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The American University in Cairo
School of Sciences and Engineering

Investigating the Mechanical and Physical Properties of Wood Plastic Composites (WPC)

BY

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A thesis submitted in partial fulfillment of the requirements for the degree of

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Under the supervision of:

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ABSTRACT

Wood and plastic wastes have been a major environmental concern not only in Egypt but also worldwide. Plastic wastes are classified as recyclable plastic such as bottles and non-recyclable plastic such as plastic bags especially contaminated bags (rejected plastic). Plastic waste is a non biodegradable material calling for an appropriate method of disposal; however, the current approach adopted in Egypt relies mainly on throwing away in dumpsites. Therefore, it is a costless raw material which needs to be invested. In this thesis, the wood waste and the rejected plastic were recycled to produce new useful product; Wood Plastic Composite (WPC), having characteristics similar or close to commercial wood. An innovative, clean, cheap, and effective yet simple technology with different procedures was introduced in this thesis to demonstrate the suitability of wood plastic composites' techniques for developing countries. Testing was done for some important mechanical properties; flexural strength and modulus, and physical properties; water absorption and thickness swelling, which has proven an acceptable final product and promising results; especially regarding the physical test. The design and analysis of experimental work was built on using design of experiments. Special type of experimental designs; design with mixtures, was adopted because it deals with dependent factors; mixture ingredients. Talc was added to the mixture as a mineral additive. The impact of factors (wood waste, plastic waste, and talc) on the physical and mechanical properties of the WPC (flexural strength and modulus, water absorption and thickness swelling) was investigated based on full analysis of variance (ANOVA). It showed that the plastic waste was the most negative affecting factor; this was contributed to the variability in batches produced in addition to the impurities content. Talc resulted in increasing the

flexural strength and modulus. Wood with size of up to 0.5mm has proven to affect the flexural modulus response negatively; when increased. A mathematical model and a response surface representing the factors and their responses; that could be used for future forecasting of the properties without performing physical experiments, were obtained for flexural strength and modulus after conducting several trials till reaching the final experimental design within the navigation space. All these trials were based on an algorithm that was introduced to reach the best feasible model and response surface. A completed residual analysis of the model was done in every trial of the algorithm; where every point within the design was analyzed, till reaching the final model. The best possible mix that enhances the flexural strength to the maximum possible was obtained when the talc was close to 30%, plastic waste 50% and wood waste (of particle size up to 1.18mm) and wood waste (particle up to 0.5mm) of average percentages of 10%. For the flexural modulus, best mix values were obtained when talc is close to 35%, plastic waste 40%, and wood waste (particle up to 1.18mm) about 15% and wood waste (particle up to 0.5mm) 10%. A comparison study; using hypothesis testing, between 7 types of commercial wood (plywood, pinewood, beech wood, maple wood, Fiberboard, Medium Density Fiber wood (MDF), and compressed wood) and WPC was conducted to validate the application of the WPC. It showed that the WPC had the lowest water absorption and thickness swelling percentages compared to others (maximum of 1.7%, average of 0.4% and standard deviation of 0.28%); in addition, it showed that WPC flexural strength performs like compressed wood. However, flexural strength and modulus were less regarding other types of wood.

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ACRONYMS & ABBREVIATIONS

ABS	Acrylonitrile Butadiene Styrene
ANOVA	Analysis of Variance
ASTM	The American Society for Testing and Materials
AUC	The American University in Cairo
CCA	Chromated Copper Arsenate
Cook's D	Cook's Distance
CV	Coefficient of Variation
DEQ	Department of Environmental Quality
DFBETAS	Difference in Betas
DFFITS	Difference in Fits
DOE	Design of Experiments
HDPE	High Density Polyethylene
ICC-EC	International Code Council – Evaluation Services
LDPE	Low Density Polyethylene
MAPP	Maleated Polypropylene
MDF	Medium Density Fiber
MSW	Municipal Solid Waste
PP	Polypropylene
PVC	Polyvinyl Chloride
R	Replication run
RPM	Revolutions per Minute
Std	Standard number
Std. Dev.	Standard Deviation
WPC	Wood Plastic Composite

CHAPTER 1 INTRODUCTION

Plastic and wood wastes have been a main environmental concern. Plastic is the biggest problem due to its high amount of waste generated, non biodegradability and the fastest depletion of natural resources regarding its short life cycle, therefore increased amount of material utilized in its production, and waste generated. The same applies to wood with lesser degree where it is depleting trees and forests and the wastes mainly are either burned or disposed; resulting in extra consumption, depletion, and pollution of nature. Several worldwide attempts have been adopted; especially in the developed countries, to take advantage of these types of waste especially with the raised need for alternatives to virgin materials (Winandy, Stark and Clemons 2004). These trials were basically built on the concept of a Cradle to Cradle approach where the material is recycled at the end of its life cycle to produce a Cradle (new) product and thus close the loop and imitate the natural ecosystem (McDonough 2002). As a consequence, this minimizes the solid waste content. Therefore, costs, energy, and depletion of virgin materials are reduced. In addition, it assures the sustainability over the incoming years for future generations' use (Youngquist, Myers and Harten 1992). Wood plastic composite (WPC) is a product that could be obtained from plastic and wood. WPC is a composite with a rapid growing usage consisting of a mixture of wood and polymeric material (Soury, et al. 2009)

1.1. WPC Value

WPC has become currently an important address of research that gained popularity over the last decade especially with its properties and advantages that attracted researchers such as: high durability, Low maintenance, acceptable relative strength and stiffness, fewer prices relative to other competing materials, and the fact that it is a natural resource) (Bengtsson and Oksman 2006) & (Winandy, Stark and Clemons 2004)). Other advantages have been strength points including (Wechsler and Hiziroglu 2007): the resistance in opposition to biological deterioration especially for outdoor applications where untreated timber products are not suitable, the high availability of fine particles of wood waste is a main point of attraction which guarantees sustainability, improved thermal and creep performance relative to unfilled plastics where It can be produced to obtain structural building applications including: profiles, sheathings, decking, roof tiles, and window trims.

On the other hand, WPCs are not nearly as stiff as solid wood; however, they are stiffer than unfilled plastics. In addition, they do not require special fasteners or design changes in application as they perform like conventional wood (Clemons and Caufield 2005).

As mentioned, the reasons for using WPC are many; however, there are other causes that enforced many countries to tend for using alternative sources to virgin materials. In the United States, for example, the U.S. Environmental Protection Agency, by the beginning of 2004, has phased out the usage of wood treated with chemicals such as the chromated copper arsenate (CCA) to prevent environmental and microbial

degradation (Yeh and Gupta 2008). As this type of wood was used in the building products' market concerned with residential applications such as decking, the need for the alternative survived the WPC market (Yeh and Gupta 2008). In Europe, environmental concerns are focused on limiting the use of finite resources and the need to manage waste disposal; therefore, the tendency to recycle materials at the end of their useful life has increased tremendously (Yeh, Agarwal and Gupta 2009). Recycling polymers in Europe was less preferred than other types of materials such as metal; however, illegality of land filling and waste management priority in many European countries were the motive to do so (Yeh, Agarwal and Gupta 2009). In addition to the enforced environmental policies, the growth of environmental awareness led to a new orientation to use wasted natural materials for different applications and industries such as the automotive, packaging and construction industries (Yeh, Agarwal and Gupta 2009).

1.2. Market Study

The awaiting market for WPC is huge due to the high production of plastics and wood which constitutes a significant amount of municipal solid waste (MSW) which is mostly disposed not recovered (Adhikary, Pang and Staiger 2008). Najafi, et al. 2007, mentioned that WPC presents a promising raw material source for new value added products due to the large amount of daily waste generation and low cost (Najafi, Tajvidi and Hamidina 2007).

WPC commercial products are increasingly replacing many products in many applications especially the construction related ones (Yeh, Agarwal and Gupta 2009)

WPCs have gained an ever larger share; especially for decks and other outdoor structures (Youngquist, Myers and Harten 1992). Other production lines of fencing, roofing, and siding have started to get a noticeable market share (Winandy, Stark and Clemons 2004). WPC usage is extensively spread especially in strips; where wood peel layers are tilted in the same direction, used in furniture industry (Augutis 2004). WPC is also used in producing panels where it is produced by mixing wood flour and plastics giving a material that can be processed similar to 100% plastic-based products (A. Wechsler, S. Hiziroglu, 2007).

Approximately one-half of all industrial materials used in the United States are wood-based; thus, the finding that the WPC market is increasing is not a surprise (Falk 1997). The growth of WPC decking in the U.S. has started from less than 1 % in mid-90's to over 10% today with growth projected by several studies to reach 20% before the end of 2010 (Winandy, Stark and Clemons 2004).

Two large sectors, the decking and fencing sector, the siding and roofing sector started to use the WPCs commercially in the U.S. (Winandy, Stark and Clemons 2004). Concerning the decking and fencing in the U.S., a study was done in 2002 which showed that there were 1.4 million new houses constructed (for single families) and 0.3 million new houses for multi-families; where the house averaged about 215 m2 made from wooden decks (Winandy, Stark and Clemons 2004). Winandy, et al. 2004, concluded that all this huge amount of consumed wood could be substituted by WPC. The U.S. decking market alone uses a sum total of nearly 18.5 million m^3 of wood where 90% uses natural treated wood and 10% WPC (Winandy, Stark and Clemons 2004).

In addition, the U.S. fencing market was divided into 45% wood, 44% metal, 7% plastic and 5% other materials (Winandy, Stark and Clemons 2004). It was calculated at $US 2.6 billion in 2002 and was expected to grow approximately 5% per year and therefore a great potential of WPC domination was expected (Winandy, Stark and Clemons 2004).

1.3. Applications

Advantages, desired properties, environmental regulations, and awareness have led to the substitution of using conventional woods with the WPC. Its production is growing over time due to its several applications (Adhikary, Pang and Staiger 2008). Main motives include:

- It can be molded in any particular mold with a variety of shapes and angles, so it can give any desired design (Takatani, Ikeda and Sakamoto 2007).
- It can be treated in the same manner as the conventional wood using the same cutting and sawing equipment (Winandy, Stark and Clemons 2004).

Therefore, it is easy to use any conventional wood workshop with WPC products that have proven to give the same functionality as conventional wood in many areas (Wechslera and Hiziroglub 2009). Various WPC products are available in the US market substituting some of the conventional wood products such as outdoor deck floors (Winandy, Stark and Clemons 2004). It is also used for railings, fences, landscaping timbers, siding, park benches, molding and trim, window and door frames, panels and indoor furniture (Winandy, Stark and Clemons 2004).

In addition, Wood plastic composites can also substitute neat plastics in applications where the need for an increase in stiffness is an addition; where the wood fiber elasticity is almost 40 times higher than that of polyethylene and the overall strength is approximately 20 times greater (Bengtsson and Oksman 2006). It has also higher thermal and creep performance compared to plastics and thus could be used in many structural building applications (Wechslera and Hiziroglub 2009).

A high potential of using WPC in a large scale to produce pallets is raised by Soury, et al. 2009; whereas the amount of consumed of wooden pallets are huge (400 million pallets) accounting for about 86% of all pallets sold worldwide. They added that due to the disadvantages of wood consisted of product degradation due to environmental factors; an alternative WPC could be the best option (Soury, et al. 2009).

WPC started to be utilized in siding market in 2003 based on studies done in 2002 that revealed that wood occupied about 17% of the materials share of the U.S. siding market (960 million square meters) (Winandy, Stark and Clemons 2004). Therefore, a promising market was opened for WPC products which gave a promising performance over other materials such as aluminum and vinyl and similar to wood (Winandy, Stark and Clemons 2004).

1.4. Objectives Of This Work

1.4.1. Problem statement and work purpose

As addressed within the introduction, wastes of plastic and wood are a major environmental concern that needs to be dealt with to minimize the amount of municipal solid waste, depletion of natural resources, saving the environment, and enhancing the sustainability concept for future generations' use. As a consequence, the purpose of this study is to take advantage of these useful wastes (unutilized fortune) by:

- *Recycling* the plastic and wood wastes; obtained mainly from contaminated plastic bags and sawdust waste, into new useful product (wood plastic composite) having characteristics similar or close to commercial wood; using a simple technology that could be adopted in Egypt and any developing country.

- Investigating the impact of parameters (wood waste, plastic waste, and talc) on the physical and mechanical properties of the WPC (flexural strength and modulus, water absorption and thickness swelling)

- Obtaining a mathematical model and a response surface representing the parameters and their responses; that could be used for future forecasting of the properties without conducting physical experiment.

- Determining the parameters that affect the model the most.

- Obtaining the best possible mix that enhances the response (mechanical and physical properties) to the maximum possible.

1.4.2. Work approach

All the experiments conducted in this work were designed from beginning using constrained mixture design of experiments to produce WPC specimens with properties that could be analyzed in a right way to obtain valid results. The manufacturing of WPC was based on extrusion technology. It started with pilot experimentations to get the feeling of the factors that should be included, excluded, and added. The mechanical and physical properties (flexural strength and modulus, and water absorption and thickness swelling) of specimens were analyzed. A response surface and a mathematical model obtained; therefore, a best possible mix was reached. Finally, a comparison was conducted between the obtained WPC and 7 types of commercial wood; using hypothesis testing, to validate the adoption of WPC.

CHAPTER 2 LITERATURE REVIEW

2.1. Plastic Waste

The market potential regarding the usage of plastic waste into other utilizations is huge due to the high amounts of its disposition which constitute the largest share of the global municipal solid waste (MSW). Kikuchi, et al 2008, mentioned that the plastic waste constitutes more than 60% of the total MSW, 22% was recovered and 78% disposed (Kikuchi, Kukacka and Robert 2008). In United States, the waste of plastics; in 2005, was calculated as 11.8% of the 246 million tons of MSW generated (USEPA 2006). In India, Plastic in municipal solid waste makes up to 9–12% by weight of the total In addition to other wastes that may contain much higher proportions of plastics (Panda, Singh and Mishra 2010).

The majority of the plastic wastes generated are disposed (Kikuchi, Kukacka and Robert 2008). However, the continuous growth of worldwide plastic consumption due to its short life cycle compared to other products; roughly 40% have duration of life cycle smaller than 1 month, and the legislations of many countries concerned with minimizing landfills content and incinerators have led to a necessity of recovering plastic waste instead of disposing ((Kikuchi, Kukacka and Robert 2008) & (Panda, Singh and Mishra 2010)).

Incineration and land filling alternatives were rejected by several countries due to their potential danger to the environment either by polluting air or land; which results in not closing the loop of Cradle to Cradle and therefore depleting natural resources.

As a consequence, the tendency towards recycling has increased (Jayaraman and Bhattacharyya 2004). Some attempts for plastic recovery resulted during 2004 in a recovery of almost 8.25 million tons (39% of total amount of plastics consumed) in Western Europe; 35,000 tons (13.48% of total imported virgin plastics) in New Zealand (Adhikary, Pang and Staiger 2008). While in 2005, the United States recycled around 5.7% of the total plastics generated (USEPA 2006). On the other hand, some states in the US like Michigan have a recycling rate that is close to 100% (Beg and Pickering 2008). In Brazil, some potential in recycling have been raised where around 15% of all plastics consumed are recycled and returned to industry (Beg and Pickering 2008).

2.2. Wood Waste

Generally, Wood waste comes from both commercial and residential activities; which could include scrap lumber, pallets, sawdust, tree stumps, branches, twigs, wooden crates and pallets, building construction and demolition, furniture manufacturing, and many others.

Wood waste is one of the main environmental concerns. In the United States, a report that was written in 1995 by CIWMB (California Integrated Waste Management Board) tells that severe problems concerned with landfill disposing were revealed (CIWMB 1995). It tells that the construction and demolition of buildings; which are mainly wood waste, generates almost twelve percent of all solid waste in California. Furthermore, the average fee for disposing of a ton of waste in a California landfill is about $30 to $35, but disposing of a ton of wood at a wood processing facility may only cost $10. In addition, the amount of wasted wood disposed in landfills in some regions in California reaches 90 percent of the total wood waste (CIWMB 1995).

Adhikary, et al. 2008, stated that a large amount of wood waste is generated from wood industry at different stages of the processing of wood; which is disposed mostly in landfills; Besides, the hazardous content of the wood waste are numerous and takes time to decompose (Adhikary, Pang and Staiger 2008).

The Department of Environmental Quality (DEQ) in the United States reported that the other alternative; that used to be used, to get rid of wood wastes instead of disposing was burning (DEQ 2009). Wood burners were used at first and as a result of their environmental hazards; represented in huge amount of smoke & ash generated directly to the atmosphere polluting air and ambient, were shut down and prohibited from being used (DEQ 2009).

Currently, a tremendous shift is done in the area of wood burning especially with the developed ideas of avoiding the environmental hazards. Therefore, the use of wood waste in WPC helps to overcome disposal and burning hazards and costs (Adhikary, Pang and Staiger 2008).

2.3. Material Utilized in WPC

Wood and plastics (virgin or recycled) with various types, grades, sizes, and conditions are the main materials utilized in WPC production. WPC is composed mainly from a plastic matrix reinforced with wood. Several ingredients of WPC are found in literature.

Najafi, et al. 2007, mentioned that WPC is a composite composed from a natural fiber/filler (such as kenaf fiber, wood flour, hemp, sisal etc.) which is mixed with a thermoplastic. They added that virgin thermoplastic materials (e.g. high and low density polyethylene (LDPE and HDPE), polypropylene (PP), polyvinyl chloride (PVC))

are commonly utilized. In addition, any recycled plastic which can melt and be processed in a temperature less than the degradation temperature of the wood filler (200 C) could be used to produce WPC (Najafi, Tajvidi and Hamidina 2007). Morton and Rossi (2003) said that the huge majority of WPC utilizes polyethylene and they classified the types of plastic used in WPC as follow: polyethylene (83%), polyvinyl chloride (9%), polypropylene (7%), others (1%) (Morton and Rossi 2003). Clemons and Caufield added that wood flour is obtained from wood wasted from wood processors. They said also that it should be from high quality and free of bark, dirt, and other foreign matter. Moreover, species are mainly selected based on regional availability of high quality flour and color. Pine, oak, and maple are the most common used in the United States (Clemons and Caufield 2005). Adhikary, et al. 2008, used recycled and virgin high density polyethylene (HDPE) with wood flour (Pinus radiata) as filler. The HDPE utilized was obtained from plastics recycling plant and sawdust was collected from a local sawmill (Adhikary, Pang and Staiger 2008)

2.3.1. Virgin or recycled (non-virgin) material in WPC production

The issue of producing WPC using virgin or recycled (non-virgin) material is been controversial. When searching in literature, various opinions were found regarding the practicality of usage, mechanical properties, physical properties, and even final product look or appearance.

Various comparisons were done between virgin and recycled materials using many conditions have shown agreements of authors with the use of recycled material and other times disagreements. However, studies based on recycled products are very limited (Adhikary, Pang and Staiger 2008) and almost all producers of the commercial

scale WPC are using plastic virgin materials (Klyosov 2007). This tendency could be due to the fear of obtaining a product with non controllable physical and mechanical properties resulted from impurities as justified by Yeh, et al. 2009, on research scale (Yeh, Agarwal and Gupta 2009). Conversely, Adhikary, et al. 2008, used in their study post-consumer HDPE which was collected from plastics' recycling plant and sawdust was obtained from a local sawmill. They have shown in their study the feasibility of making composite panels from recycled HDPE using hot-press molding technique. They added that the obtained product has proven superior dimensional stability when compared to virgin HDPE and equivalent tensile and flexural properties of the composites. On the other hand, Yeh, et al. 2009, showed that wood with recycled ABS resulted in poor and variable mechanical properties as compared to the relevant virgin ABS. They added that unlikable odor is obtained from recycled material which could be avoided by adding a thin layer of virgin polymer (Yeh, Agarwal and Gupta 2009).

Regarding physical properties, Adhikary, et al. 2008, showed that the panels gave very low water absorption and thickness swelling thus the products was considered stable in humid environment. (Adhikary, Pang and Staiger 2008). In contrast, Najafi, et al. 2007, have tested water absorption and thickness swelling of WPC obtained from sawdust and recycled and virgin plastic; HDPE and PP. The test consisted of 2 hr and 24 hr submersion tests.

The results showed that recycled WPC absorbed more than virgin, PP absorbs water more than HDPE, and the mix of recycled HDPE and PP absorbed the maximum (Najafi, Tajvidi and Hamidina 2007).

Yeh, et al. 2009, and Adhikary, et al. 2008, have found variable performance of their final product. It was justified by Adhikary, et al. 2008, by the different grades and

colors of waste stream used and the material contaminants. They said also that the impact is not still fully understood which calls for further investigations and opens a new area of research (Adhikary, Pang and Staiger 2008). Moreover, the problem was addressed by Yeh, et al. 2009, as the reuse of polymers obtained from post-consumer application caused unpleasant outcomes many times. They justified by basing their claim on the impurities contained within the material; which led to decrease the mechanical and thermal behavior. The authors added that; based on their findings, impurities would affect the product impact strength and ductility negatively to the extent even if it was of 1 % of amount. Another problem accompanied with impurities in polymers is that the cost of its disposal will be more than using virgin material (Yeh, Agarwal and Gupta 2009). Therefore, it could be concluded that the main problem lies in the variable performance or different outcomes of the same material settings when tested. This issue was also discovered in findings of this work as I will be shown in chapter 6. In addition, the results of this work (see chapter 6) will show that the main cause of variability is impurities and contaminants agreeing with authors; as mentioned above. However, the environmental savings from using non-virgin material, availability, high properties, and almost no-cost should be the stimuli behind using recycled material instead of virgin.

2.3.2. WPC additives

The majority of the WPC physical and mechanical properties are depending on mostly on the interaction developed between wood and the plastic. One of the ways to increase this interaction is adding an additive (Wechslera and Hiziroglub 2009).

Generally, the additives enhance the compatibility between hydrophilic wood and hydrophobic plastic allowing the formation of single-phase composite (Wechslera and

Hiziroglub 2009). The two main families of additives that are used with WPC are mineral additives and coupling agents. The most famous coupling agent utilized in literature by many researchers is the MAPP; on the other hand, the most famous mineral additive utilized is the talc and calcium carbonate (Klyosov 2007), (Adhikary, Pang and Staiger 2008), and (Fabiyi, et al. 2008).

Maleated polyolefins, organosilanes, and acrylic-modified polytetrafluoroethylene (PTFE) are the most famous family of coupling agents which are added to the composite with minimal percentages; typically less than 5% (Klyosov 2007), (Adhikary, Pang and Staiger 2008), (Bengtsson and Oksman 2006), and (Fabiyi, et al. 2008). Typically, coupling agents act to provide better flowability of the molten composite, therefore better compatibility obtained, and strength enhanced (Klyosov 2007). However, many arguments were raised mentioning that coupling agents do not provide strong adhesion between fiber and plastic; which is the main intended function (Klyosov 2007). In addition, coupling agents were mainly used in literature with virgin material and the literature didn't emphasize much on its use with recycled. This could be due to the coupling agent composition which is directly related with the type of polymer adopted (Klyosov 2007); whereas, the recycled polymer could be a mix of many polymers with impurities.

