire retardant % has an irregular effect on moisture content %, the highest value shows when the fire retardant is 6 %, when it raises from 0 % then it shows lowest value and then reached at highest value at 6%.

Figure 4.9: Effects of Production Parameters on Moisture Content%

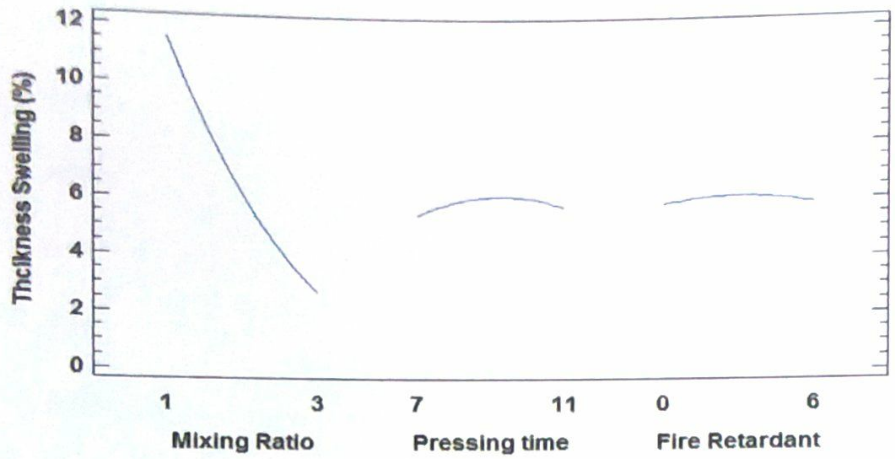


In a study it has been found that the moisture content% increases 1.34% to 3028 % with the decrease in plastic content (Yuan Leu, etal., 2011). In a study it has been found that, they got the optimal pressing time was found 10 min and in that 24h water absorption was 24.8% (Zhang etal., 2013) which is almost similar to the result of this study.

4.2.3 Thickness Swelling %

From the figure 4.10, it has been observed that the thickness swelling % decreases with the increase in the level of mixing ratio. It shows highest value in the mixing ratio level 1 (70:30) which means it decreases with the increase in LDPE. Again it has been found that pressing time has such no remarkable effect on thickness swelling %. It slightly increases and after certain a limit decreases with the increase in pressing time. It has been also observed that fire retardant % has such no remarkable effect on thickness swelling %.

Figure 4.10: Effects of Production Parameters on Thickness Swelling %



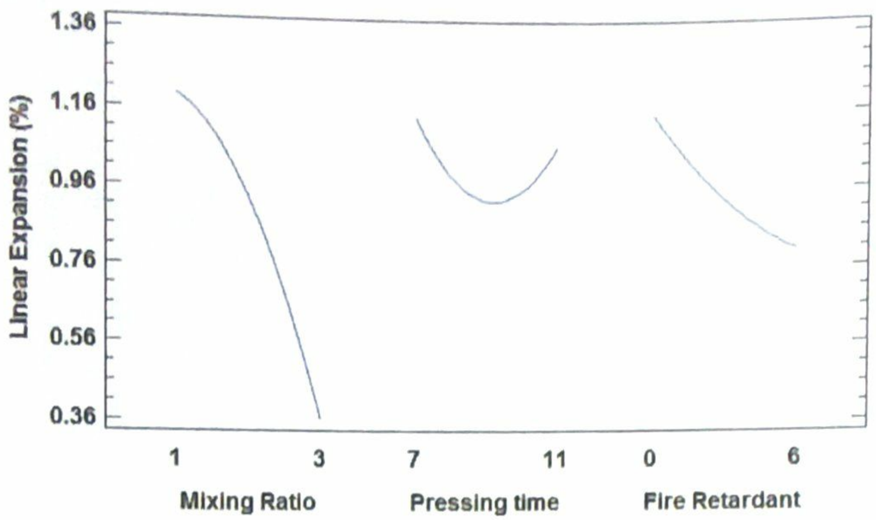
In another study it has been found that the thickness swelling of WPC decreases 1.24% to 1.04% with the increase in fire retardant % (Boric Acid+ Borax)% 4 to 8 and then increases to 1.17 % at the level of 12 % of fire retardant (Ayrilmis etal. 2011). This is quite similar to the result found in this study.