 Therefore, the intended adhesion function maybe reduced for this reason. On the other hand, an availability concern of coupling agents should be raised; whereas, it is not available in local Egyptian market or near Middle East markets; they are mainly available in The United States and Europe which is reflected at last in terms of high cost. As a consequence, mineral additive were the chosen additive to WPC in this work.

The main mineral additives adopted in literature are talc ($Mg_3Si_4O_{10}(OH)_2$), calcium carbonate, silica, glass fiber, kaoline clay ($Al_2O_3.2SiO_2.2H_2O$), wollastonite ($CaSiO_3$) (Klyosov 2007). Talc and calcium carbonate are most common additives used in WPC production due to their good outcome in enhancing mechanical properties, availability, and cheap cost (Klyosov 2007).

Additionally, talc is the most additive used in literature due its good absorption of water; to minimize the wood moisture, its natural similarity to oil in addition to its distinct platy shape (non uniform layered composition) making it a good filler for hydrophobic plastic (Klyosov 2007). In addition, talc was utilized by many researchers because it has proven that it enhances WPC mechanical properties ((Fabiyi, et al. 2008)& (Klyosov 2007)). Due to all mentioned properties of talc, adding the availability, and the low cost factor; in the local Egyptian market, talc was used in this work.

2.4. WPC Manufacturing Techniques

Various techniques were adopted in literature to manufacture WPC, however; the two main adopted techniques are extrusion and injection molding.

Typically, the extrusion process produces continuous linear profiles via forcing a melted thermoplastic through a die; on the other hand, the injection molding process produces three-dimensional items with minimizing the stages of post-manufacturing (Migneault, et al. 2009).

The manufacturing techniques adopted by Bengtsson and Oksman, 2006, were based on drying wood flour at 100 C to reach a moisture content of 0.3%. The dried wood and plastic granules were then fed to the co-rotating twin-screw extruder at

temperatures varying from 165 to 200 C. A rectangular die was used at the extruder end and the extrudates were then cooled at ambient temperature (Bengtsson and Oksman 2006). Yeh, et al. 2009, divided the WPC manufacturing process into two main parts. The first part consisted of compounding the material; using a twin-screw extruder. The second part was to obtain profiles via single-screw extruder or use injection molding to obtain a product resembling to wood in look and properties (Yeh, Agarwal and Gupta 2009). Bouafif, et al. 2009, produced WPC in a two-stage process. In the first stage, a co-rotating twin-screw extruder was used to compound wood particles with HDPE into pellets at temperatures from 180 C to 190 C. In the second stage, a reciprocating screw injection molding machine was used to inject WPC test specimens (Bouafif, et al. 2009). Soury, et al. 2009, manufactured WPC pallets by firstly producing profiles; utilizing counter-rotating twin screw extruder, and then assembling them by using nails, rivets and screws. The authors found an advantage of adopting extrusion instead of injection molding represented in the high challenge of producing one piece pallet in injection molding which could be make the wood; in the composite, burn. This is because the high shear rate in the rapid injection speed and therefore excessive heat generated causing burn to the product. On the hand, extrusion is much less generating shear and therefore heat; in addition, it is more flexible in terms of adoption of various die designs (Soury, et al. 2009).

Migneault, et al. 2009, conducted a comparison between extrusion and injection molding for producing WPC, common steps found in both include melting, shaping, and cooling; in addition, they both use screws to convey, pump, and blend the mixed component. However, they added that process parameters such as residence time, temperature, pressure, shear rate, shear stress, and cooling rate are different.

Moreover, they concluded that pressure and shearing in injection molding are higher than en extrusion regardless the process parameters mentioned (Migneault, et al. 2009). Stark, et al. 2004, compared WPC samples; composed from 50% wood flour and HDPE, obtained from extrusion and injection molding and found that they gave the same flexural modulus; however, the flexural strength and density of injected parts were higher. The authors justified that this could be resulted from the better interfacial contact in injection molding between wood and polymer; totally encapsulated wood particles within polymer matrix, resulting in higher density and therefore more strength (Stark, Matuana and Clemons 2004). However, Bledzki and Faruk 2004, have shown that WPC made from 30% hardwood particles and polyethylene resulted in similar specific bending modulus of elasticity and density for both injection molding and extrusion techniques. Conversely, injection molded WPC have shown higher specific tensile strength (Bledzki and Faruk 2004).

Concerning physical properties, Clemons and Ibach 2004, conducted sorption behavior comparison for WPC; composed from 50% of 40-mesh pine flour and HDPE, and concluded that water-soaked extruded samples absorbed and swelled more water than injection molded samples (Clemons and Ibach 2004)

2.4.1. WPC reprocessing

Another important point that should be addressed is the reprocessing of the wood plastic composite itself. Although literature did not emphasize much on this point, however; Beg and Pickering, 2008, have shown that mechanical properties of WPC samples composed from 50% fiber; reprocessed two times, increased respectively by 13.5% and 33% for tensile strength and yield modulus. In addition, after the second reprocessing time, the properties decreased till reaching the 8th reprocessing when

tensile strength and yield modulus; of a WPC with 40% fiber, was reduced to 25% and

16% respectively (Beg and Pickering 2008).

CHAPTER 3 METHODOLOGY

Conducting an experiment is a work associated with many variables. Therefore, random patterns for doing the work will make it impossible to cover all variables and guarantee work and outcomes consistency. As a consequence, the need for an organized framework for doing experiments was a necessity. One factor at a time was the old trend; used as an experimental framework methodology, that many people had relied on in the past. It consists mainly of controlling all factors; fixing their values, and varying one factor at a time. However, this method is considered invalid as it does not consider the interactions between other variables; in addition, it needs a huge number of experiments to be performed which is a time and money waste (Montgomery and Runger 2003). Unfortunately, many practitioners are still using this method which doesn't assure by any means obtaining valid results (Montgomery and Runger 2003). Accordingly, an alternative method was needed to do an organized framework with interaction consideration and minimum possible number of experiments which will draw valid conclusions. Design of experiments (DOE) is the alternative method that was used in this work. DOE saves time, money, and effort by providing valid results with minimum number of experiments. DOE has a crucial role in engineering design, development, and improvement of manufacturing processes. Developed products and processes from designed experiments have led to better performance, higher reliability, and lower overall costs. In addition, Designed experiments are a reason for lead time reduction for engineering design and development activities (Montgomery and Runger 2003).

In an experiment that is built from the beginning using DOE, purposeful changes could be done in the controllable variables of the system or process. In addition, observation of the resulting system output data and decisions could be made about which variables are responsible for the observed changes in output performance (Montgomery and Runger 2003). When designing experiments, there are responses, controllable factors, and uncontrollable factors. Controllable factors are the parameters set to predefined levels. Uncontrollable factors are the ones that cannot be controlled in actual operations, but may be controlled during experimentation i.e. weather conditions, natural disasters… Responses are the output results obtained from experiments (Montgomery and Runger 2003).

Typically, DOE has two main tasks. The first is setting efficient experimental design points i.e. building an efficient design with minimum number of distinct runs or experiments. Distinct runs are the most important runs' settings of the experiment at which response behavior is best tracked; therefore valid conclusion could be drawn and a valid model of the response could be obtained. The second task is analyzing the factors involved within the experiments and showing the most important ones i.e. knowing the most affecting factors on the response. As an example; if pressure, temperature, and cooling rate were factors involved in a designed experiment considered with measuring the strength of a material, The results of the DOE would show which factor of the three or their interactions is affecting the response (strength) more.

3.1. Design Of Experiments (DOE)

3.1.1. DOE types

DOE has three main branches; experiments with dependent, independent, and hybrid factors.

Experiments with dependent factors are concerned with factors having certain levels that are interacting in an experiment and are independent each other affecting the response in a certain way. Several experimental designs are available in this case including factorial, Box-Wilson Central Composite, and Orthogonal designs. Factorial design is a type of design where runs are performed at all possible combination of factors' levels are examined. The number of runs is counted by this formula: $no. of\ levels^{no.of\ variables}$ (Montgomery and Runger 2003). For example, if the number of levels is 2 and the variables 4, the total number of runs required is 16. Box-Wilson Central Composite Design is known as central composite design. It contains an imbedded factorial design in addition to center points that is increased with a group of star points. The main reason behind adding star points to allow curvature estimation (Montgomery and Runger 2003). Orthogonal design is distinguished with its ease of use for allocating level and its efficiency. In the orthogonal design, factors' settings involve allocating levels by using an orthogonal array designed by Taguchi. It is based on a standard table containing number of levels in columns and number of factors in rows arranged in a way defined by Taguchi to get the number of experiments and combinations; factors with required level in each particular experiment, minimizing the number of experiments needed when comparing to full factorial design.

Experiments with hybrid factors are a combination between independent and dependent factors' experiments (Myers and Montgomery 2002). It is a mixture design; explained in the next section, extended with some independent factors.

Design with mixtures is a type of experimental design where all factors are dependent on each other (Myers and Montgomery 2002). In other words, factors don't have levels rather they have percentages in a mixture, which is the case in this work (explained in details in Experiments with mixtures).

3.1.2. DOE steps

The next steps are required to perform an experimental design:

- Problem statement
- Choice of factors and their corresponding levels or percentages and constraints.
- Choice of response variable(s)
- Baseline experimentation (phase I experiments)
- Choice of experimental design
- Performing the experiment (phase II experiments)
- Statistical analysis
- Conclusions and recommendations

The problem statement in this work is to obtain a WPC product from recycled materials with the best possible physical and mechanical properties using simple and effective technology. These steps were applied in this work and would be presented all along this thesis; where a constrained mixture design was used using D-optimality criterion to obtain the best possible model for the flexural strength and modulus responses.

3.2. Pilot Experimentations (Baseline Or Phase I Trials)

These experiments are called the pilot, baseline, or phase I experiments (Elsayed 2009). It is a phase consisting of running initial experiments to get more experience and knowledge about the factors included, determine the important ones to be investigated further, and exclude the unimportant i.e. getting the feeling of interconnected components. In addition, its outcomes give the necessary data to set bounds and constraints on factors involved (see" Factors involved in this work" section). As well, it is the key which gives the guidelines for the necessary manufacturing techniques; as it was shown in this work. Moreover; based on results obtained from pilot experiments, sequential modifications were done leading to the final adopted manufacturing technology. These experiments were run in random patterns to estimate the general behavior of factors; however, extreme settings of factors should be experimented to be able to add boundaries and restrictions.

3.2.1. Prerequisite Stage

This stage consisted of making assumptions to start the experiments with. It was based on literature review and a local market survey. The first experimental settings were built based on this stage; where literature was the first key giving the way for a manufacturing technology. Two methods for the manufacturing of wood plastic composites were suggested: injection molding method and extrusion and compression molding method (Klyosov 2007). Then, a local market survey was conducted to check the availability of machines needed for these two technologies.

It showed high availability of extrusion technologies; manufactured locally, and relative cheap cost. Conversely, injection molding machines mainly are not manufactured locally, less available and more expensive than extruders. Therefore, the decision was made to use the extrusion method.

3.2.2. Stage 1

The main target of this stage was to check the adequacy of the chosen technology via its applicability using virgin plastic and wood waste. Virgin plastic was used to block any effect that could be accompanied with plastic waste. As a result, the three manufacturing steps; extrusion, heating, and compression have proven efficiency and gave a feasible product.

3.2.3. Stage 2

Plastic waste was utilized instead of virgin within experimentations. Several problems appeared in this stage regarding wood and plastic wastes. As a consequence, the product which was obtained suffered in many cases from a non homogenous grains' distribution in the final product i.e. plastic and wood weren't distributed evenly in the product. Therefore; it was suspected to obtain non consistent properties if large sheets were decided to be produced. This problem was avoided in stage 3 when meshing and shredding process were added.

The main problem accompanied with plastic waste was the formation of volatile organic compounds which affected the product negatively and called for adding bounds and constraints for this factor (see "Factors involved in this work" section).

On the other hand, wood waste has caused problems concerning the formation of water bubbles; due to its hydrophilic nature, and the uneven grain distribution within the product which allowed the formation of water bubbles and voids within the wood plastic matrix.

3.2.4. Stage 3

This stage was the last one before going to the main experimental design adopted in this work (phase II experiments). It included many modifications that started by the introduction of shredding and meshing operations to avoid non homogenous products obtained in stage 2. 8 different sizes of sieves were adopted ranging from 400 to 1300 Micrometers to mesh the wood; where only 2 sizes; 500 and 1180 Micrometers, were decided to be used in phase II as they gave the highest flexural strength and modulus properties compared to the other 6 sieves. The 500 Micrometers' sieve was selected from a range of sizes that is commonly used in literature for the production of WPC; ranging from 50 to 700 Micrometers (Klyosov 2007), and the 1180's one was selected based on a claim that increasing in particle size would ameliorate properties (Klyosov 2007).

The second important modification was the need to do something concerning the wood humidity and tendency to absorb water; hydrophilic nature. Drying wood before usage was the first step to minimize wood water content and adding talc was the second (for more details see "Factors involved in this work" section).

On the other hand, decisions concerned with process variables (furnaces' temperatures and extruder's temperatures and speed) were taken in this stage also.

Mainly, the limits' selection of all these variables was based on the product obtained; whereas, burned products will call for decreasing temperature; for example. Typically, I was required to obtain a well cooked product yet not burned with the minimum possible time. Therefore, Furnaces' temperature; used for wood drying and forming the paste, were set based on these main criteria (for more details see chapter 4). Extruder's temperature was decided to be set at a specific degree to avoid solidification or overheating of the product (for more details see chapter 4). Extruder's speed was set at 19 RPM; because when the speed was higher than 20 RPM, the product obtained wasn't coherent and well mixed. In other words, the time wasn't sufficient to merge plastic and wood where the plastic wasn't well heated. In contrast, when the speed was less than 18 RPM the product obtained was overheated; therefore the plastic liquidified and stuck within the extruder.

Based on the previous justifications, levels of process variable were decided not to be included within phase II experimentations.

3.3. Experiments With Mixtures

Experiments with mixture are adopted when factors involved are not independent. The factors represent the ingredients of a mixture that add up to a complete product (Myers and Montgomery 2002). In other words, the product obtained is sum of all percentages of factors or ingredients; by weight or other criteria. In this type of design, the response is a function of ingredients' percentages (Design-Expert 2010).

The products obtained from this type of experimental design are described as complete blends, binary blends, and pure blends (Myers and Montgomery 2002).

A complete blend is a product which is made up of 3 or more ingredients. For example, X1+X2+X3+…Xn = 1, Where Xi for i=1, 2, 3…n are the ingredients' percentages of the mixtures of a total number of n ingredients. A *binary blend is* a product which is made up of 2 ingredients. For example, X1+X2= 1, Where Xi for i=1 and 2 are the ingredients percentages of the mixtures of a total number of 2 ingredients. *A pure* blend is a product which is made up of one ingredient, X = 1.

However, sometimes blends couldn't be described in this simple way when an experimenter decided; for example, to use factors that are obtained from fractions of other factors within the same experiment for specific experimental purposes; which happens typically in chemical blends (Myers and Montgomery 2002). In addition, it is common that the range of a factor used within experimentation couldn't be fully utilized due to technical or other restrictions i.e. the factor's range from 0% to 100% couldn't be fully used; for example, it could be used from 0% to 70% only. These bounds on the factors could be lower, upper (as the previous example), or both. To illustrate, it is said that factor or ingredient A; for example, shouldn't be less than 10%; lower bound, and shouldn't be more than 70%; upper bound. Also, there could be constraints on factors; such as the sum of two from three factors, for example, shouldn't be less than 60%. In these cases, pseudocomponents are used to rescale the real components to be able to describe design points (Myers and Montgomery 2002).

3.3.1. Factors involved in this work: description, upper, lower bounds, and constraints

Four factors were used to produce WPC: plastic, talc, and wood waste with size of up to 1.18mm and 0.5mm. For illustration, these factors will be described respectively as X1, X2, X3, and X4. Table 3-1 presents all the bounds and constraints in this work.

Table 3-1: upper, lower bounds, and constraints of factors

Upper and lower bounds	Constraints
40 ≤X1≤ 70	15≤ X3+X4≥50
0 ≤X2≤ 35	
0≤ X3≤ 50	X1-X3-X4≥0
0≤ X4≥ 50	

X1 is the percentage of plastic within the mix. It is a mix of HDPE and LDPE with ratios of 25% to75% respectively. It is a waste product obtained from municipal waste and no virgin materials were used. It is composed of shredded plastic waste obtained mainly from garbage plastic bags which are highly contaminated. The utilization of this type of plastic would save the environment as these bags are non biodegradable materials which are mostly thrown away in a dumpsite; therefore, it is a costless unutilized resource calling for investment. In addition, the highest percentage of WPC produced commercially worldwide is based on Polyethylene (Klyosov 2007). X1 was utilized in this work with percentages varying from 40% to 70% (table 3-1). The higher bound of X1 was decided based on pilot experimentations. When the percentage exceeded 75% then, volatile organic compounds; resulted from melted plastic, were produced during extrusion in addition to unknown gases which could be resulted from

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the contaminations in plastic (bearing in mind it is a product obtained from contaminated plastic bags). The production of these gases caused a continuous blowing of the extruder; which called for shutting down several experiments for safety reasons and to avoid possible hazard. Therefore, it was decided afterwards not to increase the amount of plastic more than 70%. On the other hand, the adoption of the lower bound was based on the non-coherent burned product obtained when the percentage of plastic was 35% or less during pilot experimentations. This could be due to the high wood (filler) amount not meeting enough plastic to be merged in a matrix.

X2 is the percentage of talc $(Mg_3Si_4O_{10}(OH)_2)$ within the mix. It is a part of the phyllosilicate minerals which is used as a mineral additive to this mix. It is characterized with its ability to absorb water; therefore, minimize the humidity of wood; which is characterized with its hydrophilic nature, as a consequence enhancing mechanical properties of WPC (Sun-Young Lee 2008). In addition, talc has a natural affinity to oil; therefore, it works as good filler for hydrophobic plastic (Klyosov 2007). Before using talc; within pilot experimentations, water formation within the product was a major problem especially with mixtures containing high filler content; 45% or more. These products failed easily with minor load application when tested; flexural strength was 2 Mpa or less and the modulus didn't exceed 250 Mpa. However, results after using talc were far higher and the effectiveness of its usage was proven at the end of this work (see chapter 6). The upper bound was based on literature recommendations; where talc gave highest flexural strength an modulus at 27% talc (Noel and Clark 2005) & (Klyosov 2007). Therefore, it was decided to use an upper bound of 35%.

X3 and **X4** are the percentage of meshed wood waste with size of up to 1.18mm and 0.5mm. X3 and X4 are sawdust wastes obtained from wood workshops; which are typically thrown away in dumpsites. The main problem of wood waste lies in its hydrophilic nature; unlike the contamination issue of plastic where wood is obtained from wood workshops. It absorbs water and humidity in an immense way. Two actions were taken to solve this problem; drying wood and adding talc. The upper bound of X3 and X4 was decided not to increase 50% as the plastic should be at least 40% to produce coherent product and 10% would be considered as a basic percentage of talc to get rid of wood water content. However, runs with zero talc percentage were also conducted to measure its effect.

The first constraint tells that the total wood wastes percentage shouldn't be less than 15% and more than 50%.The lower bound was needed because there should be a minimum amount of wood waste in the product as talc was mainly added to enhance a problem correlated with wood. The upper bound was added to make the total wood wastes acting in consistency X3 and X4. In other words, making the total of wood waste less than or equal 50% in all cases. The second constraint says that the plastic percentage should be more than the total wood wastes percentage. It is an assurance condition for avoiding a case such as 40% plastic, 50% wood, 10% talc; where non-coherent product was suspected in this mix (containing high filler content) as explained in the plastic section.

3.4. Constrained Mixture Designs

When the choice is made to start with the mixture design, various selections are available. Simplex designs are commonly adopted whenever the components form a simplex region i.e. the factors' ranges are equal (Design-Expert 2010). In other words, when there aren't upper and lower bounds or constraints. However, the case in this work is violating this condition; therefore, non-simplex designs would be adequate. Generally, when lower and upper constraints are active in a mixture experiment, the feasible design space is an irregular hyperpolytope (Myers and Montgomery 2002). Extreme vertices, D-optimal, and distance based are known types of hyperpolytope designs. D-optimal design was used in this work as it will be explained further in this section. The method of work of these designs is based on a selection of some candidate points from the constrained region; due to the existence of limits and/or constraints, following the algorithm of the chosen design. The candidate points are the total set of feasible points from which the actual design points can be selected. For any design space, an infinite number of individual points are available within that space; yet the experimental design limits it to specific types of points. These points are called with specific names such as: vertices, center points, axial check blends, centroids, etc. As an example, if a quadratic model with 5 factors was decided to be performed, a design space having 245 potential points would be used. This set of 245 points is the "candidate list of points" or the "candidate points" from which 21 only of these points are actually chosen to estimate the model coefficients.

Generally, there are two main steps required to do the design. The first step is considered with obtaining a set of reasonable candidate points to be used in the

selection of design points. It should be based on the on the model order wished to be used. It is recommended to use one of the following models based on practice (Myers and Montgomery 2002): Linear, quadratic, cubic or special cubic model. However, other hybrid models; such as the partial quadratic, could be a better estimator in some cases as it will be shown in this work. In *Linear models*, the candidate points would include vertices, overall centroid, edge of centers, and axial points located halfway between overall centroid and vertices. On the other hand, *Quadratic models* would include vertices, overall centroid, edge of centers, axial points, and constraint plane centroid. In addition, the candidate points of *Cubic or special cubic model* would include vertices, overall centroid, axial points, constraint plane centroid, and thirds of edges (see Appendix 11). Quadratic model was used as a starting model in this work; as it is a mid-way between linear and higher degree models allowing the formation of several other models without wasting much runs. Moreover, Quadratic models were recommended as a starting initial model when the case is not simplex (Myers and Montgomery 2002) & (Design-Expert 2010). However, the last model obtained that gave the best fit; for the flexural strength and modulus, was a partial quadratic model (a hybrid of linear and quadratic) (see chapter 6).

The second step is the usage of a convenient method to select and identify points and their coordinates in the constrained design space.

Typically, there are various designs; each having its algorithm such as: Distance based, extreme vertices, CONAEV, D-optimal, and others (Myers and Montgomery 2002). **Extreme vertices design** is formed by the combination of upper and lower bounds constraints. Set of points within the constrained region were suggested by McLean

and Anderson to be used as the basis for the design including overall centroid, points at the center of the edges, faces. However, Myers and Montgomery recommended the usage of one of the two other methods as they are most commonly used in practice. Therefore, this choice was excluded from the selection in this work. **Distance based design** is based on the technique of uniform spreading of design points over the feasible region. The algorithm that is utilized; for points selection, is based on a simple criterion of putting points to cover the boundary of the region then adding interior points only when these points are farther from the points already in the design. In other words, it is a point's choice using coordinate exchange to achieve the maximum spread throughout the design region. However the selected points using this technique might not be sufficient for model coefficients estimation, nor an estimate of pure error or lack of fit could be provided (Design-Expert 2010). Therefore, this type was excluded from the selection of this work also. **D-optimal design** is called "D-optimal design" or sometimes other alphabetic letters are used based on the optimality criteria. However, this design is used to select points for any mixture design in a constraint region (Myers and Montgomery 2002). This type of design needs a set of reasonable candidate points from which it chooses the design points.

It works mainly by the selection of a set of points minimizing the variances of model regression coefficients by adopting the technique of loading up vertices points. In addition, it should be noted that when the number of variables increases the likelihood of choosing interior points in a design with a reasonable number decreases. Therefore, the tendency to use designs other than the D-optimal; such as the distance based, is not recommended. Specifically, when the number of variables is four or

more, the usage of D-optimal criterion is recommended. This is due to the fact that distance criterion lean to choose interior points in a feasible region and thus the variances of the model regression coefficient are not minimized; for that reason experimenters are more oriented to use D-optimality because the concept of minimizing variances is pleasing (Myers and Montgomery 2002). I.e. for the same region, D-optimality would place 2 internal points and distance criterion 4. In addition, distance criterion is not recommended for physical experiments; which is the case in this work (Design-Expert 2010). Moreover, D-optimality is powerful tool in the identification of the most crucial variables. Therefore; for all the precedent reasons and the fact we are dealing in this work with four variables in a physical experiment, D-optimal design was adopted in this work. However, a common problem with D-optimal designs that it depends heavily on the number of runs (Myers and Montgomery 2002). In other words, if different number of runs were adopted in several trials the results will differ much.

Yet, this problem was solved in this work by relying on Myers and Montgomery recommendations of generating several D-optimal designs with varying the number of replicates and the total number of runs in each trial to reach an appropriate fit and minimum error. Therefore, 2 main actions were taken in this work to accommodate this problem:

1. An algorithm for navigating the points within the design space was used for the sake of performing several trials in a consistent way and obtaining the best possible model with the minimum possible number of trials. The algorithm is represented in figure 1- chapter 6.

2. A design with excessive number of runs was built; 51 runs and 10 replications (see Appendix 2), where the quadratic model used to fit data required only 21 points; 6 for model fit, 5 for the lack of fit, and 5 for error estimation (Design-Expert 2010) & (Montgomery and Runger 2003).

Another reason for adding additional runs (51 runs and 10 replications – see Appendix 2) at the first design is the fear of requiring a higher order model; if the quadratic was inadequate. Moreover, getting an estimate of error; this is why replications were added. Typically, a quadratic model needs only 21 runs; therefore the additional runs in this work gave a good space for navigating the model using the algorithm guidelines (see figure 1- chapter 6). Generally, it is recommended to add 8 to 10 additional runs than the minimum required to fit the model (Myers and Montgomery 2002).

Half of these runs (4 to 5) are to be replicates of some points in the design; for the estimation of the error and the other half new distinct points should be added to investigate the lack of fit for the model. The number of replicates is related with the design adopted. In D-optimal quadratic models, replicates are usually four to five points (Myers and Montgomery 2002). Design-Expert uses in this case; as a default, 5 replicates in the design. It was justified by providing 4 degrees of freedom to estimate pure error and generally improve the quality of the design (Design-Expert 2010). Therefore, it was decided to add ten replicates; to give a larger space, were decided to be run in the initial design. The replicates were automatically generated by the Design-Expert and due to the tendency of D-optimal criterion to load up the vertices of the region with design points; most of replicates were vertices points.

3.5. Software Used

Design-Expert 8.0.1 is the software; offered by Stat-Ease Inc, which was used for the design of experiments during this work and afterwards doing all the necessary analysis. All the analysis output of flexural strength and modulus obtained in this work from Design-Expert is presented and discussed in chapter 6. It is one of the best specialized software in experimental design as it is used by many specialized researchers in this area and many books such as Myers and Montgomery in Response Surface Methodology book. The version 8.0.1 utilized in this work is produced in 2010.

CHAPTER 4 MANUFACTURING PROCESSES

4.1. Processes In Brief

The manufacturing processes of the WPC were performed at AUC Environmental Management Labs. All the processes have gone through several pilot experimentations till reaching the procedures described above (for more details see chapter 3).

The processes start by meshing the wood waste into 2 predetermined grades using sieves of sizes giving wood waste particles up to 0.5 mm and 1.18 mm to obtain a homogenous saw dust material. Based on pilot experimentations, these two sizes were used as they gave highest flexural properties (strength and modulus) of the final product in comparison with several sieves of different sizes utilized during trials. The meshed wood waste is then taken to be dried in a furnace for 4 hours to eliminate the moisture within wood waste particles up to almost 100% (see table 4-1). The furnace temperature is set at 115 C. This temperature was decided not to be increased more than 115 C due to safety reasons as to avoid wood waste burning. After drying takes place, the wood waste is then mixed with plastic and talc (if any) in a jar (container) using a mixer and then the mix are fed into the single screw extruder. The talc is added as a mineral additive to enhance the properties of the final product. Two electric heaters are used at the beginning and end of the extruder. Temperatures were set at 120 C for the first one and 150 C for the second one. The reason behind the temperature's selection will be explained in details in the "Processes in details" section. The resulted extrudates are left to be cooled at room temperature then taken

to the shredder to form small particles with identical dimensions which will make the paste formed from the last process more homogenous. During the shredding operation the furnace was turned on for about half an hour to warm up at 140 C; the temperature allowing to form a homogenous mix of the paste without burning. Then, the shredded particles are fed into the furnace to form a paste which is formed after about 15 min. Therefore, it is taken to the hydraulic compression molding machine to be pressed. A die with specific dimensions is used to satisfy the requirement of testing; as will be explained further in Chapter 5. Finally, trimming and cutting processes into the exact specified dimensions are done to make the product ready for testing in accordance with the requirements of the testing standards (See chapter 5).

4.2. Process Flow Chart

Figure 4-1 : Illustrative process flow chart of WPC manufacturing

4.3. Processes In Details

4.3.1. Wood waste meshing

The wood waste utilized is formed from sawdust with fine particle sizes. This type of wood waste generally is a by-product of wood sawing which ranges from 20 to 5000 µm (Klyosov 2007). Based on literature, the common adopted sizes of wood utilized for the production of WPC range from 50 to 700 Micrometers; where increasing particle size results in better flow of molten composite, lower mold shrinkage, and higher flexural modulus (Klyosov 2007). In other words, better properties are obtained when the size approaches the 700 Micrometers. Based on this claim, it was decided; during pilot experimentations, to test 8 different sieves ranging from 300 to 1300 Micrometers for the sake of obtaining the best possible accepted product. Increasing the size more than 700 Micrometers intended to experiment the claim mentioned above of "the increase in size would ameliorate properties". As a result, two sieves were decided to be used with sizes of 500 Micrometers (0.5mm) and 1180 Micrometers (1.18mm) as they gave higher flexural strength and modulus in comparison with the other 6. Moreover, it was decided to use various mixtures of these two sizes during main experiments; as it was suspected that a mixture of two sizes may ameliorate properties.

4.3.2. Wood waste drying

The dryer used was set at 115 C to avoid wood waste burning. The meshed wood waste is left for 4 hours in the dryer to get rid of the moisture (see figure 4-2). It was assured that the moisture was totally eliminated through a test that was done.

The test consisted of taking samples of the 2 wood waste types; sizes of up to 0.5mm and 1.18mm, utilized within experiments and weighs it. Then, it was left in the dyer for 2 hours then weighed.

Each hour after the second hour, it was weighed. At the 5th and 6th hour the weight was not changed for the two types (see table 4-1). Therefore, it was concluded that 4 hours was sufficient to dry the meshed wood waste. Table 4-1 contains the weights of a sample with a size up to 1.18 mm with the corresponding hours.

Table 4-1 : Wood waste drying time

Time	Hours in furnace	Weight (grams) – measured	Weight (grams) – Calibrated	Percentage of water lost (drying)
9:00 AM	0	25.6	25.65	0
11:00 AM	2	24.7	24.75	3.51
12:00 PM	3	24.1	24.15	5.85
1:00 PM	4	23.8	23.85	7.02
2:00 PM	5	23.8	23.85	7.02
3:00 PM	6	23.8	23.85	7.02

The experiment was conducted for two wood waste samples; utilized during the manufacturing process, with sizes of up to 0.5 mm and 1.18 mm. It was repeated 3 times to guarantee the results.

Figure 4-2: Putting the wood waste to be dried

The scale used (see figure 4-3) was calibrated using linear regression method based on standard weights brought from the polymers lab at the American University in Cairo (see figure 4-4). The regression was performed based on the data shown in Table 4-2 obtained from the scale.

Weight (grams) –Standard	Weight (grams) – measured
100	99.9
50	50
20	19.9
10	9.9
5	5
2	2

Table 4-2: Scale measurement of standards

Regression equation obtained for calibration is as follow: **x=(y+0.034)/0.999**. Where y is the dependent variable consisting of the measured weight in grams and x is the

independent variable of the calibrated weight. The equation was applied to the weights measured of the wood waste in table 4-1 and calibrated readings obtained.

Figure 4-3: The set of standard weights used for calibration

Figure 4-4: The scale used

4.3.3. Extruding

Before feeding the extruder (single screw extruder), the plastic is mixed; using a mixer, with wood waste and talc (if any). The plastic used; which is composed of a mix of HDPE and LDPE with 25 % to 75 % respectively, has the shape of small particles. This mix is composed of shredded plastic waste obtained mainly from garbage plastic bags which are highly contaminated. The talc is used as a mineral additive; to enhance mechanical properties, with percentages varying from 0 to 30 percent by weight of the total. The mix is then being fed into the hopper of the extruder and the process starts (see figure 4-5). Setting the two heaters at 120 C; for the first one, and 150 C; for the second, the extrudates are produced and the sample is accomplished and extrudates obtained within about 17 min for a 1.5 kg used (see figure 4-6 and 4-7). Intuitively, a warm up period for the heaters of about an hour was a prerequisite. The temperatures' settings were dependent mainly on the plastic utilized as it has major effects on the process and therefore the final product obtained. These effects were discovered during pilot experimentations; which were reflected when the

temperature range of the extruder was increased to 135 C (1st one) and 165 C (2nd one), the melt was overheated, liquidified, then stuck around the screw as it turned with its rotation without flowing. This caused the process to stop as it prevented the flow to continue and jammed the whole process. In other instances, when the temperature was dropped to 105 C (1st one) and 135 C (2nd one), the melt was solidified causing the blockage of the flow. Therefore the settings of 120 C ± 5 and 150 C ± 5 were applied.

The difference in temperature is due to start with a primary heating then increase it to the final one as not to cause a sudden increase in temperature within the mix and therefore caused an incremental pressure resulted in exploding the mix from extruder outlet rather than flowing. Other effects accompanied with the contaminated plastic used were explained further in chapter 3.

Figure 4-5: Feeding the extruder with the mixture

Figure 4-6 The hot extrudates coming out of the extruder

Figure 4-7: The final shape of the extrudate

4.3.4. Shreddering

The extrudates (see figure 4-7) are crushed in the shredder (see figure 4-8) forming small particles with identical sizes to be fed into the furnace. The shredding operation was important as it avoided bad distribution of the mix during furnace heating within experimentation. As this process at first; during the pilot trials, was done without shredding which resulted in several cases of non homogenous final product. The main reason behind this that the extrudates have different sizes and the material's concentration within each extrudate wasn't distributed the same. Therefore, it was decided to use a shredder.

Figure 4-8: The shredder adopted in crushing the extrudates

4.3.5. Furnace

The furnace works for about 15 min at 140 C to form a paste which is then taken to be pressed (see figure 4-9). The temperature was decided based on pilot trials aimed at forming a homogenous paste without burning.

Figure 4-9: The furnace used for heating the shredded particles to form a paste

4.3.6. Compression molding

The hydraulic compression molding machine used consists of a hydraulic press with parallel platens that apply the pressure (see figure 4-11). A pressure of 40 bars is applied. The steel die used is a custom made one with dimensions of 42 * 12 * 12 cm (see figure 4-10) to accommodate the size required for the ASTM D 4761 test of flexural test (see chapter 5 for more details). A sample obtained after pressing is show in figure 4-12.
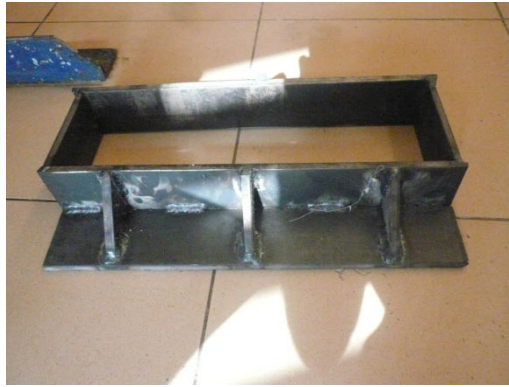
Figure 4-10: The custom made die

The required thickness is obtained via right weight selection for the mix and adjusted using thickness cutter (Rabou).

Figure 4-11: The hydraulic press used

Figure 4-12: The sample obtained after pressing

4.3.7. Trimming

A trimming process (see figure 4-13) is done for the sample giving final dimensions of 40 * 10 cm to be ready for the testing in accordance with the standard requirements (see chapter 5).

4.3.8. Thickness cutter (Rabou)

The thickness is corrected via cutting the sample obtained to the 1 cm thickness required by the standard (see chapter 5). It was done at a workshop in Helwan governorate.

Figure 4-13: The trimmer used to cut edges

CHAPTER 5 TESTING

Two main important areas of testing were performed: mechanical and physical properties of the WPC obtained product. It was decided to test the flexural characteristics in the mechanical properties testing stage as they are the ultimate range that the product can sustain under severe usage conditions. In addition, they are of a major importance for many international codes related to housing and construction requirements such as the "Acceptance criteria for deck board span ratings and guardrail systems" and the " Building Officials and Code Administrators International, 1999 (BOCA) National Building Code" (Klyosov 2007).

The physical property chosen was the water absorption and thickness swelling testing. The selection here was based on the nature of the WPC product; as it contains a considerable percentage of plastic which is characterized by its hydrophobic nature towards water. Therefore, the product was estimated to better perform than alternative wood products in areas where subjected to water. Accordingly, this test was decided to be performed.

5.1. Flexural Test

Flexural test is one of the mechanical tests performed on products. Other mechanical tests could include shear, tensile, impact, creep, and other tests. Flexural test is frequently done on relatively flexible materials such as polymers, wood and composites (Instron 2010). Flexural test is a method to measure the material behavior when subjected to simple beam loading.

It is known for some materials as the transverse beam test. The specimen in this test is supported with 2 ends and a load is to be applied with a known feed rate. Maximum fiber stress and strain are calculated and plotted in a stress-strain diagram. As a result, the flexural strength and modulus are obtained. ((Klyosov 2007)& (Instron 2010)).

Flexural strength and modulus particularly are the important characters to be tested (Klyosov 2007). In addition, they are of a major importance for the International code council- evaluation services (ICC-ES) acceptance criteria and other codes depending on the type of application such as the AASHTO LRFD Code used for plank decks ((Klyosov 2007)& (Nowak and Eamon 2008)).

Flexural strength or modulus of rupture is defined as the maximum fiber stress that could be developed in a tested sample just before cracking or breaking (Instron 2010). The term fiber here has nothing to do with actual fiber; however, it means the material near the sample surface where the maximum strains happen during loading (Klyosov 2007). Sometimes, when the elasticity of the material is so high that it would not crack, the flexural yield strength is reported instead of the flexural strength (Instron 2010). Typically, it is reported when a maximum fiber strain of 5% is reached ((D6272 2002)& (D790 2008)).

Flexural modulus is defined simply as the stress to strain ratio in the flexural deformation. It is calculated; based on the apparatus used definition, from the stress to deflection curve slope where the curve has no linear region. A line; that is drawn from the origin to the specified point, is fitted to the curve to determine slope (Instron 2010). Klyosov (2007) defined its calculation method in other words, as it is a tangent drawn to the steepest initial straight line portion of the load deflection curve. He also

mentioned that it is basically a load at which the specimen deflects by 1 inch. (Klyosov 2007). The flexural test was conducted in this work in accordance with the ASTM D 4761-05. It intended to get the values of the flexural strength and the flexural modulus.

5.1.1. Types of flexural test

The flexural testing is done typically in 4 methods or types: 3-point loading, 1/3-point loading (4-point load), 1/4-point loading (4-point load), or uniform loading (Klyosov 2007). In all cases, the specimen is supported with 2 edges and a load is to be applied with a known feed rate. However, the difference is mainly based on the number of load noses applied; that could be one or two, and the distance between these noses; the maximum bending moment. In the case of 3-point loading, the specimen is loaded with one nose in the middle of the specimen support span; the distance between the 2 support edges. Therefore, the maximum axial fiber stress is positioned directly under the loading nose. While in the 4-point loading, the maximum axial fiber stress is uniformly distributed between the loading noses. The 1/3 (see figure 5-1) and 1/4 (see figure 5-2) points tell that each of the two loads is applied at one third or one fourth of support span from the respective ends (D6272 2002).
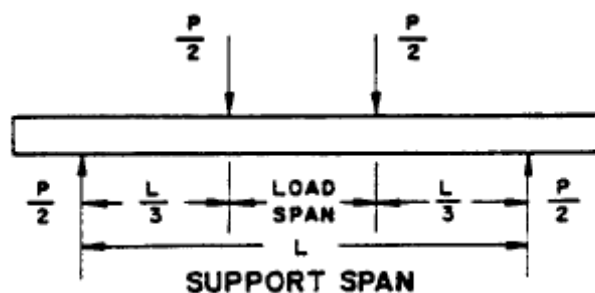
Figure 5-1: /3-point loading diagram (4-point load). ASTM D 6272-02 (D6272 2002)

The uniform distributed loading is performed using one of the 3-point or 4-point load. The uniform load is calculated using standard equations. (Klyosov 2007)

The method conducted in this work consisted of 1/3-point loading (4-point load). Referring to the catalogue of the testing apparatus used (Instron - Bluehill Lite), the wood and composites are commonly tested using 4 points loading flexural test (Instron 2010); because most of the applications are based on distributed loads all over the product. As for the usage of 1/3 point instead of 1/4, some sources such as the ASTM D7032 and ASTM D6109 recommended using 1/3 point loading instead of 1/4 (Klyosov 2007). On the other hand, uniformly distributed testing is not used commonly due to technical difficulties and it is rather calculated using standard equations (Klyosov 2007). So, it was decided to use the 1/3 point loading (4-point load) as it is also more gentle and realistic in terms of application.

5.1.2. Experimental settings

Basically, 1/3 point loading (4-point load) method was adopted in accordance with the requirements of ASTM D 4761-05; which also gave full details for the apparatus and fixtures settings. A graphical display for the test settings (in the standard) made it

easier to be applied. Accordingly, the experimental settings could be divided into three main parts:

i.   **the dimensions of the specimen:**

ASTM D 4761-05 defined the span to depth ratio required as 1:32; however, the standard mentioned that a change could be done to this ratio but should be documented. The span length chosen within the test was 32 cm and the specimen depth was 1 cm. Where the span is the greatest dimension perpendicular to the direction of the applied load; the length of the span is determined as the distance between the center lines of edges supporting the specimen (see figure 5-3). While the depth is the smallest dimension parallel to the direction of the applied load and perpendicular to the span (see figure 5-3) (D4761 2005). Due to this requirement, custom molds was made and utilized to get the required dimensions as mentioned in details in the manufacturing processes part in chapter 4.
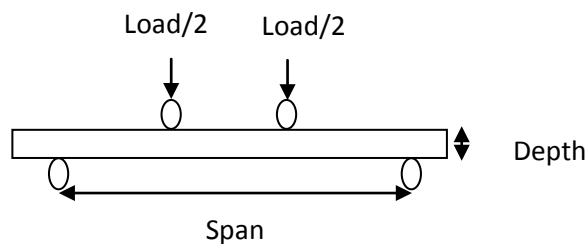
Figure 5-3: Graphical explanation of the span and depth

ii.   **the design of the fixture:**

A Special fixture was needed; in accordance with ASTM D4761, to perform the test. It should have 2 equal concentrated points of load application spaced equidistant between the supports. It was therefore "custom made" (see figure 5-4)

at AUC Workshops with the dimension between the centers of rollers; of 3cm diameter, of 16 cm. The ASTM D4761 stated that the utilized bearing plate should be as wide as the specimen is broad. In addition, its length should be no less than one half the specimen depths as shown in figure 5-4. The fixture was then attached to the load ram.

iii.   **other requirements:**

ASTM stated that the testing machine needs a reaction frame represented in the utilized supporting edges , a loading mechanism for applying load at specified rate which is the ram at 20mm/min as a feed rate, and a force measuring apparatus which is the computer attached to the machine online. Figure 5-4 and 5-5 present these components.
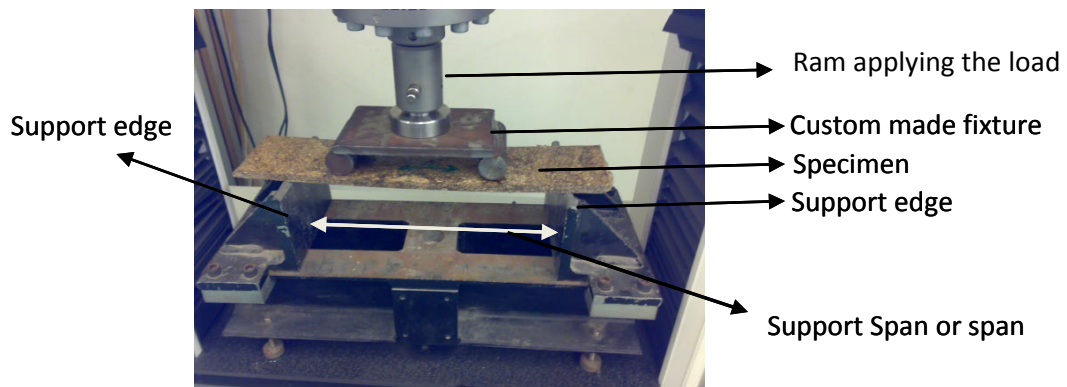
Figure 5-4 :The Setting of the flexural test
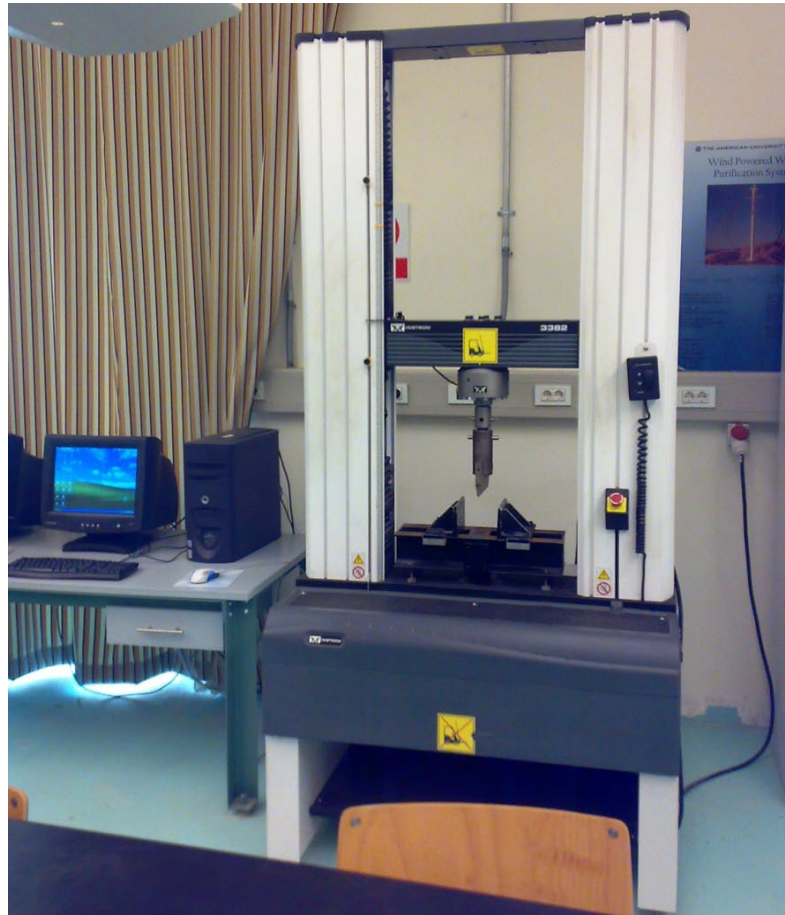
Figure 5-5: working station - AUC testing lab: test apparatus (Instron) and computer

5.1.3. Apparatus used: setup, software and outcome

i. Instron's model 3300:

Instron is a testing machine having properties of performing tensile (pull), compression (push), flex (bend), peel, and cyclic type of testing. It is attached to an online computer and via computer software all the required orders are taken (see figure 5-5). It is available at AUC Testing Labs and was utilized to perform the flexural test. The software utilized; Instron Bluehill Lite, is designed to run Instron's Model 3300 Material Testing Systems.

ii. Instron setup:

First of all, the two edges; consisted of the support span, were installed. Then, the custom made fixture was attached to the load ram and the specimen was concentrated between the support edges. The machine was afterwards turned on and the computer. The fixture was adjusted till touching the specimen surface. The next step was to adjust the computer software settings.

iii. Instron Bluehill Lite settings:

The computer settings required four major set of data regarding the specimen's dimensions, fixture type, feed rate control, and output type as follow:

1. The specimen's dimensions: the thickness and width were added; 1 cm and 10 cm.

2. The fixture type: 4 points fixture type was added. The support span and loading span (the distance between the centers of loading rollers) were added respectively, 32 cm and 16 cm.