In a study it has been found that the density increases 0.26% to 1.57% with the decrease in plastic content (Yuan Leu, etal., 2011). In a study it has been found that, they got the optimal pressing temperature 10 min and in that 24h thickness swelling of 5.69% (Zhang etal., 2013) which is almost similar to the result of this study.

4.2.4 Linear Expansion %

From the figure 4.11, it has been observed that the linear expansion % decreases with the increase in the level of mixing ratio means with the increase in LDPE. It shows highest value in the mixing ratio level 1 (70:30). Linear expansion% shows highest value in pressing time 7 minutes then decreases with the increase in the pressing time and after a certain time it decreases with the further increase in pressing time. Again it has been observed that the linear expansion% decreases with the increase in fire retardant %.

Figure 4.11: Effects of Production Parameters on Linear Expansion%.

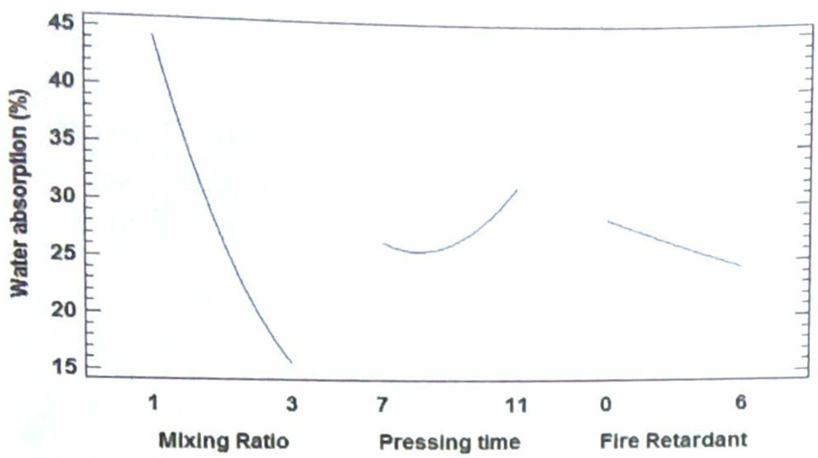


As at level 1 which contains 70 % of wood flour, soak water more than level 3 which contain 50 % of LDPE. So, linear Expansion is high at mixing ratio level 1. Pressing time has an irregular effect on linear expansion as it has a complex interaction with the other factors. The effect of fire retardant % has got almost a similar trend with the water absorption %. With the increase of fire retardant % the linear expansion decreases gradually as the water absorption%.

4.2.5 Water Absorption %

From the figure 4.12, it has been observed that the water absorption percentage decreases with the increase in the level of mixing ratio means with the increase in LDPE. It shows highest value in the mixing ratio level 1 (70:30). Water absorption percentage shows highest value in pressing time 11 minutes. It slightly decreases with the increase in pressing time at first then sharply increases with the increase in the pressing time. Again it has been observed that the water absorption percentage decreases with the increase in fire retardant percentage.

Figure 4.12: Effects of Production Parameters on Water Absorption %



In a study it has been found that, in compared with the control specimens, the water resistance of the uncoupled specimens significantly decreased with increasing the FR content with the addition of the coupling agent and when the MAPP and the corresponding FR contents were increased from 2 to 4 wt. % and 4 to 8 wt. %, respectively (Ayrilmis etal. 2011). In another study it has been found that the water absorption % of WPC increase 1.88%, 1.98% and 2.20% with the increase in fire retardant % (Boric Acid+ Borax)% 4, 8 and 12 % respectively ,which was 1.60 at 0% level (Ayrilmis etal. 2012). The interaction of other factors can decrease the water absorption % with the increase in the fire retardant % in this study.