3. The feed rate control: A 20 mm/min feed rate was added.

4. The output type: consists of adding the form of output needed. The measurements required (flexural modulus and strength), charts (stress-strain diagrams),and statistics needed for the obtained data (mean, mode, median, standard deviation...)

After adjusting the computer software settings, the machine was ordered to start the test and the ram moved downwards applying the load via the rollers of the loading fixture over the specimen. The automatic control and data recordings then started till the completion of the test and specimen's fixture reached.

iv. **Instron Bluehill Lite output:**

After the completion of the test, the ram returns to its initial positioning and a new specimen was added. The output of the test consisted of a stress strain diagram and a table containing the details of each specimen as shown in appendix 1 and 2.

5.2. Water Absorption And Thickness Swelling Testing

Water absorption and thickness swelling test was adopted as it is the best estimator of the performance of the WPC product when subjected to water. Especially when the intended use is outdoor application, the importance of this test increases more.

On the other hand, regarding the WPC high plastic content; 40 to 70 percent, it was estimated due to the plastic hydrophobic nature that the WPC product would perform better than alternative wood types in areas where subjected to water. Consequently, it was decided to do the water absorption test based on the ASTM D 1037. This standard was chosen as it is one of the tests used to evaluate the water absorption and thickness swelling of wood based fiber and particle panel material. In addition, it is one of the tests recommended by Klyosov to be applied for the WPC products when testing water absorption (Klyosov 2007).

5.2.1. Methods of the test

There are two methods to perform this test based on the ASTM D1037: Method A and B. Method A consists of expressing the water absorption and thickness swelling as a percent after two hours and then after 22 hours of submersion. Method B consists of

expressing the water absorption and thickness swelling as a percent only at one time after 24 hours of submersion. Based on Klyosov, method A is commonly not in use with WPC products therefore method B was adopted (Klyosov 2007).

5.2.2. Experimental settings

i. conditioning

The experiment started with the conditioning into the required humidity and temperature of the room in accordance with the ASTM D 1037 (Room relative humidity 65 plus or minus 5%, and temperature of 20 plus or minus 3 C). Potable water was used in the experiment. (D1037 2006)

ii. measurements

Two sets of measurements were required at first, initial and after the 24 hour submersion period. In addition, a measurement set was needed to get the humidity content; where the specimens were measured after being dried in an oven. The measured sets consisted of weight, length, width, and thickness. Weight was measured with an error of 0.1 g; based on the scale utilized, and thickness was measured at four points.

iii. procedures

- The specimens were submerged under 25 mm of potable water maintained at a temperature about 20 C. Specimens were placed horizontally and vertically based on an argument that was made stating it doesn't matter to put the specimens horizontally or vertically (Klyosov 2007).

- The specimens were then left to drain for about 10 min, and then the excess surface water was removed. The weight of the specimen was subsequently taken. Thickness, length, and width were afterwards determined. The first step of the test was accomplished here.

- The next step starts after drying the specimens in an oven at about 120 C to determine the moisture content. Specimens were left in the oven for a minimum of 6 hours and up to 10.5 hours; based on the drying rate of each specimen. A reading each hour after the fourth hour was taken; random specimens were weighted within the first three hours of drying and proven were not enough to dry specimens, till all specimens reach a steady state measurement. In other words, when the specimen reaches a weight that is unchangeable from a certain hour to the other.

5.2.3. Apparatus used

- Water Basins with large sizes to accommodate specimens were utilized. The basins were filled with water and the specimens submerged so that they are 25 mm under the water.

- Digital scale with a measurement error of plus and minus 0.1 g

- Vernier caliper; with a measurement error of plus and minus 0.1 mm, to measure the thickness, Length, and width.

5.2.4. Results

All results were recorded and expressed as percentages of water absorbed (weight, thickness, and volume increase) and percentage of water released (moisture calculations based on the weight decrease) during the test based on the two incoming equations:

- Percentage of water absorbed = ((W1-Wo)/Wo) *100 or ((T1-To)/To) *100 or ((V1-Vo)/Vo) *100

- Percentage of moisture = ((W2-Wo)/Wo) *100

Where:

Wo: the original weight of the specimen

W1: the weight of the specimen after submerged for 24 hour

To: the original thickness of the specimen

T1: the thickness of the specimen after submerged for 24 hour

Vo: the original volume of the specimen

V1: the volume of the specimen after submerged for 24 hour

W2: the weight of the specimen after dried in oven

The results of this test (Appendix 3) were then taken to be analyzed using Design Expert software (see Chapter 6)

CHAPTER 6 RESULTS AND ANALYSIS

After the completion of testing stage, all results were obtained and analysis stage started. The results of the flexural test; flexural strength and modulus, obtained from Instron Bluehill Lite output were taken to be analyzed using Stat-Ease Design-Expert 8.0.1 software. On the other hand, it wasn't found necessary to use Design-Expert for analyzing the output of water absorption and thickness swelling test due to the fact that the results showed minor absorption and dimensions' variability which didn't call for detailed analysis; However, a simple analysis using MS Office Excel 2007 was done. After finishing the analysis and obtaining the corresponding outcomes, a comparison between the WPC and 7 other types of commercial wood was conducted for the sake of testing the hypothesis of application adequacy based on the flexural and water absorption properties. The results showed an ultimate superiority of WPC in water absorption area as it was the best in terms of minimum water affected compared to others. Nonetheless, the flexural results weren't so promising which is shown in details this chapter in "Analysis of flexural test" section. All the performed tests on the 7 wood types were done with the same settings, equipment, and conditioning of the WPC.

6.1. Analysis Of Flexural Test

The flexural test; intended to obtain flexural strength and modulus values, was conducted in this work in accordance with the ASTM D 4761-05. All details of this test

are explained in chapter 5. Initially, Design-Expert was utilized for designing the bigger mixture constraint region; consisted of 51 specimens and 10 replications (appendix 2). The region choice was based on a selection of a large space to be able to do many trials to come up with the best possible fitting model (explained further in chapter 3). Then, a basic initial design consisted of 22 specimens (12 for the design, 5 for the lack of fit estimation, and 4 to 5 replications for error estimation) was adopted (appendix 4). It used a D-optimal design with a quadratic model (explained further in chapter 3). Due to the fact that infinite trials could be done continuously till reaching a feasible model in addition to the required validation step, an algorithm was introduced to navigate the design points within the space of the bigger mixture constraint region to get the best possible model fit and validate it (see figure 6-1).

6.1.1 Design space navigation algorithm

Typically, this algorithm is a finite sequence of instructions applied till reaching the most feasible model fit of the flexural strength and modulus; using Design-Expert. The application of this algorithm helped reaching a final model with appropriate fitting equation, minimum possible error, and lack of fit. The flowchart shown in Figure 6-1 explains the steps of the algorithm. The initial design of the flexural strength and modulus started with a quadratic model till reaching a partial quadratic model

Start Building the design based on an initial model

Model adequate

Model not adequate

If **NO** other models are suggested by DE

Use suggested model by DE

If other models are suggested by DE

Outlier elimination and adding new points to the model

Model not adequate

Model not adequate

Model adequate

Model reduction (outliers' elimination)

Model not adequate

Model not adequate

Model adequate

Extend the model with new pts (validation)

2 times loop of changing new added pts

Model not adequate

Model adequate

Done

Model refinement (outliers' elimination)

Model not adequate after 2 times loop

Acronyms & Abbreviations:

DE: Design-Expert
Pts: Points

Flowchart legend

Action

Start/end

Decision

Flow direction
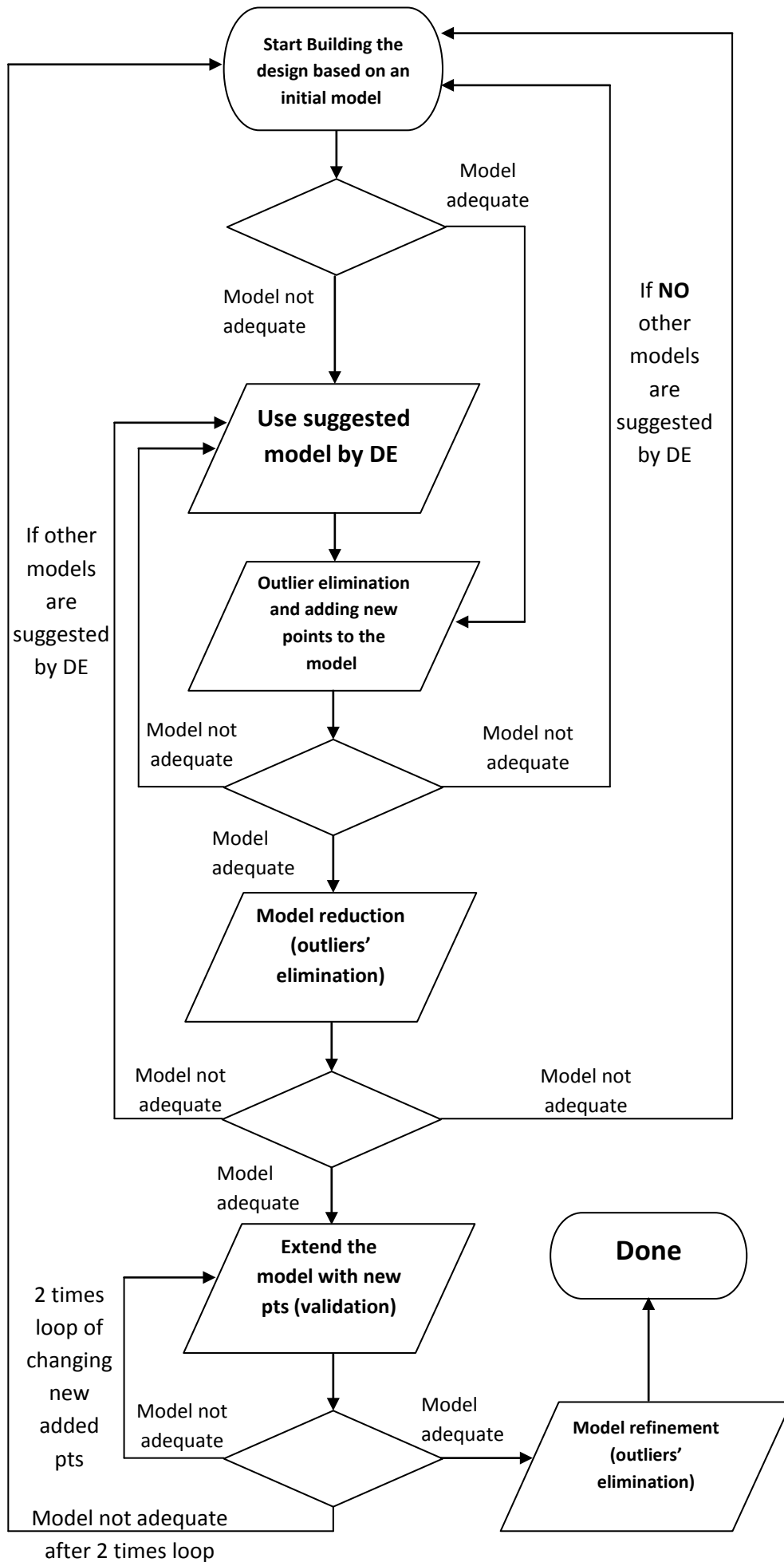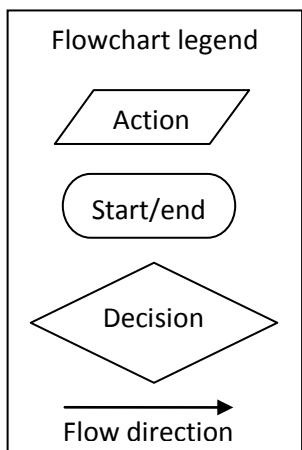
Figure 6-1 : Design space navigation algorithm

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in the last step. It is a linear + squared model telling it is a combination of 1st and 2nd degree terms. For validating the model, a step was added to the algorithm "Extend the model with new pts (validation)"; where distinct points were added to the model to make sure it is working well (will be explained further in the next section). It was followed by a removal of outlier step to minimize any noise affecting the model. The *Decision* of an adequate model or not was based on the model analysis; represented in "Fit Summary", "ANOVA", "Case statistics", and "Graphical displays", obtained from Design-Expert output. Each one will be explained further the "Design-Expert analysis" section.

i.   model verification and validation

Model verification and validation are the main reason behind trusting an obtained model or not. Typically, it would affect the algorithm utilized to navigate the design space and consequently all the steps of analysis. In other words, the algorithm will stop when the model is verified and validated.

Model verification is making sure that the model performs as intended (Macal 2005); i.e. ensuring that no mistakes have been made in implementing the model. However, no computational model will be 100% free of errors. A properly structured good software; Design-Expert, will increase level of certainty in the model (Macal 2005). Typically what are tested are the proper implementation of the algorithm and the minimum model content of errors, mistakes, or bugs. Model validation is concerned with whether the model is representing and imitating the performance of a real world system (Macal 2005).

The ultimate goal of model validation is to make the model useful in the sense that it provides accurate information about the system being modeled, and to make the model actually used (Macal 2005). This is achieved when the model predictions are almost matching experimental data.

In this work model verification was done to the "adequate model" obtained after the "Model reduction (outliers' elimination)" step in the algorithm; where it was checked for errors, and the algorithm proper implementation. The next step was added to the algorithm to validate the model; where additional experimental data were added to check the model prediction adequacy or the truly imitation of real word system. The final step of model refinement was necessary due to the fact that some of the added data are outliers; especially that the plastic dealt with is obtained from reject of rejects waste.

6.1.2 Output analysis

In this section, the analysis of the output obtained from Design-Expert will be discussed in details. Only the output of the last model will analyzed as it will be a replication of presenting or going through all models; same steps will be performed each time, till reaching the last step in the algorithm i.e. the best adequate model. The final design of the flexural modulus and strength are shown respectively in appendices 5 and 6. The output of Design-Expert contains four sections: Fit Summary, ANOVA, Diagnostic Case Statistics, and graphical display.

i. Fit Summary

The first thing to be looked at is the "Fit Summary". It is used mainly for the comparison between different model types fitting the inputted data. In addition, it gives initial information about the adequacy of a certain model. Design-Expert is provided also with a comparison tool that recommends one of the presented models (linear, linear + squared, quadratic, cubic…) to be used by simply writing "suggested" beside the intended model. Design-Expert utilizes a special scoring system called "Whitcomb Score" for the selection criteria (Design-Expert 2010). All the analysis is based on a 5% significance level ($\alpha=0.05$). This level is the preset level utilized by the model and adopted in many experimental design applications (Design-Expert 2010) & (Myers and Montgomery 2002). The "Fit Summary" contains 4 sections: Summary, Sequential model sum of squares, Lack of fit tests, and Model summary statistics. Typically, what is important to be looked at are the Summary and Model summary statistics as they contain all the important data of the 4 sections.

The Summary is a précis of the most important criteria in the 3 other sections in the "Fit Summary". It contains all the important parameters needed for the comparison; sequential p-value, lack of fit p-value, adjusted R-squared, and predicted R-squared, between models (linear, quadratic, cubic…) as shown in table 6-1 and 6-2. Results showed that the partial quadratic or (linear + squared) model was recommended in the case of flexural modulus and strength (table 6-1 and 6-2). The selection was based on the lowest sequential p-value, highest values of lack of fit p-value, adjusted and predicted R-squared values as explained above.

The sequential p-value shows the accumulating improvement in the model fit as terms of the intended model are added, which should be the minimum value among others i.e. The smallest p-values (Prob>F) found in table 6-1 and 6-2 indicate that adding partial quadratic (linear+squared) terms has improved the model of the flexural modulus and strength. Lack of fit p-value should be the maximum among all values. When prob>Fvalue < 0.05 indicates a high lack of fit, telling that variation in model points significantly differs from variations in the replicated points; which is not desired and could lead to an inadequate model i.e. If a model shows lack of fit, it should not be used to predict the response (Design-Expert 2010). On the other hand, when prob>Fvalue > 0.1 indicates a low lack of fit which is the case of partial quadratic (linear+squared) in table 6-1 and 6-2. Adjusted R-Squared and predicted R-Squared should be the maximum values among others. In this case, the results showed that partial quadratic (linear+squared) model is the best selection in the two cases (see table 6-1 and 6-2).

Table 6-1: Summary table of the flexural modulus obtained from Design-Expert

Summary Source	Sequential p-value	Lack of Fit p-value	Adjusted R-Squared	Predicted R-Squared	
Linear	0.0016	0.6949	0.5111	0.2327	
Linear+Squared	0.0372	0.9089	0.6930	0.4104	Suggested
Quadratic	0.1355	0.8582	0.6479	0.1610	
Special Cubic	0.4952	0.8760	0.6396	-0.4272	

Summary					
Source	Sequential p-value	Lack of Fit p-value	Adjusted R-Squared	Predicted R-Squared	
Linear	0.0265	0.8795	0.3389	0.0394	
Linear+Squared	0.0271	0.9955	0.6414	0.4420	Suggested
Quadratic	0.0980	0.9927	0.5943	0.1359	
Special Cubic	0.9229	0.9588	0.3742	-13.6591	

The second essential table is the Model Summary Statistics where the PRESS is the additional important value that should be evaluated. It is the predicted residual error sum of squares (PRESS) that is a measure of how the model is fitting each point in the design (Design-Expert 2010). Typically, it is a residual value; difference between actual and predicted values when the run under investigation is removed (Design-Expert 2010), needing to be minimized as possible. The lowest relative value is the chosen one as the case of partial quadratic (linear+squared) models in table 6-3 and 6-4. R-squared value found in table 6-4 and 6-5 is disregarded due to the fact that it measures the amount variation around the mean explained by the model without being adjusted to the number of terms added to the model; as the case of adjusted R-squared. The problem here is that when the number of terms increases the R- squared could be inflated and gives higher values even if the added terms are insignificant. The standard deviation; intuitively, should be minimum as found in partial quadratic (linear+squared) models in table 6-3 and 6-4.

Based on these criteria, the partial quadratic (linear+squared) model was chosen for the modeling the flexural strength and modulus and then going to the details of the chosen model by looking at ANOVA.

Table 6-3: Model summary statistics table for flexural modulus

Model Summary Statistics						
Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS	
Linear	187.38671	0.5844686	0.5111	0.2327	1102278	
Linear+Squared	148.50384	0.8004296	0.6930	0.4104	846923.5	Suggested
Quadratic	159.0219	0.806365	0.6479	0.1610	1205327	
Special Cubic	160.88367	0.8738755	0.6396	-0.4272	2050260	

Table 6-4: Model summary statistics table for flexural strength

Model Summary Statistics						
Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS	
Linear	1.5498005	0.4490821	0.3389	0.0394	62.81993	
Linear+Squared	1.1413457	0.7808855	0.6414	0.4420	36.49433	Suggested
Quadratic	1.2140809	0.7971468	0.5943	0.1359	56.51072	
Special Cubic	1.5078277	0.8261729	0.3742	-13.6591	958.6537	

ii. ANOVA

Analysis of variance section generated from the output of Design-Expert produce three main outcomes: ANOVA table, R-squared table, and the final fitting equation.

Table 6-5: ANOVA table of flexural modulus

Analysis of variance table						
Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Block	326823.78	2	163411.892			
Model	1144512.1	4	286128.034	15.6758752	< 0.0001	significant
Linear Mixture	839622.07	3	279874.025	15.3332416	< 0.0001	significant
D^2	304890.06	1	304890.06	16.7037758	0.0009	significant
Residual	292044.21	16	18252.763			
Lack of Fit	159850.59	13	12296.1995	0.27904978	0.9562	not significant
Pure Error	132193.61	3	44064.5379			
Cor Total	1763380.1	22				

The ANOVA table of the flexural modulus (table 6-5) showed that the D^2 and the linear mixture; represented in the A value or the plastic value, have significant effect. They have values with low p-value (Prob>F) where it is less than 0.05. It tells that these factors are affecting the model as they are significant factors. Therefore, the model is affected and significant as shown in table 6-5, where It is less likely that any factor of the model has a significant effect on the response. The results therefore show that the plastic utilized (A) and the wood with size of up to 0.5mm (D^2) are affecting the model behavior in a certain way. This could be a result of the presence of impurities in the material used; especially, that the material was obtained from the waste rejects. It should be also noted that the Block is the batch of plastic utilized, where 3 batched were adopted in this work; each batch contains waste residues (oil, sand, food, liquids…) and impurities different than the other, which has an effect also on the plastic factor (A) and the whole model. However, the effect was not extended to harm the model as a whole; rather, good result of lack of fit (low lack of fit) shows a positive indicator; telling that variation in model points doesn't differ from variations in the replicated points. In addition, the R-squared table results were promising; as it will be explained (see table 6-7).

Table 6-6: ANOVA table of flexural strength

Analysis of variance table						
Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Block	25.29041	2	12.6452051			
Model	51.067344	7	7.29533484	5.60029396	0.0060	significant
Linear Mixture	29.368491	3	9.78949697	7.51494785	0.0052	significant
A^2	21.062478	1	21.0624784	16.1686987	0.0020	significant
B^2	3.8226219	1	3.82262185	2.93445148	0.1147	
C^2	0.0445442	1	0.04454423	0.03419456	0.8567	
D^2	0.6908754	1	0.69087539	0.53035335	0.4817	
Residual	14.32937	11	1.30266998			
Lack of Fit	4.3168698	9	0.4796522	0.09581068	0.9955	not significant
Pure Error	10.0125	2	5.00625			
Cor Total	90.687124	20				

The ANOVA table of the flexural strength (table 6-6) showed that the A^2 and the linear mixture; represented in the A value or the plastic value, have significant effect. Therefore, the model is also affected and significant as shown in table 6-6. The results therefore show that the plastic utilized (A), as the case of flexural modulus, is the most affecting factor. However, the effect also in this case was not extended to harm the whole model; based on low lack of fit obtained and good results of R-squared table as it will be explained (see table 6-8).

R-squared table consists of the model standard deviation, mean, coefficient of variation, predicted residual error sum of squares (PRESS), R-Squared, Adj R-Squared, Pred R-Squared, and adequate precision.

Std. Dev.	135.1028	R-Squared	0.7967
Mean	1061.2591	Adj R-Squared	0.7459
C.V. %	12.7304	Pred R-Squared	0.5792
PRESS	604554.0398	Adeq Precision	13.2264

The most important values for evaluation are the Adj and Pred R-Squared (Myers and Montgomery 2002) & (Design-Expert 2010). Adj and Pred R-Squared should have relatively high values and they should be within 0.2 of each other. Otherwise, outliers should be investigated, different order for the model could be adopted, model should be reduced, or blocks' effect may need to be inspected (Design-Expert 2010). The adjusted R-Squared is explained as the variation amount around the mean explained by the model and adjusted for the number of terms in the model (Design-Expert 2010) i.e. when the number of terms that doesn't add up to the model increases the value of Adjusted R-Squared decreases. This gives a bad indicator calling for terms reduction, looking for outliers, or maybe large block effect. Therefore, higher adjusted R-Squared is desirable. The predicted R-squared is the amount of variation around the mean in the new data explained by the model (Design-Expert 2010). When the value of predicted R-squared is negative; this is an indicator of having an: outlier in the data, number of runs are close to the number of model parameters, or high leverage points with high Cook's values affecting the model. Therefore, higher predicted R-Squared is desirable. Results have shown in table 6-7 and 6-8 adequate adjusted and predicted R-squared values and in two cases they are within 0.2 of each other.

Table 6-8: R-squared table of flexural strength

Std. Dev.	1.1413	R-Squared	0.7809
Mean	9.0652	Adj R-Squared	0.6414
C.V. %	12.5904	Pred R-Squared	0.4420
PRESS	36.4943	Adeq Precision	10.8525

Adequate Precision is a signal to noise ratio. It compares the range of the predicted values at the design points to the average prediction error. Ratio > 4 indicates an adequate signal and this model can be used to navigate design space. (Design-Expert 2010). The results of both the flexural strength and modulus shows adequate signals and the models could navigate design space (see table 6-7 and 6-8).

Standard deviation, mean, CV, and R-Squared value are general guidelines for the model and shouldn't be mainly used to judge the model.

The final fitting equations of the flexural strength and modulus are partial quadratic having first and second order terms. Their mathematical formulas are as follow:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_4 x_4 + \beta_5 x_1^2 + \beta_6 x_2^2 + \beta_7 x_3^2 + \beta_8 x_4^2 + \varepsilon$$

Where y is the response; **_flexural strength_**, β_0 is the intercept, β_1 is the coefficient of the plastic waste percentage, x_1 is the percentage of plastic waste within the mix, β_2 is the coefficient of talc percentage, x_2 is the percentage of talc within the mix, β_3 is the coefficient of wood up to 1.18mm percentage, x_3 is the percentage of wood up to 1.18mm within the mix, β_4 is the coefficient of wood up to 0.5mm percentage, x_4 is the percentage of wood up to 0.5mm within the mix, and ε is the error term.

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_4 x_4 + \beta_5 x_4^2 + \varepsilon$$

Where y is the response; *flexural modulus*, β_0 is the intercept, β_1 is the coefficient of the plastic waste percentage, x_1 is the percentage of plastic waste within the mix, β_2 is the coefficient of talc percentage, x_2 is the percentage of talc within the mix, β_3 is the coefficient of wood up to 1.18mm percentage, x_3 is the percentage of wood up to 1.18mm within the mix, β_4 is the coefficient of wood up to 0.5mm percentage, x_4 is the percentage of wood up to 0.5mm within the mix, and ε is the error term.

The final fitting equations (expected equations) of the flexural strength and modulus are obtained from Design-Expert as follow:

Flexural Strength	=
1.294569813	* A (Plastic)
-0.139061441	* B (Talc)
-0.443198433	* C (Wood 1.18)
-0.305335411	* D (Wood 0.5)
-0.014896061	* A (Plastic)^2
-0.004052364	* B (Talc)^2
+0.000516824	* C (Wood 1.18)^2
-0.00090095	* D (Wood 0.5)^2

Flexural Modulus	=
5.578595234	* A (Plastic)
+20.13905792	* B (Talc)
+16.86695494	* C (Wood 1.18)
-12.8462427	* D (Wood 0.5)
+0.863717373	* D (Wood 0.5)^2

The next section is more concerned with the *inside (details)* of the data; where individual points will be discussed to analyze residuals, detect outliers, and other points affecting the response. The diagnostic case statistics and the graphical display section are studied when the results of Fit Summary and ANOVA are approved.

iii. The diagnostic case statistics

In this section many outputs will be discussed. As a good start for this analysis, the diagnostic case statistics table produced is a good guiding beginning. It is table containing most of the values that will be discussed; which are: Actual Value, Predicted Value, Residuals, Leverage, Cook's Distance, Internally Studentized Residuals, Externally Studentized Residuals, Influence on Fitted Value (Difference in Fits) DFFITS, and Difference in Betas (DFBETAS). The tables of flexural modulus and strength are found in appendices 7 and 8. An important notice should be considered regarding the tables that the predicted values include block correction i.e. the block effect is included in the model equations in these tables. Before going to the analysis, an explanation of the significance of all these values is essential. Afterwards, the graphical displays of these numbers will make it easier for evaluation as Design-Expert produces all these number in graphical form.

The residual column is the first to be looked at. It is calculated by subtracting the Actual Value (response output) from the Predicted Value (resulting from the final equation after block correction). Typically, the lowest the better and when it is zero the model is exactly like the actual response which is so rare to happen. However, residuals should not be used to compare values as are and judge points.

These values don't have a cut off or reference to be compared at. Therefore, it was needed to use internally and externally studentized residuals for this analysis. The internally and externally studentized residuals are the values of residuals adjusted to the square root of mean square error $\sqrt{MSE}$ of the model (Habing 2004); it is the value of residual found in ANOVA table. Therefore, the residual value by itself is not

sufficient as it should be adjusted to the $\sqrt{MSE}$. For example, if a residual value was 100 and $\sqrt{MSE}$ was 1000, therefore the residual is not significant. On the other hand, if the residual value was 100 and $\sqrt{MSE}$ was 50, this is a primary indicator for a significant point.

The externally studentized residual is the residual at a certain run i; difference between observed and expected value, adjusted by the $\sqrt{MSE_i}$ when the run i is not considered in calculations (Habing 2004). It is calculated as follow: $t_i = \frac{e_i}{\sqrt{MSE_i(1-h_{ii})}}$, where t_i is the externally studentized residual for run i, e_i is the residual at run i, $\sqrt{MSE_i}$ is the mean square of error calculated for the model when the run i is excluded from the analysis, and h_{ii} is the leverage value for the same run (Habing 2004). Internally studentized residual has the same concept as the external, except that it is adjusted by the $\sqrt{MSE}$ with the inclusion of the run in question. The equation therefore is as follow: $t_i = \frac{e_i}{\sqrt{MSE(1-h_{ii})}}$, where the $\sqrt{MSE}$ is the mean square of error calculated for the model without the exclusion of the run i in question (Design-Expert 2010). Typically, internally and externally studentized residuals are needed to be minimized as possible; the closer to zero the better. Usually, the limits are set in Design-Expert at ±3σ (Design-Expert 2010).

Large values of externally studentized residuals should be investigated; where the target should be a zero residual (Design-Expert 2010).

The leverage (hii) is an indicator value for the correlation between the actual and predicted values at a certain point (Design-Expert 2010). In other words, how close to each other (observed and predicted) they are. When the leverage value of a certain

point is high, this is an indicator that the model would pass by or approaches this point; thus any error associated with this point would affect the whole model negatively and would be included in all predictions when its Cook's and DFFITS value are high *i.e. we cannot judge a leverage value until we see its Cook's Distance and DFFITS value*. A leverage of a point is between "0" and "1". A point with leverage of "1" tells that the residual is zero and the model pass by this point and the observed and predicted values are the same. A point is considered having a high leverage when its value is greater than twice the average hii; calculated as the number of model parameters (including intercept and blocks) divided by the total number of runs.

The DFFITS and Cook's value are correlated respectively to the externally and internally studentized residuals. The DFFITS value is the externally studentized residual of a run; which is the residual corrected to the MSE root when the run in question is removed, adjusted by its leverage value (Habing 2004). Its equation is as follow:

$\text{DFFITS} = t_i \left(\frac{h_{ii}}{1 - h_{ii}} \right)^{1/2}$, where t_i is the externally studentized residual for run i, h_{ii} is the leverage value for the same run (Habing 2004).

The same concept applies to the Cook's value; which is the internally studentized residual of a run (the residual corrected to the MSE root) adjusted by its leverage value (Habing 2004). Its equation is as follow: $\text{Di} = \frac{r_i^2}{p} \left(\frac{h_{ii}}{1 - h_{ii}} \right)$, where Di is the Cook's value for run i, p is the number of parameters included in the model (intercepts are included), r_i is the internally studentized residual, and the h_{ii} is the leverage value (Habing 2004).

The concept of the relationship between Cook's and leverage is applied the same with the DFFITS except that the DFFITS is keen to get the effect on the model of a certain run when its value is removed. On the other hand, Cook's is interested with the effect of a specific run on the model. However, they share both the adjusted values for their respective leverage values.

Therefore, when looking at the values of Di and DFFITS, leverage values for the same runs should be also observed. Consequently, when Di or DFFITS are high and the leverage is high too, it is a bad indicator telling that the error of this point will be propagated in the whole model affecting it negatively. This point could be a mistake in reading, outlier, or design point far from remaining cases… Table 6-9 explains a set of conditions proposed and their respective probable outcomes.

In a Cook's graph analysis, points should be close to each other and zero to indicate accepted points. Meanwhile, large relative values should be revised alongside with its leverage values (Habing 2004). DFFITS values should close to zero and less than the outer limits of ±2 (Design-Expert 2010).

Table 6-9: leverage Vs Cook's Distance/DFFITS probable outcomes

Leverage (hii)	Cook's Distance (Di)/ DFFITS	Comments
High	High	bad indicator telling that the error of this point will be propagated in the whole model (outlier)
High	Low	Ideal case. Good point which will not propagate its error affecting the model.
Low	High	This is a bad estimate of this point but does not affect the whole model
Low	Low	Not effective point

Difference in Betas (DFBETAS) is calculated for each coefficient in the model. It measures the effect of a certain observation (actual run) on each coefficient (Design-Expert 2010). DFBETAS produces a graph for each coefficient. Typically, when the observations have minimum effect on the coefficients, the values are close to zero; which is the target here. The outer limits are set at ±2 (Design-Expert 2010).

iv. Graphical displays

All the values obtained from the Diagnostic case statistics table are presented in a graphical form; making the analysis easier. Therefore, the flexural modulus and strength results of the Diagnostic case statistics will be discussed in this section based on their respective graphs.

Starting by the leverage, Cook's D, DFFITS, and externally studentized residuals of the flexural modulus: by looking at the DFFITS and Cook's D graphs (figure 6-2), all points fall within limits and no patterns are shown; however, run 3, 13, and 20 show relatively high values which is also clear in the externally studentized residuals (figure 6-2).

Relating these graphs to the leverage is the most important step as mentioned above. When looking at the runs in the respective leverage graph, it is found that /they have relatively low values which don't call for a reconsideration of these runs (figure 6-2). By looking at the DFBETAS graphs (appendix 9), no effect was shown on coefficients except for the coefficient D and D^2 which were relatively affected by runs 13 and 20. However, these runs are within limits but they have relatively high values (0.603, -0.735) for D and (-0.874, 1.065) for D^2 which doesn't call for further consideration.
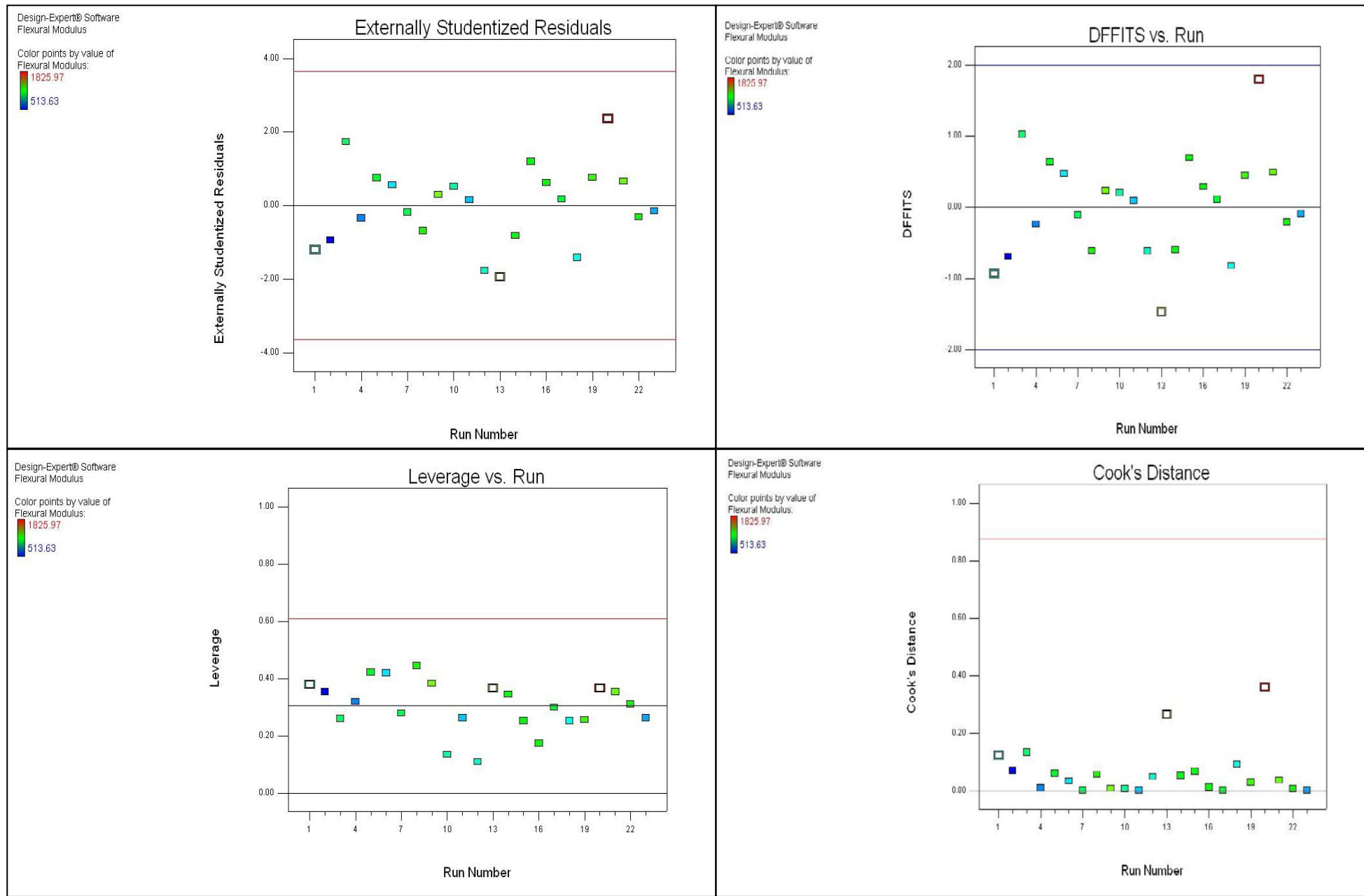
Figure 6-2: Cook's, DFFITS, leverage, and Externally studentized residuals for flexural modulus

Normal probability plot for the studentized residuals shows an almost straight line; which tells it is normally distributed (figure 6-3).
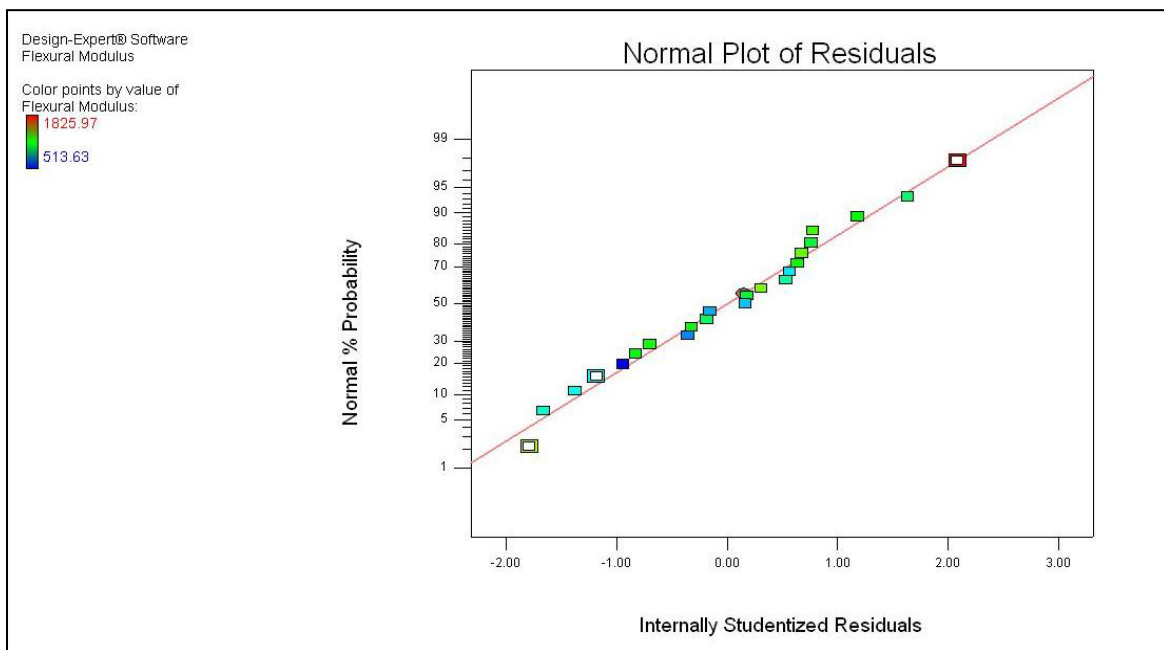
Figure 6-3: normal probability plot for residuals of flexural modulus

Residuals VS blocks graph (figure 6-4) shows that the internally studentized residuals are not aligned in the same way i.e. each block is affecting the output in a different way. This is mainly due to different batches (blocks) of plastic waste material; which is a plastic obtained from rejects of rejected waste (contaminated with unknown components), causing different effects on the product. Therefore, the effect on the residuals was not consistent. However, all values fall within limits which tell a feasible output.
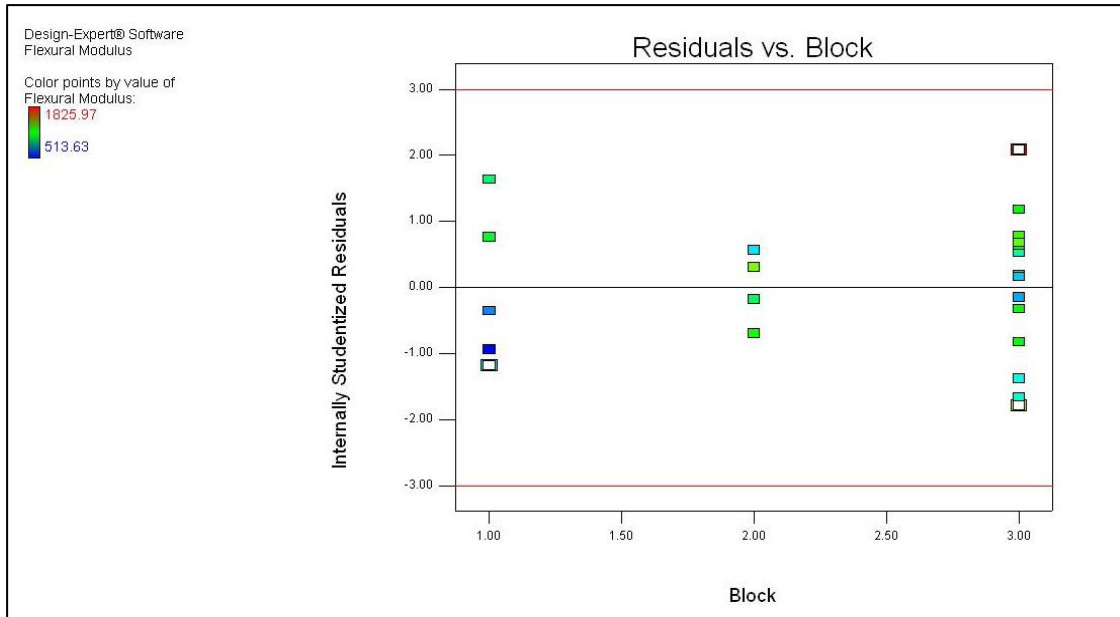
Figure 6-4: residuals VS. block of flexural modulus