The results showed the WPCs with different wood and plastic contents and the sample amount of the coupling agent and lubricant, with the increase of wood content, the maximum water adsorption of the WPCs increased (Yuan Leu, etal., 2011). In a study it has been found that, they got the optimal pressing temperature was found 10 min and in that 24h water absorption was 24.8% (Zhang etal., 2013) which is almost similar to the result of this study.

The result Summary was given in the following table:

Table 4.3: Result Summary

Run	Mixing ratio (level)	Pressing time (min)	Fire retardant %	MOE	MOR	Density (Kg/m³)	MC %	TS %	LE %	WA %
1	3.0	9.0	0.0	1468.38	11.56	0.79	2.84	16.52	2.48	0.36
2	2.0	11.0	0.0	1562.61	11.33	0.81	3.38	30.98	5.29	0.89
3(C)	**2.0**	**9.0**	**3.0**	**1601.82**	**13.02**	0.78	3.25	25.5	5.91	0.9
4(C)	**2.0**	**9.0**	**3.0**	**1663.74**	**12.95**	0.81	3.2	25.15	6.32	0.94
5(C)	**2.0**	**9.0**	**3.0**	**1680.09**	**12.7**	0.79	3.27	28.67	6.19	0.95
6	3.0	9.0	6.0	950.71	11.56	0.74	2.92	17.6	2.21	0.21
7	2.0	11.0	6.0	1376.87	12.87	0.68	3.58	32.03	6.77	1.09
8	1.0	9.0	6.0	1482.14	12.78	0.74	4.53	35.71	11.08	1.3
9	1.0	7.0	3.0	1171.39	11.65	0.69	4.28	43.26	12.58	1.21
10	2.0	7.0	0.0	1420.63	12.41	0.79	3.21	29.08	4.83	1.97
11	2.0	7.0	6.0	1048.31	11.25	0.79	3.45	24.82	4.38	0.66
12	1.0	11.0	3.0	1064.88	10.19	0.71	4.16	53.37	9.36	1.29
13	3.0	11.0	3.0	1131.75	9.42	0.68	2.83	17.48	2.99	0.8
14	3.0	7.0	3.0	1025.89	10.47	0.68	2.86	16.55	1.55	0.52
15	1.0	9.0	0.0	1572.04	12.3	0.75	4.24	50.61	11.69	1.43

Chapter Five: Conclusion

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Timber shortage is increasing in worldwide so scientific use of timber wastages like saw dust in composite board production can contribute greatly to meet the requirement. The utilization of Low density polyethylene (LDPE) in composite board production can also helps to reduce the negative impact on environment of utilization of toxic materials as the surface of WPC would slowly degrade with continuous exposure to UV light. In this purpose the statistical optimization tool was executed which helps in reducing the waste of raw materials. Again derringer's desirability function was employed for the multi-response optimization of production parameters. Here the maximum MOE and MOR were found in the condition of 60:40 (wood: LDPE) ratio, pressing time 9 minutes and 3% fire retardant. The maximum optimized MOE and MOR were found by the optimal conditions of mixing ratio 60:40, pressing time 9 minutes and 0 fire retardants percentage. The optimized MOR and MOE were found 13.129 N/mm^2 and 1781.0 N/mm^2 respectively. The highest Density was found 0.81Kg/cm^3. The lowest moisture content %, water absorption %, Thickness swelling % and linear Expansion % was found 2.83%, 0.21%, 16.52% and 1.55% respectively at different combinations. Finally a confirmation study was executed to prove the adequacy of Response surface methodology with Derringer's desirability function for the design and optimization of the process parameters for the Wood-LDPE composite board production.

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