Finally, a 3-D graph is obtained representing the response surface model as shown in figure 6-5. It shows that the flexural modulus highest values; or best mix, were obtained when talc is close to 35%, plastic waste 40%, and wood waste (particle up to 1.18mm) about 15% and wood waste (particle up to 0.5mm) 10%.
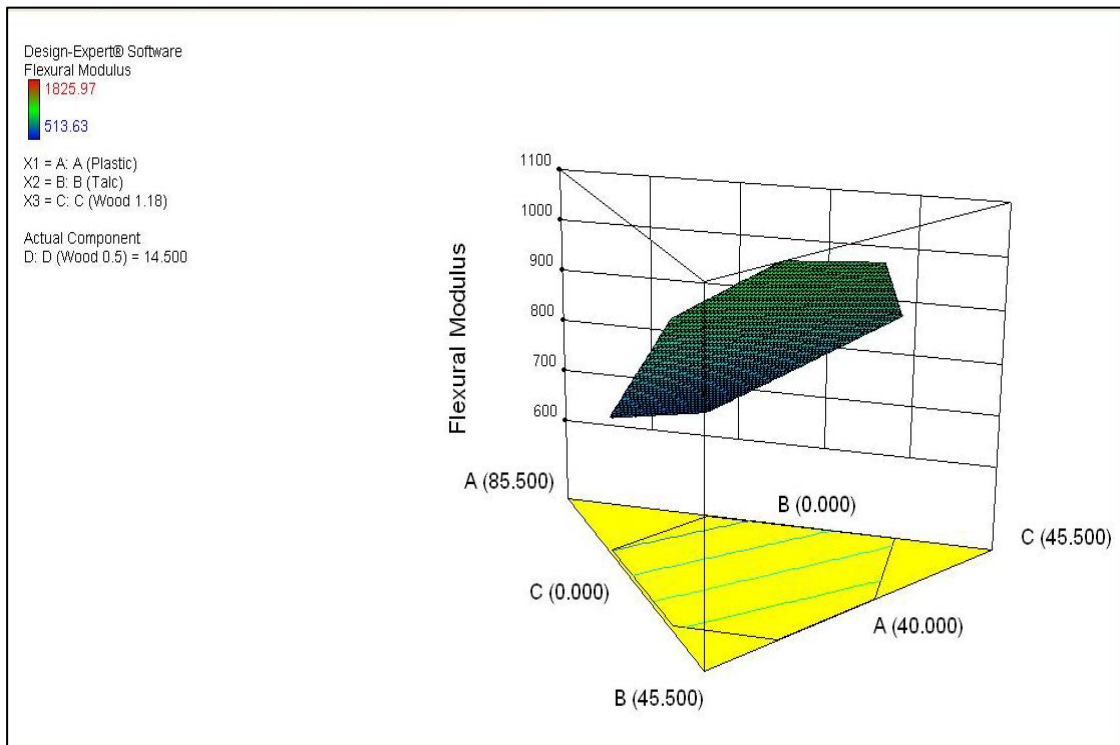
Figure 6-5 : 3-D graph of flexural modulus response

Going to the leverage, Cook's D, DFFITS, and externally studentized residuals of the flexural strength: by looking at the DFFITS and Cook's D graphs (figure 6-6), all points fall within limits and no patterns are shown where points are randomly scattered around the zero line in DFFITS and close to zero in Cook's D; however, run 4 is relatively high in Cook's D and falls out of limit in DFFITS. Consequently, it felt out in the externally studentized residuals; as DFFITS and externally studentized residuals are tightly connected. In addition, run 1 has relatively high values in Cook's D, DFFITS, and externally studentized residuals; However, it falls within limits. On the other hand, run 1 and 4 could not be judged before looking at their respective leverage values. It is shown that their leverage values are lower than the average and lower than most of the runs. Therefore, run 1 doesn't call for a reconsideration; yet, run 4 needs to be further analyzed. When checking the DFBETAS graphs (appendix 10), effect was shown on coefficients B, B^2, C, and C^2 which were relatively affected by runs 1 and 4. In the case of C and C^2, runs 1 and 4 are relatively high but not that far from other runs; in addition, they fall within limits. Yet in B and B^2 case, run 4 shows a tendency to reach control limits (±2); where it has the following values (1.829, -1.583). Moreover, run 1 was relatively close to other values. In conclusion, run 1 doesn't need further consideration; however run 4 was a strong signal although it has a low leverage value. When returning to know the reason behind the results of run 4, it was found that it is a replication for the run 1 specimen. The 2 specimens were produced at the same day and by inspecting them, residues from the furnace; used to form the WPC paste, were found. Therefore, this could be the reason behind these variations.
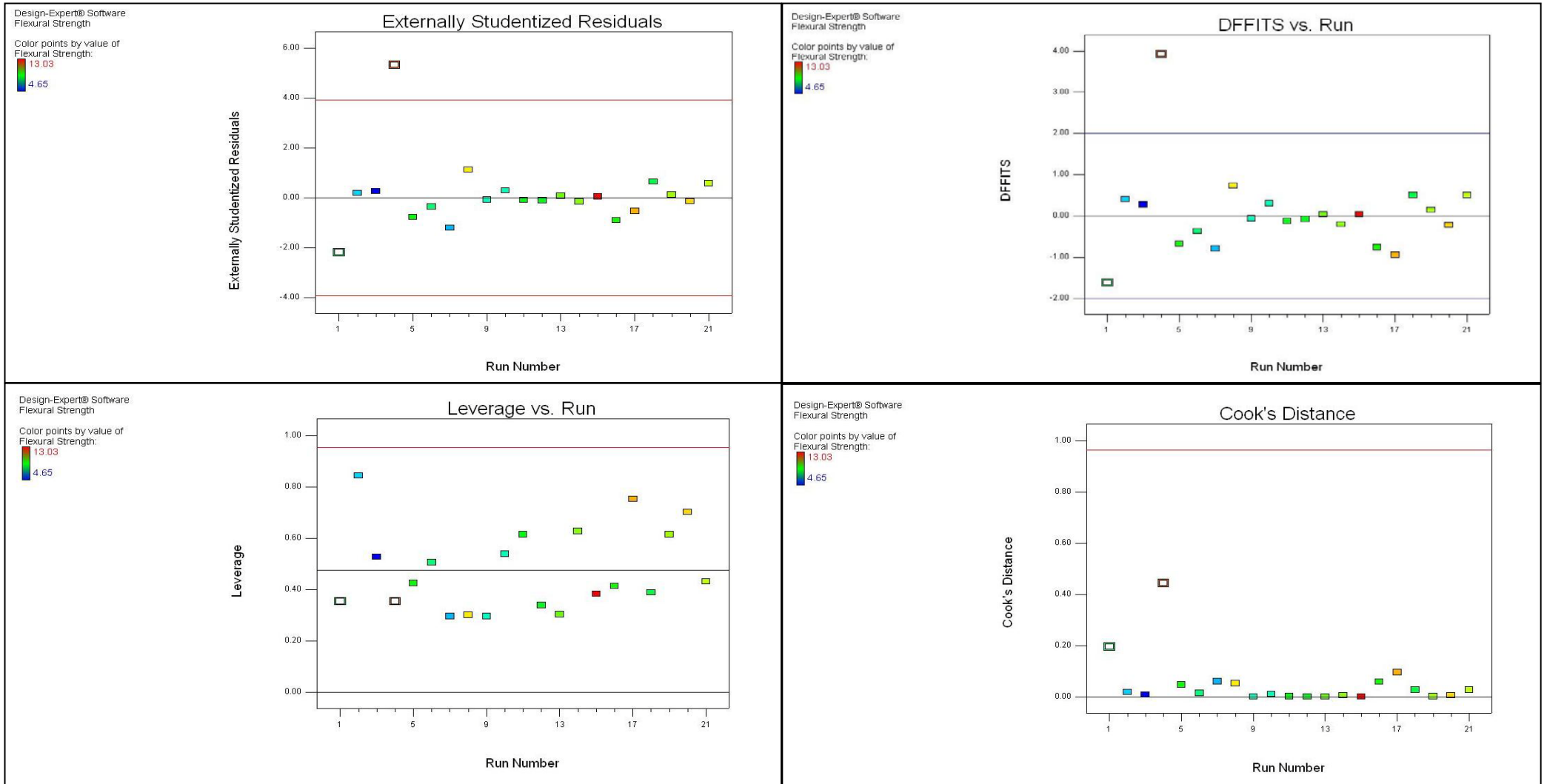
Figure 6-6 : Cook's, DFFITS, leverage, and externally studentized residuals for flexural strength

Normal probability plot for the studentized residuals shows an almost straight line; which tells it is normally distributed except for run 1 and 4 (figure 6-7).
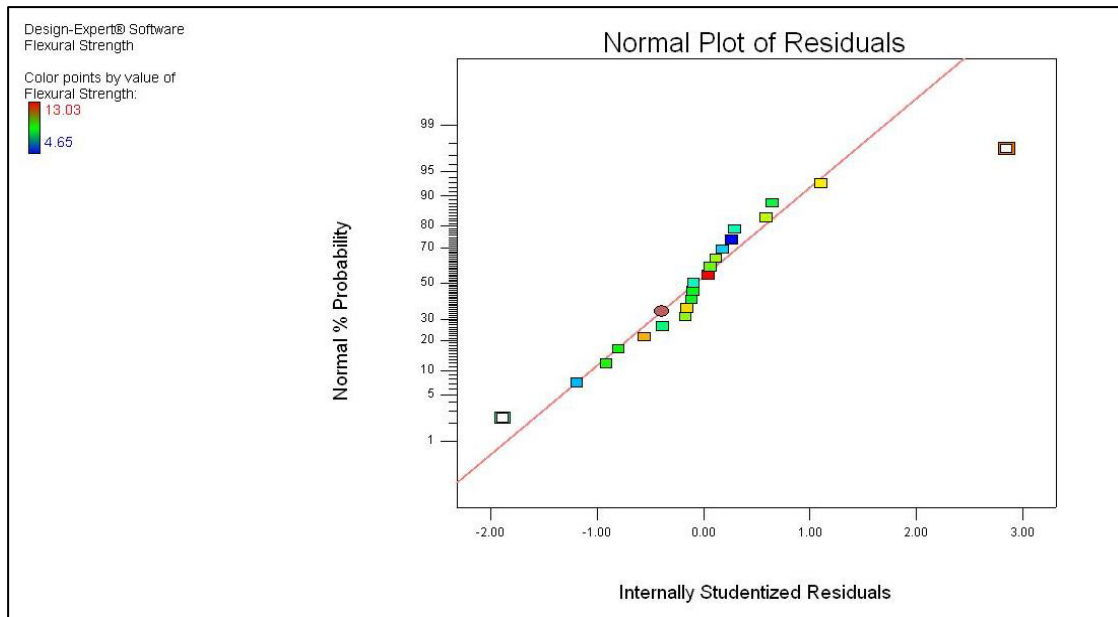
Figure 6-7: normal probability plot for residuals of flexural strength

Residuals VS blocks graph (figure 6-8) shows that the internally studentized residuals are not aligned in the same way as in the case of flexural modulus. Run 4 approaches the upper limit. However, all values fall within limits which tell a feasible output.
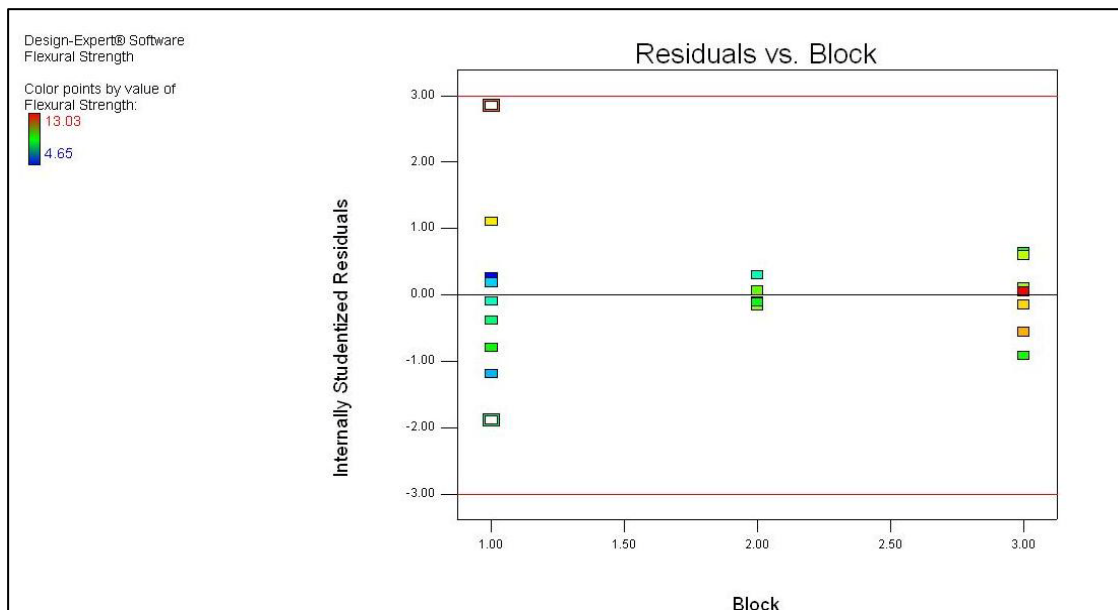
Figure 6-8: residuals VS. block of flexural strength

Finally, a 3-D graph is obtained representing the response surface model as shown in figure 6-9. It shows that flexural strength highest values; or best mix, were obtained when the talc is close to 30%, plastic waste 50% and wood waste (of particle size up to 1.18mm) and wood waste (particle up to 0.5mm) of average percentages of 10%.
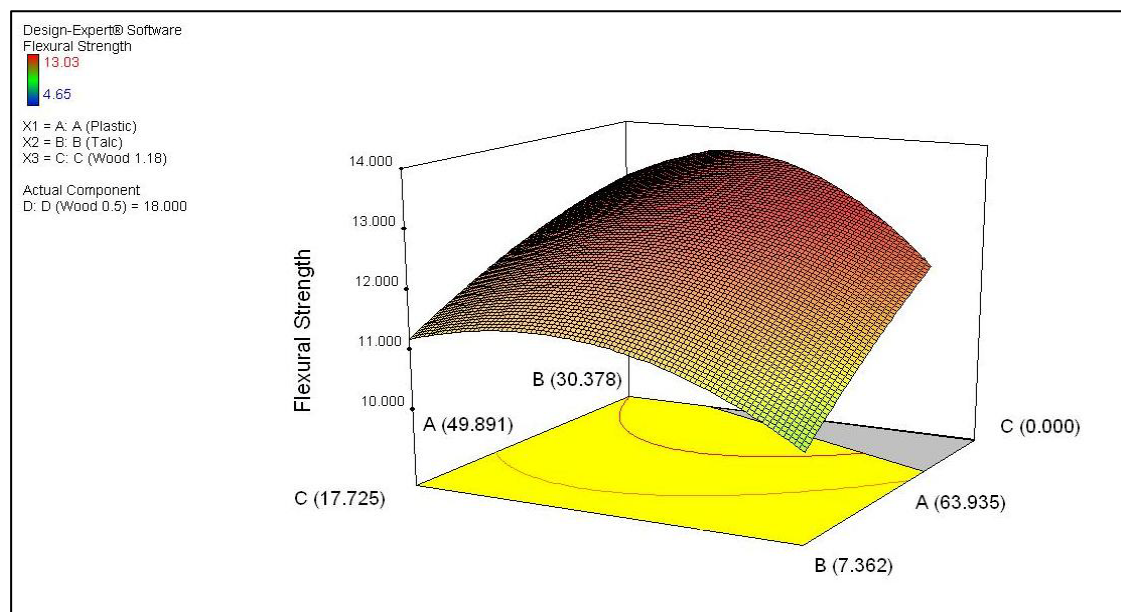

Figure 6-9: 3-D graph of flexural strength response

6.2. Analysis Of Water Thickness And Swelling Test

The results of the water absorption test and thickness swelling were so promising. The interaction with water was minimal and almost no clear effect was apparent. Therefore, the need for conducting a detailed analysis here to check the effect of each factor and to model the response wasn't necessary. However, 56 specimens were tested divided into two parts: 51 runs covering the distinct runs; generated using Design-Expert, covering the vertices, overall centroid, edge of centers, axial

points, and constraint plane centroid; and 5 replicate runs (table 6-10). Simple presentation of percentages; their averages and variability, will be an adequate estimator in this case. All the percentages obtained were based on the next equations:

% of change in weight (water absorbed) = ((W1-Wo)/Wo) *100

% of change in volume (water absorbed) = ((V1-Vo)/Vo) *100

% of moisture = ((W2-Wo)/Wo) *100

Where:

%: the percentage sign

Wo: the original weight of the specimen

W1: the weight of the specimen after submerged for 24 hour

Vo: the original volume of the specimen

V1: the volume of the specimen after submerged for 24 hour

W2: the weight of the specimen after dried in oven

Referring to table 6-10, the average percentage of water absorption obtained was 0.4% with a standard deviation of 0.28%, range of 1.6%, maximum of 1.7%, and a minimum of 0.09%.

The WPC thickness swelling test showed also minor dimensional variability; explained in terms of volume, shown in table 6-10. The average volume change was averaged 0.45% with a standard deviation of 1.38%, range of 5%, maximum of 5%, and a minimum of 0%.

The moisture content calculation was the last step in this test. The average moisture change was averaged 0.16% with a standard deviation of 0.06%, range of 0.37%, maximum of 0.44%, and a minimum of 0.07% (see table 6-10).

Minor absorption is obvious in these results. Furthermore, these results were compared to several types of commercial wood in the next section to validate its usage in areas subjected to water.

The next table (table 6-10) is a presentation of results of water absorption test of 56 specimens; where the distinct runs have a symbol of (Std) and the replication runs have a symbol of (R). Volume and weight values before and after water submersion are represented and their associated increase percentages. In addition, humidity; obtained from the original and the dried weights is also shown in the table. The mixture's ingredients (% of plastic, talc, and wood) of each run are shown in Appendix 2, where the same symbols (Std and R) with their respective numbers (1, 2,…51) are represented.

No.		cm^3	cm^3	%	Gm	gm	%	%
specimen		Volume before	Volume after	Volume increase	Weight before	Weight After	Weight increase	Humidity
R	1	440	440	0	494.1	496.2	0.425	0.168
R	2	394	394	0	457.4	459.2	0.394	0.130
R	3	396	396.8	0.20	397.7	398.7	0.251	0.175
R	4	420	420	0	499.2	500.6	0.280	0.232
R	5	417.9	420	0.50	470.4	472.2	0.383	0.122
Std	1	400	400	0	434.7	442.2	1.725	0.223
Std	2	433.4	433.4	0	421.8	423.7	0.450	0.220
Std	3	411.6	411.6	0	431.6	434.5	0.672	0.224
Std	4	370	370	0	435	437.1	0.483	0.441
Std	5	396	396	0	417.3	418.9	0.383	0.191
Std	6	396	396	0	450.5	450.9	0.089	0.133
Std	7	400	400	0	413.9	415.1	0.290	0.193
Std	8	436.48	436.48	0	500	500.5	0.100	0.100
Std	9	464.6	464.6	0	546.3	547.8	0.275	0.146
Std	10	440	440	0	561.9	562.6	0.125	0.134
Std	11	378.1	378.1	0	476.9	478.5	0.336	0.132
Std	12	398	398	0	542.8	543.5	0.129	0.181
Std	13	402	422.1	5	442.4	446.7	0.972	0.220
Std	14	464.6	464.6	0	538.6	542	0.631	0.198
Std	15	455.4	455.4	0	465.7	468.2	0.537	0.171
Std	16	420	420	0	527.2	530.3	0.588	0.176
Std	17	422.1	424.2	0.50	478.5	480.1	0.334	0.226
Std	18	448.8	448.8	0	517.7	518.8	0.212	0.138
Std	19	396	415.8	5	478.7	484.8	1.274	0.221
Std	20	460	460	0	553	556.2	0.579	0.169
Std	21	440	460	4.55	488.7	489.9	0.246	0.198
Std	22	462	462	0	476.8	478.9	0.440	0.147
Std	23	400	400	0	431.4	432.6	0.278	0.112
Std	24	400	400	0	394.6	396.4	0.456	0.203
Std	25	453.1	453.1	0	485.9	487.2	0.268	0.185
Std	26	430.1	430.1	0	503	504.5	0.298	0.109
Std	27	353.52	353.52	0	472.2	473.3	0.233	0.122
Std	28	417.9	417.9	0	534.9	535.9	0.187	0.070
Std	29	417.9	417.9	0	536.4	538.1	0.317	0.127
Std	30	415.8	415.8	0	478	479.1	0.230	0.080
Std	31	440	440	0	486.7	489.6	0.596	0.161
Std	32	416	416	0	494.3	496.2	0.384	0.164
Std	33	435.6	435.6	0	486.9	488.8	0.390	0.146
Std	34	426.3	426.3	0	475.6	476.5	0.189	0.142
Std	35	437.8	437.8	0	541.4	543.4	0.369	0.106
Std	36	433.4	433.4	0	549.3	549.9	0.109	0.180
Std	37	351	351	0	437.4	438.7	0.297	0.175
Std	38	440	462	5	532.4	534.4	0.376	0.132
Std	39	464.6	464.6	0	524.9	527.9	0.572	0.211
Std	40	440	460	4.55	526.3	527.8	0.285	0.134
Std	41	440	440	0	521	522.8	0.345	0.105
Std	42	433.4	433.5	0.02	525.7	527.3	0.304	0.131
Std	43	440	440	0	476.6	479.8	0.671	0.268
Std	44	462.3	462.3	0	526.9	532.1	0.987	0.177
Std	45	440	440	0	485.5	486.3	0.165	0.122
Std	46	435.6	435.6	0	500.1	501.6	0.300	0.122
Std	47	433.4	433.4	0	484.1	485	0.186	0.145
Std	48	440	440	0	522.4	524.8	0.459	0.105
Std	49	392	392	0	502.8	504.1	0.259	0.078
Std	50	437.8	437.8	0	533.7	535	0.244	0.104
Std	51	400	400	0	477.9	479.3	0.293	0.160

Table 6-10: Water absorption percentages of WPC

6.2.1 Comparative analysis

After the analysis, a comparison step between commercial wood and WPC was necessary. It was conducted for the sake of testing the hypothesis of application adequacy based on the flexural and water absorption properties. 7 types of wood were used for comparison. They were tested exactly the same way as the WPC was in flexural and water absorption tests. Same settings, testing machines, conditions, and dimensions of WPC specimens were adopted. In other words, they were treated as WPC. This step was decided to guarantee consistency of results and validation of comparison. Specially that commercial wood samples were obtained from a local wood workshop in Helwan. Therefore, their specific properties could not be obtained from online The obtained properties of these wood types are displayed in table 6-11; where the minimum value of the 5 values of water absorption and thickness swelling test was presented to make a worst case scenario regarding the comparison of WPC. Average and standard deviation (st. dev.) were added in table 6-11 for flexural properties (modulus and strength).sources so the decision was made to do the tests. 5 samples of each wood type were tested; making the total of 35 samples.

Table 6-11: Water absorption and flexural properties of 7 types of commercial wood

	Type	% water absorbed	Humidity %	% volume increase	flex modulus average	flex modulus st. dev.	flex strength average	flex strength st. dev.
1	*Fiber*	83.91	31.34	13.04	2388.53	617.52	16.06	2.84
2	*Pine wood (Mosky)*	21.81	12.25	6.96	13756.50	3284.94	83.30	18.26
3	*Plywood*	25.59	11.84	4.65	4524.85	232.23	17.27	1.69
4	*Compressed wood (Conter)*	35.04	10.41	1.01	2270.78	296.86	9.06	0.76
5	*Beech*	45.88	8.14	8.2	13148.47	813.49	108.63	4.57
6	*MDF*	60.89	7.36	8.69	2259.99	794.74	15.11	2.90
7	*Maple*	14.51	9.68	6.55	10649.21	876.78	78.02	3.28

For doing the comparison, a hypothesis testing is performed for unknown unequal variances with the assumption of equal Mieu values for the null hypothesis explained in table 6-12 as follow (Montgomery and Runger 2003):

Table 6-12: Hypothesis testing procedures table

Null Hypothesis	Test Statistic	Alternative Hypothesis	Criteria of rejection
$H_0: \mu_1 = \mu_2$ $\sigma_1^2 \neq \sigma_2^2 \ unknown$	$$t_0 = \frac{\bar{X}_1 - \bar{X}_2}{\sqrt{\frac{S_1^2}{n_1} + \frac{S_2^2}{n_2}}}$$ $$\nu = \frac{\left(\frac{S_1^2}{n_1} + \frac{S_2^2}{n_2}\right)^2}{\frac{\left(S_1^2/n_1\right)^2}{n_1 + 1} + \frac{\left(S_2^2/n_2\right)^2}{n_2 + 1}} - 2$$	$H_1: \mu_1 \neq \mu_2$ $H_1: \mu_1 > \mu_2$ $H_1: \mu_1 < \mu_2$	$\|t_0\| > t_{\alpha/2,\nu}$ $t_0 > t_{\alpha,\nu}$ $t_0 < -t_{\alpha,\nu}$

Starting by testing the flexural strength; where the 21 specimens found in Appendix 6 were used for comparison as they were the ones used to generate the final fitting equation of the flexural strength. Beech, Maple, and Pine (Mosky) were excluded from the comparison as they show far higher strength (see table 6-11) making the comparison pointless. The significance level is assumed to be 0.05 (α=0.05); as a default level set (Design-Expert 2010).

Table 6-13 presents the results of the comparison between the WPC and compressed wood (Conter) based the procedures in table 6-12:

Table 6-13: Hypothesis testing comparison of the flexural strength of WPC and compressed wood

$\overline{X_1}$	9.065	WPC
$\overline{X_2}$	9.057	Compressed wood (Conter)
S_1^2	4.534	WPC
S_2^2	0.570	Compressed wood (Conter)
n_1	21	WPC
n_2	5	Compressed wood (Conter)
t_0	0.015	
$t_{\alpha/2,\nu}$	2.064	α=0.05 , ν=24
ν	23.395	
Then,	$|t_0| < t_{\alpha/2,\nu}$	
	Therefore,	**μ1 = μ2**

The results show that the WPC performs as the compressed wood (Conter) in flexural strength; where equal μ's were proven. The same steps were applied to the other 3 wood types and the final results showed that the μ of WPC is less than the μ of fiber wood, MDF, and plywood.

Going to the flexural modulus; where the 23 specimens found in Appendix 5 were used for comparison as they were the ones used to generate the final fitting equation of the flexural modulus. Beech, Maple, and Plywood were excluded from the comparison as they show far higher modulus (see table 6-11) making the comparison pointless. Table 6-14 presents the results of the comparison between the WPC and compressed wood (Conter) based the procedures in table 6-12:

Table 6-14: Hypothesis testing comparison of the flexural modulus of WPC and compressed wood

$\overline{X_1}$	1061.259	WPC
$\overline{X_2}$	2270.782	Compressed wood (Conter)
S_1^2	80153.642	WPC
S_2^2	88123.511	Compressed wood (Conter)
n_1	23	WPC
n_2	5	Compressed wood (Conter)
t_0	-8.325	
$t_{\alpha/2,\nu}$	2.365	α=0.05
ν	6.524	
Then,		$\|t_0\| > t_{\alpha/2,\nu}$
	Therefore,	μ1 not equal μ2
	$t_{\alpha,\nu}$	1.895
	$-t_{\alpha,\nu}$	-1.895
	Therefore,	$t_0 < -t_{\alpha,\nu}$
	Then	μ1 < μ2

The results show that the WPC performs less than the compressed wood (Conter) in flexural modulus; where equal μ's were not proven. The same steps were applied to the other 3 wood types and the final results showed that the μ of WPC is less than the μ of fiber wood, MDF, and Pine wood (Mosky). Therefore, WPC has proven a less flexural modulus than the 7 wood types; however, the values were not that far regarding fiber, MDF, and compressed wood (Conter). In addition, it has proven adequate flexural strength when compared to compressed wood.

Finally, comparing the water absorption was not necessary as percentages of the 7 wood types shown in table 6-11 were far higher than WPC which didn't call for doing any test. However, the minimum value of volume increase percentage of compressed wood (Conter) was relatively close to the corresponding WPC percentages. Therefore, it was necessary to check up the detailed values of the compressed wood. It was found that its maximum value was 6.92%, range of 5.91%, average of 3.7%, and a standard deviation of 2.43% (see table 6-15).

When comparing these results to the corresponding results of WPC (table 6-15), it could be concluded that the compressed wood has a low minimum value of volume increase percentage; however, its mean and standard deviation has shown higher percentages than WPC which tells that its volume is much more affected with water and didn't call for further analysis.

Table 6-15: volume increase percentages of WPC and compressed wood (Conter)

% of volume increase from water absorption of Compressed wood (conter)				
min	max	Range	Average	ST dev
1.01	6.92	5.91	3.73	2.44
% of volume increase from water absorption of WPC				
min	max	Range	Average	ST dev
0	5	5	0.45	1.38

CHAPTER 7 CONCLUSION & RECOMMENDATION

Based on the analysis, it could be said that models were proven to be accepted and their representation of runs was quite satisfying; however some modifications could be added to increase the adequacy of these models. The main thing needed to be worked at is the plastic utilized. It was noted from the ANOVA that there is a high plastic effect in the flexural modulus and strength models. This was a sign for a needed follow-up for the source of this plastic. First of all, it was clear that there is a block or batch effect which was explained in chapter 6. These blocks were the batches of plastic used during experimentations. Other factors; including wood and talc, were not considered in blocks' effect as they were obtained from one batch. Therefore, this is a primary indicator for the reason of plastic effect; where there is a variation from producing a batch to the other at the manufacturer's site. Yet, this wasn't the main reason behind the high effect of plastic. Conversely, the major cause was the high variability at the source of this plastic, where it is obtained from the reject of rejects waste (mainly contaminated plastic bags: a mix of high and low density polyethylene). This factor could have been avoided if the plastic producer washed it in a suitable way after sorting from other wastes. Moreover, better plastic could be obtained if there was an available garbage sorting policy in Egypt. However, if this product was decided to be extended to production scale, a collaborative joint effort between the plastic's producer and the manufacturer of WPC could be established to guarantee the best possible plastic. It could include better sorting policies at the source, site, and addition of more washing steps.

The addition of talc has benefited the mechanical properties; flexural strength and modulus, in a noticeable way. It is seen from figure 6-5; presenting the 3-D response surface of flexural modulus, that the orientation of the surface towards highest values of the modulus when the talc had a high percentage of 35%, plastic lowest percentage of 40%, wood (C) and (D) average percentages of 15% and 10%. In addition, the same effect was noticed in the 3-D graph of flexural strength (figure 6-9); when the talc had a high percentage of 30%, but plastic 50%, wood (C) and (D); 10% and 10%, had average percentages which showed that strength tended to the maximum. Therefore, talc was a beneficial addition to the mixture where it reflected good outcomes; in addition, it showed no specific effect which was apparent in the two models during the ANOVA and the other analysis.

Wood with size of up to 0.5mm (D) has reflected a negative effect on flexural modulus; which was unexpected at the beginning of the research. This was apparent in the significant value of (D^2) shown in ANOVA table of the flexural modulus; however, average values of D (based on the constraint and limits of this component in this paper) has proven to get the highest values of flexural strength and modulus as shown in the 3-D response graphs (see figure 6-5 and 6-9). On the other hand, wood with size of up to 1.18mm (C) performed in an acceptable way which was doubtful especially with its relative large grain sizes which wasn't common in literature.

As a consequence, the results were quite satisfying; even with some considerations, regarding the plastic utilized. However, it was shown that the WPC water absorption properties were superior regarding its minimal percentages of water absorbed,

dimensional change, and humidity content. This was further validated when the comparison was made to commercial wood types, which rings the bell for an incoming strong competitor in the areas where wood is subjected to water. Outdoor applications, fences, sheets, pools sides coverings, and many other areas related water presence could be a potential market. The flexural strength outcome was quite encouraging; especially, WPC has proven equal in means when compared to compressed wood. However, the moduli were lower than commercial woods; yet, relatively close.

The fact that we are dealing with a product resulted from waste has several environmental and cost benefits. From the narrow environmental point of view, the plastic and wood wastes are quite a problem needed to be solved where the main way of getting rid of them is done by throwing them away in a dumpsite; bearing in mind that the plastic is a non- biodegradable waste. As a global environmental view, the recycling of plastic will save much energy, power, and pollution exerted to get and process oil. On the other hand, using wood waste will save a considerable amount of trees and will increase the Green environment. As a cost benefit, these wastes are non valuable products or priceless products which could be an asset for a big industry generating much outcome for its investors without money on raw material spent.

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APPENDIX 1: SAMPLE OF THE STRESS-STRAIN DIAGRAMS (STD 28 AND STD49 - CHECK APPENDIX 2 FOR MIXTURE'S INGREDIENTS) GENERATED BY THE SOTWARE OF THE TESTING MACHINE (INSTRON - BLUEHILL LITE)

Flex Test
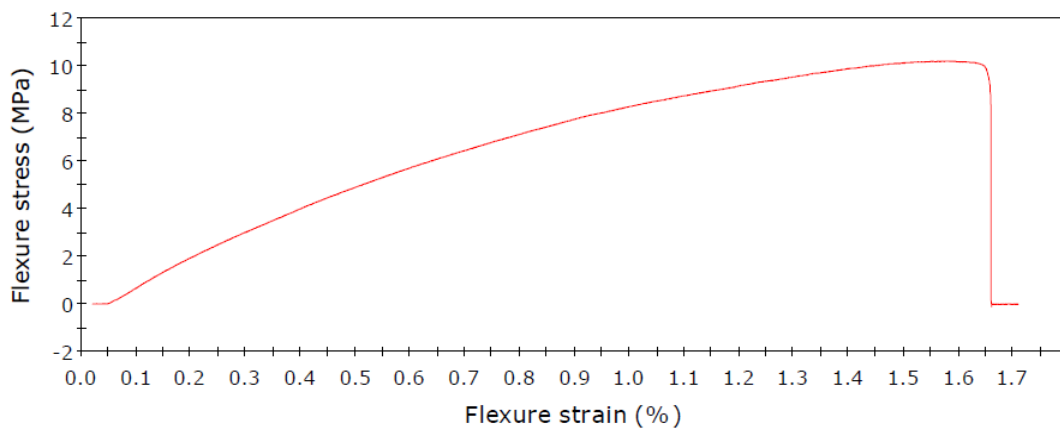
Flex Test
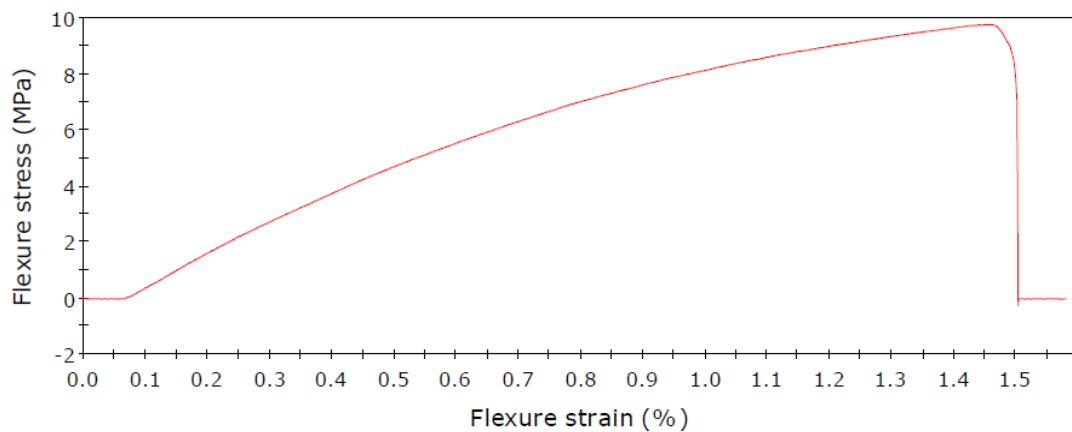
APPENDIX 2: FULL DESIGNED GENERATED BY DESIGN-EXPERT INCLUDING THE OUTPUT OF THE TESTING MACHINE (INSTRON - BLUEHILL LITE)

Std	Block	Type	A (Plastic)	B (Talc)	C (Wood 1.18)	D (Wood 0.5)	Flexural Modulus	Flexural Stress
			%	%	%	%	(Mpa)	(Mpa)
1	1	Vertex	50.0	0.0	50.0	0.0	829.7	7.1
2	1	Vertex	50.0	0.0	0.0	50.0	878.6	8.9
3	1	Vertex	40.0	20.0	40.0	0.0	692.7	6.4
4	1	Vertex	40.0	20.0	0.0	40.0	1826.0	7.8
5	1	Vertex	70.0	0.0	30.0	0.0	690.2	4.7
6	2	Vertex	70.0	15.0	15.0	0.0	579.3	7.3
7	1	Vertex	70.0	0.0	0.0	30.0	513.6	6.2
8	2	Vertex	70.0	15.0	0.0	15.0	811.4	8.7
9	2	Vertex	40.0	35.0	25.0	0.0	1118.4	9.4
10	2	Vertex	50.0	35.0	15.0	0.0	1328.4	10.0
11	3	Vertex	40.0	35.0	0.0	25.0	856.8	7.8
12	3	Vertex	50.0	35.0	0.0	15.0	1113.7	10.8
13	1	CentEdge	50.0	0.0	25.0	25.0	866.1	7.9
14	2	CentEdge	45.0	10.0	45.0	0.0	1200.0	9.6
15	1	CentEdge	60.0	0.0	40.0	0.0	1032.8	9.4
16	2	CentEdge	45.0	10.0	0.0	45.0	1272.0	9.5
17	1	CentEdge	60.0	0.0	0.0	40.0	1114.6	11.1
18	3	CentEdge	40.0	20.0	20.0	20.0	1249.9	9.0
19	2	CentEdge	40.0	27.5	32.5	0.0	1499.1	9.9
20	2	CentEdge	40.0	27.5	0.0	32.5	1585.0	10.1
21	3	CentEdge	70.0	7.5	22.5	0.0	1052.0	10.9
22	3	CentEdge	70.0	0.0	15.0	15.0	771.8	8.3
23	3	CentEdge	70.0	15.0	7.5	7.5	564.3	6.8
24	1	CentEdge	60.0	25.0	15.0	0.0	433.1	5.0
25	3	CentEdge	70.0	7.5	0.0	22.5	953.7	10.1
26	1	CentEdge	60.0	25.0	0.0	15.0	1095.6	12.5
27	3	CentEdge	45.0	35.0	20.0	0.0	922.9	9.0
28	3	CentEdge	40.0	35.0	12.5	12.5	1184.7	10.2
29	3	CentEdge	50.0	35.0	7.5	7.5	1007.9	9.5
30	3	CentEdge	45.0	35.0	0.0	20.0	604.1	6.4
31	1	PlaneCent	60.0	25.0	7.5	7.5	833.4	10.0
32	3	PlaneCent	45.0	10.0	22.5	22.5	850.0	8.9
33	3	PlaneCent	40.0	27.5	16.3	16.3	1104.4	12.2
34	3	PlaneCent	70.0	7.5	11.3	11.3	910.8	10.2

Std	Block	Type	A (Plastic) %	B (Talc) %	C (Wood 1.18) %	D (Wood 0.5) %	Flexural Modulus (Mpa)	Flexural Stress (Mpa)
35	3	PlaneCent	60.0	0.0	20.0	20.0	1790.8	11.9
36	2	PlaneCent	45.0	35.0	10.0	10.0	1178.8	9.2
37	3	PlaneCent	53.3	17.5	0.0	29.2	905.7	9.5
38	3	PlaneCent	53.3	17.5	29.2	0.0	1159.0	11.6
39	2	AxialCB	51.7	8.8	32.3	7.3	1316.3	10.8
40	3	AxialCB	51.7	8.8	7.3	32.3	1548.6	13.0
41	2	AxialCB	46.7	18.8	27.3	7.3	1350.7	11.3
42	3	AxialCB	46.7	18.8	7.3	27.3	1573.2	10.0
43	2	AxialCB	61.7	8.8	22.3	7.3	956.6	8.6
44	2	AxialCB	61.7	16.3	14.8	7.3	1197.5	10.8
45	3	AxialCB	61.7	8.8	7.3	22.3	960.6	10.3
46	3	AxialCB	61.7	16.3	7.3	14.8	970.8	10.4
47	2	AxialCB	46.7	26.3	19.8	7.3	905.2	9.7
48	2	AxialCB	51.7	26.3	14.8	7.3	1056.6	10.2
49	3	AxialCB	46.7	26.3	7.3	19.8	1182.0	9.8
50	3	AxialCB	51.7	26.3	7.3	14.8	1208.3	10.7
51	3	Center	53.3	17.5	14.6	14.6	936.4	9.1
R 1	3	Vertex	50.0	0.0	50.0	0.0	1303.5	12.9
R 4	1	Vertex	40.0	20.0	0.0	40.0	1409.1	12.2
R 9	1	CentEdge	60.0	25.0	15.0	0.0	292.6	4.9
R 8	2	AxialCB	46.7	26.3	19.8	7.3	893.4	8.7
R 5	3	CentEdge	50.0	0.0	0.0	50.0	1293.6	11.3
R 2	3	CentEdge	50.0	0.0	25.0	25.0	1164.8	10.4
R 7	2	Vertex	70.0	15.0	15.0	0.0	572.2	6.8
R 3	3	Vertex	70.0	0.0	0.0	30.0	589.9	7.2
R 6	3	CentEdge	70.0	0.0	15.0	15.0	735.1	8.2
R 10	1	Vertex	70.0	0.0	0.0	30.0	353.6	5.4

APPENDIX 3: WATER ABSORPTION AND THICKNESS SWELLING TEST RESULTS

No. Specimen		cm^3 Volume before	cm^3 Volume after	% Volume increase	gm Weight before	gm Weight After	% Weight increase	% Humidity	cm Thickness before	cm Thickness After	% Thickness increase
R	1	440	440	0	494.1	496.2	0.425	0.168	1.0	1.0	0.0
R	2	394	394	0	457.4	459.2	0.394	0.130	1.1	1.1	0.0
R	3	396	396.8	0.20	397.7	398.7	0.251	0.175	1.0	1.0	0.0
R	4	420	420	0	499.2	500.6	0.280	0.232	1.1	1.1	0.0
R	5	417.9	420	0.50	470.4	472.2	0.383	0.122	1.1	1.1	0.0
Std	1	400	400	0	434.7	442.2	1.725	0.223	1.0	1.0	0.0
Std	2	433.4	433.4	0	421.8	423.7	0.450	0.220	1.2	1.2	0.0
Std	3	411.6	411.6	0	431.6	434.5	0.672	0.224	1.1	1.1	0.0
Std	4	370	370	0	435	437.1	0.483	0.441	1.1	1.2	4.5
Std	5	396	396	0	417.3	418.9	0.383	0.191	1.1	1.1	0.0
Std	6	396	396	0	450.5	450.9	0.089	0.133	1.0	1.0	0.0
Std	7	400	400	0	413.9	415.1	0.290	0.193	1.1	1.1	0.0
Std	8	436.48	436.48	0	500	500.5	0.100	0.100	1.1	1.1	0.0
Std	9	464.6	464.6	0	546.3	547.8	0.275	0.146	1.1	1.1	0.0
Std	10	440	440	0	561.9	562.6	0.125	0.134	1.0	1.0	0.0
Std	11	378.1	378.1	0	476.9	478.5	0.336	0.132	1.1	1.1	0.0
Std	12	398	398	0	542.8	543.5	0.129	0.181	1.1	1.1	0.0
Std	13	402	422.1	5	442.4	446.7	0.972	0.220	1.1	1.1	0.0
Std	14	464.6	464.6	0	538.6	542	0.631	0.198	1.1	1.1	0.0
Std	15	455.4	455.4	0	465.7	468.2	0.537	0.171	1.1	1.1	0.0
Std	16	420	420	0	527.2	530.3	0.588	0.176	1.1	1.1	0.0
Std	17	422.1	424.2	0.50	478.5	480.1	0.334	0.226	0.9	0.9	0.0
Std	18	448.8	448.8	0	517.7	518.8	0.212	0.138	1.1	1.1	0.0
Std	19	396	415.8	5	478.7	484.8	1.274	0.221	1.0	1.0	0.0
Std	20	460	460	0	553	556.2	0.579	0.169	1.1	1.1	0.0
Std	21	440	460	4.55	488.7	489.9	0.246	0.198	1.0	1.0	0.0
Std	22	462	462	0	476.8	478.9	0.440	0.147	1.1	1.1	0.0
Std	23	400	400	0	431.4	432.6	0.278	0.112	1.1	1.1	0.0
Std	24	400	400	0	394.6	396.4	0.456	0.203	1.0	1.0	0.0
Std	25	453.1	453.1	0	485.9	487.2	0.268	0.185	1.1	1.1	0.0
Std	26	430.1	430.1	0	503	504.5	0.298	0.109	1.1	1.2	4.5
Std	27	353.52	353.52	0	472.2	473.3	0.233	0.122	1.1	1.1	0.0
Std	28	417.9	417.9	0	534.9	535.9	0.187	0.070	1.2	1.2	0.0
Std	29	417.9	417.9	0	536.4	538.1	0.317	0.127	1.1	1.1	0.0
Std	30	415.8	415.8	0	478	479.1	0.230	0.080	1.1	1.1	0.0

No. Specimen	cm^3 Volume before	cm^3 Volume after	% Volume increase	gm Weight before	gm Weight After	% Weight increase	% Humidity	cm Thickness before	cm Thickness After	% Thickness increase
Std 31	440	440	0	486.7	489.6	0.596	0.161	1.0	1.0	0.0
Std 32	416	416	0	494.3	496.2	0.384	0.164	1.2	1.2	0.0
Std 33	435.6	435.6	0	486.9	488.8	0.390	0.146	1.1	1.1	0.0
Std 34	426.3	426.3	0	475.6	476.5	0.189	0.142	1.1	1.1	0.0
Std 35	437.8	437.8	0	541.4	543.4	0.369	0.106	1.2	1.2	0.0
Std 36	433.4	433.4	0	549.3	549.9	0.109	0.180	1.2	1.2	0.0
Std 37	351	351	0	437.4	438.7	0.297	0.175	1.2	1.2	0.0
Std 38	440	462	5	532.4	534.4	0.376	0.132	1.0	1.0	0.0
Std 39	464.6	464.6	0	524.9	527.9	0.572	0.211	1.1	1.1	0.0
Std 40	440	460	4.55	526.3	527.8	0.285	0.134	1.2	1.2	0.0
Std 41	440	440	0	521	522.8	0.345	0.105	1.1	1.1	0.0
Std 42	433.4	433.5	0.02	525.7	527.3	0.304	0.131	1.0	1.1	5.0
Std 43	440	440	0	476.6	479.8	0.671	0.268	1.2	1.2	0.0
Std 44	462.3	462.3	0	526.9	532.1	0.987	0.177	1.1	1.1	0.0
Std 45	440	440	0	485.5	486.3	0.165	0.122	1.0	1.1	5.0
Std 46	435.6	435.6	0	500.1	501.6	0.300	0.122	0.9	0.9	0.0
Std 47	433.4	433.4	0	484.1	485	0.186	0.145	1.0	1.0	0.0
Std 48	440	440	0	522.4	524.8	0.459	0.105	1.0	1.0	0.0
Std 49	392	392	0	502.8	504.1	0.259	0.078	1.0	1.0	0.0
Std 50	437.8	437.8	0	533.7	535	0.244	0.104	1.1	1.1	0.0
Std 51	400	400	0	477.9	479.3	0.293	0.160	1.0	1.0	0.0

APPENDIX 4: INITIAL DESIGN GENERATED BY DESIGN-EXPERT FOR THE FLEXURAL MODULUS AND STRENGTH (1ST STEP IN THE DESIGN SPACE NAVIGATION ALGORITHM)

Std	Run	Block	Type	Comments	A (Plastic) %	B (Talc) %	C (Wood 1.18) %	D (Wood 0.5) %	Flexural Modulus (Mpa)	Flexural Stress (Mpa)
3	1	Block 3	Plane	std 38	53.33	17.50	29.17	0.00	1159	11.59
13	2	Block 3	Vertex	std 18	40.00	20.00	20.00	20.00	1249.85	9.02
6	3	Block 1	Vertex	std 1	50.00	0.00	50.00	0.00	829.68	7.13
12	4	Block 3	CentEdge	std 22	70.00	0.00	15.00	15.00	771.83	8.29
22	5	Block 3	Vertex	R5	50.00	0.00	0.00	50.00	1293.64	11.27
21	6	Block 1	Vertex	std 2	50.00	0.00	0.00	50.00	878.68	8.92
10	7	Block 2	Vertex	std 8	70.00	15.00	0.00	15.00	811.38	8.74
9	8	Block 3	Edge	std 28	40.00	35.00	12.50	12.50	1184.68	10.18
5	9	Block 1	Vertex	std 3	40.00	20.00	40.00	0.00	692.68	6.37
14	10	Block 1	Vertex	std 13	50.00	0.00	25.00	25.00	866.08	7.87
4	11	Block 1	Vertex	std 5	70.00	0.00	30.00	0.00	690.16	4.65
16	12	Block 1	Vertex	std 7	70.00	0.00	0.00	30.00	513.63	6.16
11	13	Block 3	Vertex	std 12	50.00	35.00	0.00	15.00	1113.71	10.75
19	14	Block 1	Vertex	std 4	40.00	20.00	0.00	40.00	1825.97	7.8
8	15	Block 2	Interior	std 48	51.67	26.25	14.79	7.29	1056.56	10.2
20	16	Block 1	Vertex	R4	40.00	20.00	0.00	40.00	1409.1	12.15
7	17	Block 3	Vertex	R1	50.00	0.00	50.00	0.00	1303.45	12.85
2	18	Block 2	Vertex	std 9	40.00	35.00	25.00	0.00	1118.44	9.39
1	19	Block 2	Vertex	std 6	70.00	15.00	15.00	0.00	579.29	7.29
17	20	Block 3	Vertex	R3	70.00	0.00	0.00	30.00	589.93	7.21
18	21	Block 3	Interior	std 40	51.67	8.75	7.29	32.29	1548.61	13.03
15	22	Block 3	Vertex	R2	50.00	0.00	25.00	25.00	1164.82	10.44

APPENDIX 5: FINAL DESIGN OBTAINED FROM DESIGN-EXPERT FOR THE FLEXURAL MODULUS

Std	Run	Block	Type	Comments	A (Plastic)	B (Talc)	C (Wood 1.18)	D (Wood 0.5)	Flexural Modulus
				Standard number used in Appendix 2	%	%	%	%	Mpa
22	1	Block 1	Vertex	Std 1	50.00	0.00	50.00	0.00	829.68
8	2	Block 1	Vertex	Std 7	70.00	0.00	0.00	30.00	513.63
12	3	Block 1	Edge	Std 15	60.00	0.00	40.00	0.00	1032.84
11	4	Block 1	Vertex	Std 5	70.00	0.00	30.00	0.00	690.16
15	5	Block 1	Edge	Std 17	60.00	0.00	0.00	40.00	1114.56
9	13	Block 1	Vertex	R 4	40.00	20.00	0.00	40.00	1409.1
17	6	Block 2	Vertex	Std 8	70.00	15.00	0.00	15.00	811.38
2	7	Block 2	Interior	Std 48	51.67	26.25	14.79	7.29	1056.56
16	8	Block 2	Vertex	Std 14	45.00	10.00	45.00	0.00	1199.97
10	9	Block 2	Vertex	Std 10	50.00	35.00	15.00	0.00	1328.36
13	10	Block 3	Interior	Std 45	61.67	8.75	7.29	22.29	960.57
5	11	Block 3	CentEdge	Std 22	70.00	0.00	15.00	15.00	771.83
14	12	Block 3	Plane	Std 37	53.33	17.50	0.00	29.17	905.66
1	14	Block 3	Plane	Std 38	53.33	17.50	29.17	0.00	1159
7	15	Block 3	Vertex	R 2	50.00	0.00	25.00	25.00	1164.82
18	16	Block 3	Interior	Std 49	46.67	26.25	7.29	19.79	1182.02
4	17	Block 3	Vertex	Std 12	50.00	35.00	0.00	15.00	1113.71
20	18	Block 3	Vertex	Std 13	50.00	0.00	25.00	25.00	866.06
6	19	Block 3	Vertex	Std 18	40.00	20.00	20.00	20.00	1249.85
19	20	Block 3	Vertex	Std 4	40.00	20.00	0.00	40.00	1825.97
21	21	Block 3	Vertex	R 1	50.00	0.00	50.00	0.00	1303.45
3	22	Block 3	Edge	Std 28	40.00	35.00	12.50	12.50	1184.68
23	23	Block 3	CentEdge	R 6	70.00	0.00	15.00	15.00	735.1

APPENDIX 6: FINAL DESIGN OBTAINED FROM DESIGN-EXPERT FOR THE FLEXURAL STRENGTH

Std	Run	Block	Type	Comments	A (Plastic)	B (Talc)	C (Wood 1.18)	D (Wood 0.5)	Flexural Modulus
				Standard number used in Appendix 2	%	%	%	%	Mpa
13	1	Block 1	Vertex	std 4	40.00	20.00	0.00	40.00	7.8
4	2	Block 1	Vertex	std 3	40.00	20.00	40.00	0.00	6.37
3	3	Block 1	Vertex	std 5	70.00	0.00	30.00	0.00	4.65
21	4	Block 1	Vertex	R 4	40.00	20.00	0.00	40.00	12.15
14	5	Block 1	Vertex	std 2	50.00	0.00	0.00	50.00	8.92
9	6	Block 1	Vertex	std 13	50.00	0.00	25.00	25.00	7.87
11	7	Block 1	Vertex	std 7	70.00	0.00	0.00	30.00	6.16
16	8	Block 1	Edge	std 17	60.00	0.00	0.00	40.00	11.09
20	9	Block 1	Vertex	R 3	70.00	0.00	0.00	30.00	7.21
1	10	Block 2	Vertex	std 6	70.00	15.00	15.00	0.00	7.29
6	11	Block 2	Vertex	std 8	70.00	15.00	0.00	15.00	8.74
19	12	Block 2	Interior	std 43	61.67	8.75	22.29	7.29	8.62
18	13	Block 2	Interior	std 47	46.67	26.25	19.79	7.29	9.71
17	14	Block 2	Vertex	std 10	50.00	35.00	15.00	0.00	9.97
12	15	Block 3	Interior	std 40	51.67	8.75	7.29	32.29	13.03
8	16	Block 3	Vertex	std 18	40.00	20.00	20.00	20.00	9.02
2	17	Block 3	Plane	std 38	53.33	17.50	29.17	0.00	11.59
7	18	Block 3	CentEdge	std 22	70.00	0.00	15.00	15.00	8.29
5	19	Block 3	Edge	std 28	40.00	35.00	12.50	12.50	10.18
15	20	Block 3	Vertex	R 5	50.00	0.00	0.00	50.00	11.27
10	21	Block 3	Vertex	R 2	50.00	0.00	25.00	25.00	10.44

APPENDIX7: DIAGNOSTIC CASE STATISTICS FOR FLEXURAL MODULUS

Standard Order	Actual Value	Predicted Value	Residual	Leverage	Internally Studentized Residual	Externally Studentized Residual	Influence on Fitted Value DFFITS	Cook's Distance	Run Order
1	1159	1249.634	-90.634	0.345022	-0.828921885	-0.82040949	-0.595444193	0.051707	14
2	1056.56	1077.415	-20.8546	0.279016	-0.181791743	-0.176201165	-0.109612524	0.001827	7
3	1184.68	1220.948	-36.2681	0.310261	-0.323235151	-0.313997981	-0.210595105	0.006714	22
4	1113.71	1093.162	20.54789	0.298829	0.181631431	0.176045462	0.114927668	0.002009	17
5	771.83	752.8713	18.95869	0.262879	0.16344607	0.158388259	0.094586977	0.001361	11
6	1249.85	1159.549	90.30128	0.25581	0.774796762	0.764675961	0.448326762	0.029479	19
7	1164.82	1026.993	137.8265	0.251824	1.17941517	1.195094902	0.693345219	0.066885	15
8	513.63	615.9429	-102.313	0.353467	-0.941826141	-0.938299647	-0.693778009	0.069279	2
9	1409.1	1601.746	-192.646	0.366443	-1.791438613	-1.93999046	-1.475400277	0.265171	13
10	1328.36	1295.596	32.76433	0.383001	0.308741245	0.299831895	0.236230122	0.008453	9
11	690.16	729.9932	-39.8332	0.319367	-0.357375306	-0.347416525	-0.237979406	0.008561	4
12	1032.84	842.8768	189.9632	0.259135	1.633562154	1.732775093	1.024792215	0.13334	3
13	960.57	893.7738	66.7962	0.133835	0.531235614	0.518963828	0.203996049	0.006229	10
14	905.66	1117.759	-212.099	0.108889	-1.663059008	-1.770532372	-0.618913317	0.04828	12
15	1114.56	1036.297	78.26331	0.4214	0.761561235	0.751117581	0.641012252	0.060343	5
16	1199.97	1270.235	-70.2649	0.443961	-0.697463699	-0.685822489	-0.612818466	0.055486	8
17	811.38	753.0249	58.35514	0.419377	0.566849545	0.554445243	0.471209594	0.033155	6
18	1182.02	1103.774	78.24628	0.174353	0.637385618	0.625133469	0.287270297	0.012256	16
19	1825.97	1601.746	224.2244	0.366443	2.085094082	2.365723129	1.799178208	0.359231	20
20	866.06	1026.993	-160.933	0.251824	-1.377147086	-1.420244221	-0.823967652	0.091192	18
21	1303.45	1230	73.44994	0.353243	0.676015276	0.664101818	0.490795728	0.035657	21
22	829.68	955.7604	-126.08	0.378739	-1.18398548	-1.20016974	-0.937078708	0.122085	1
23	735.1	752.8713	-17.7713	0.262879	-0.153209534	-0.148453429	-0.088654053	0.001196	23

Note: Predicted values include block corrections.

# APPENDIX 8: DIAGNOSTIC CASE STATISTICS FOR FLEXURAL STRENGTH

Standard Order	Actual Value	Predicted Value	Residual	Leverage	Internally Studentized Residual	Externally Studentized Residual	Influence on Fitted Value DFFITS	Cook's Distance	Run Order
1	7.29	7.06142	0.22858	0.537974	0.2946374	0.2820408	0.3043402	0.010108	10
2	11.59	11.90755	-0.31755	0.752067	-0.5587549	-0.5404771	-0.9413231	0.094703	17
3	4.65	4.440341	0.209659	0.526705	0.2670115	0.2554145	0.2694408	0.007934	3
4	6.37	6.287865	0.082135	0.843384	0.1818426	0.1736413	0.4029474	0.017807	2
5	10.18	10.09752	0.082481	0.614705	0.1164237	0.1110741	0.1402975	0.002163	19
6	8.74	8.810366	-0.07037	0.614506	-0.0992974	-0.0947188	-0.1195889	0.001572	11
7	8.29	7.711166	0.578834	0.387614	0.6480735	0.6300593	0.5012666	0.026584	18
8	9.02	9.819006	-0.79901	0.412743	-0.9135212	-0.9060553	0.7595919	0.058653	16
9	7.87	8.176877	-0.30688	0.50504	-0.3821746	-0.3668327	-0.370549	0.014903	6
10	10.44	9.931326	0.508674	0.430525	0.5905886	0.5722499	0.4975629	0.026369	21
11	6.16	7.300235	-1.14023	0.29521	-1.1900017	-1.2155603	-0.7867064	0.059315	7
12	13.03	12.98783	0.042167	0.382741	0.0470243	0.0448404	0.0353092	0.000137	15
13	7.8	9.533947	-1.73395	0.3532	-1.8890067	-2.1912441	-1.6192565	0.194857	1
14	8.92	9.611156	-0.69116	0.424328	-0.7981257	-0.7840227	-0.6731196	0.046954	5
15	11.27	11.36561	-0.09561	0.701612	-0.1533461	-0.1463663	-0.2244393	0.005529	20
16	11.09	10.0354	1.054603	0.300539	1.104817	1.1172097	0.7323249	0.052447	8
17	9.97	10.08698	-0.11698	0.627386	-0.1679003	-0.1602922	-0.2079936	0.004747	14
18	9.71	9.648381	0.061619	0.303242	0.0646777	0.0616795	0.0406906	0.000182	13
19	8.62	8.722856	-0.10286	0.338068	-0.110766	-0.1056702	-0.0755175	0.000627	12
20	7.21	7.300235	-0.09024	0.29521	-0.0941737	-0.0898273	-0.0581359	0.000371	9
21	12.15	9.533947	2.616053	0.3532	2.8499953	5.3129341	* 3.93	0.443545	4

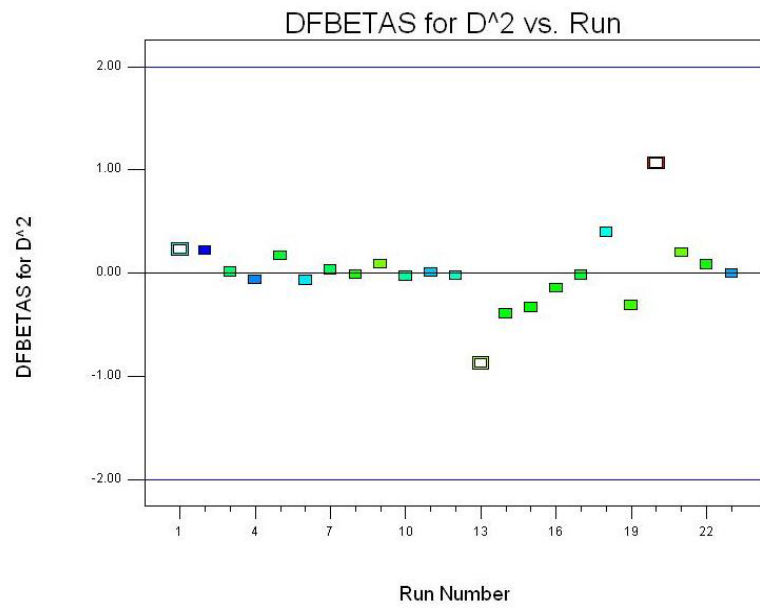Note: Predicted values include block corrections.

* Exceeds limits

APPENDIX 9: FLEXURAL MODULUS DFBETAS GRAPHS FOR EACH COEFFICIENT
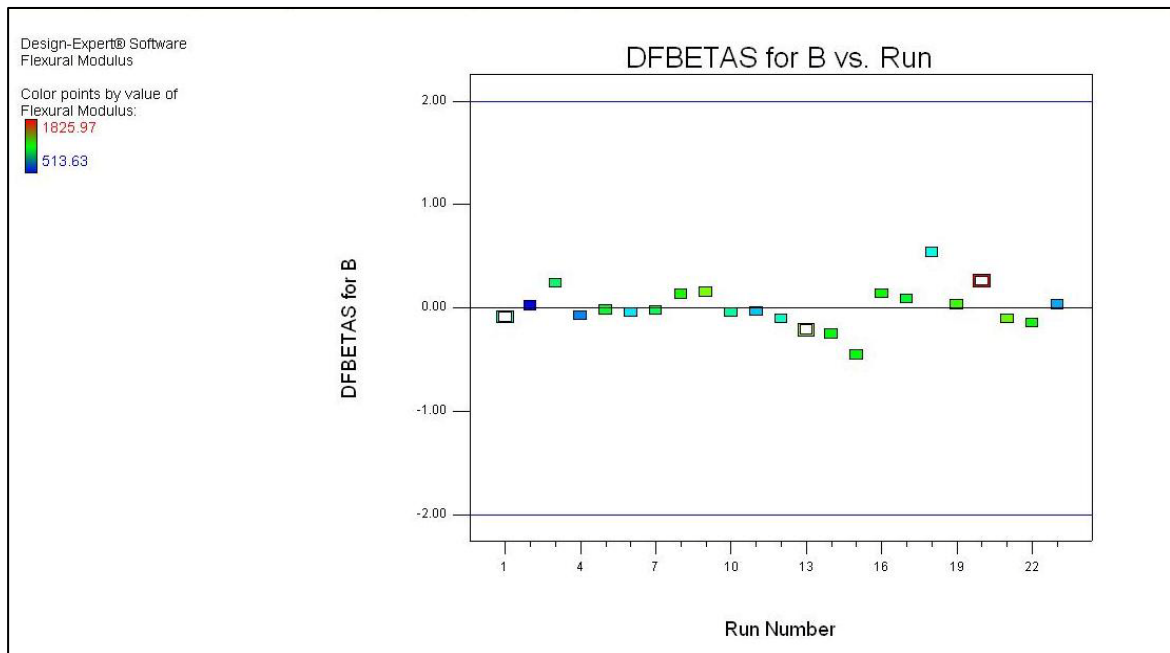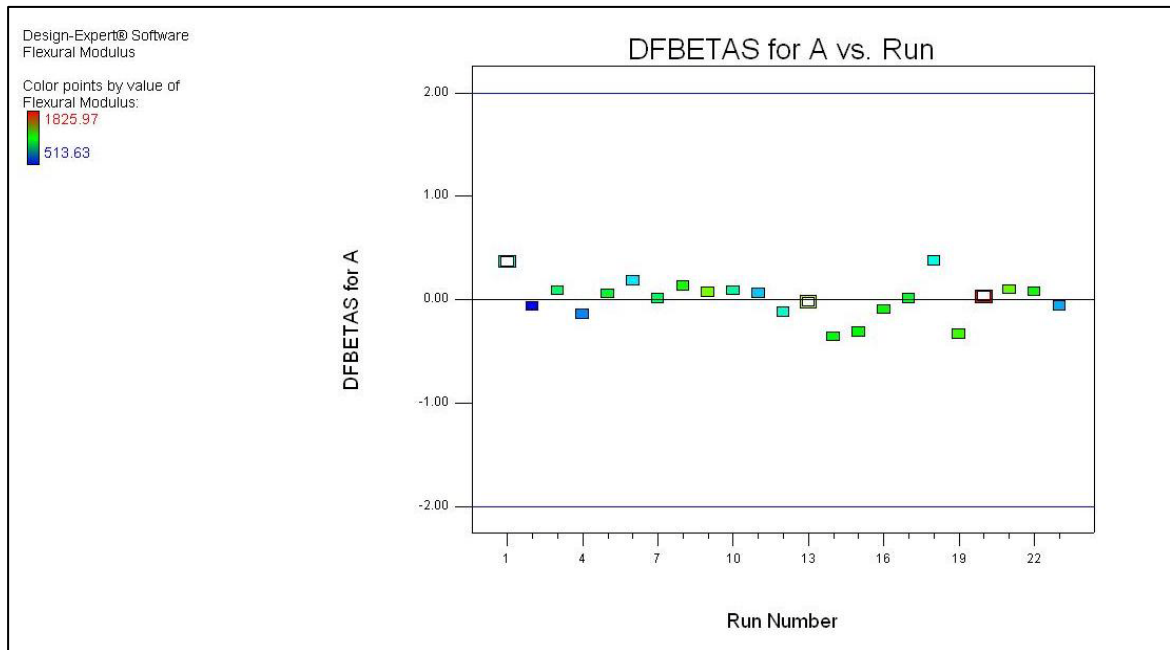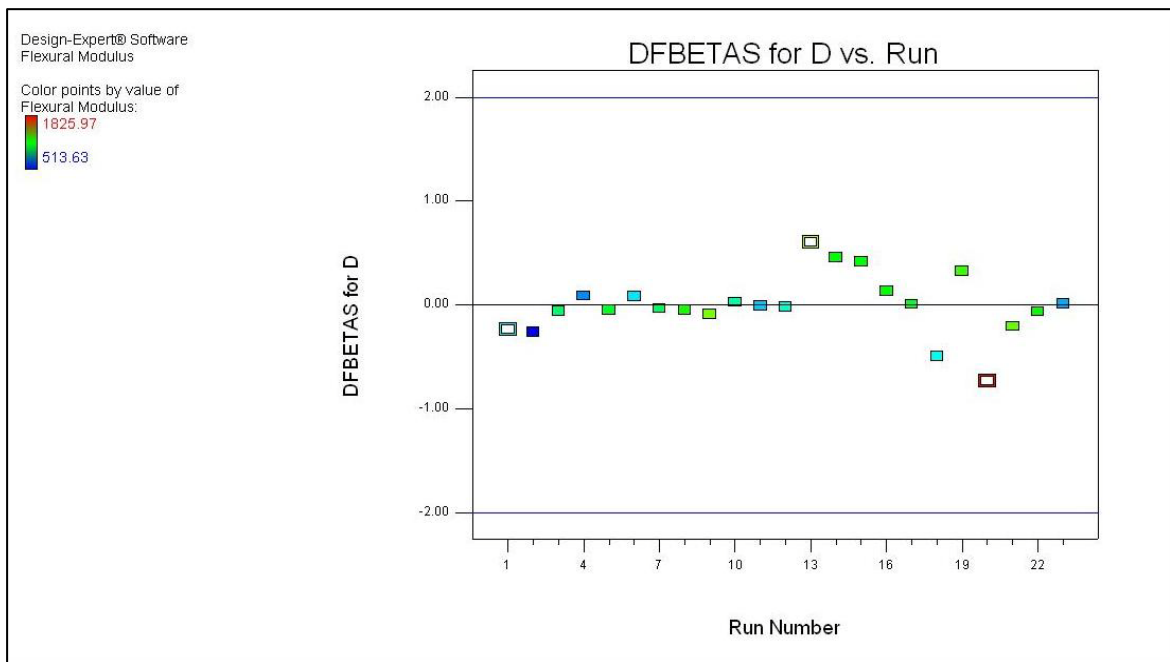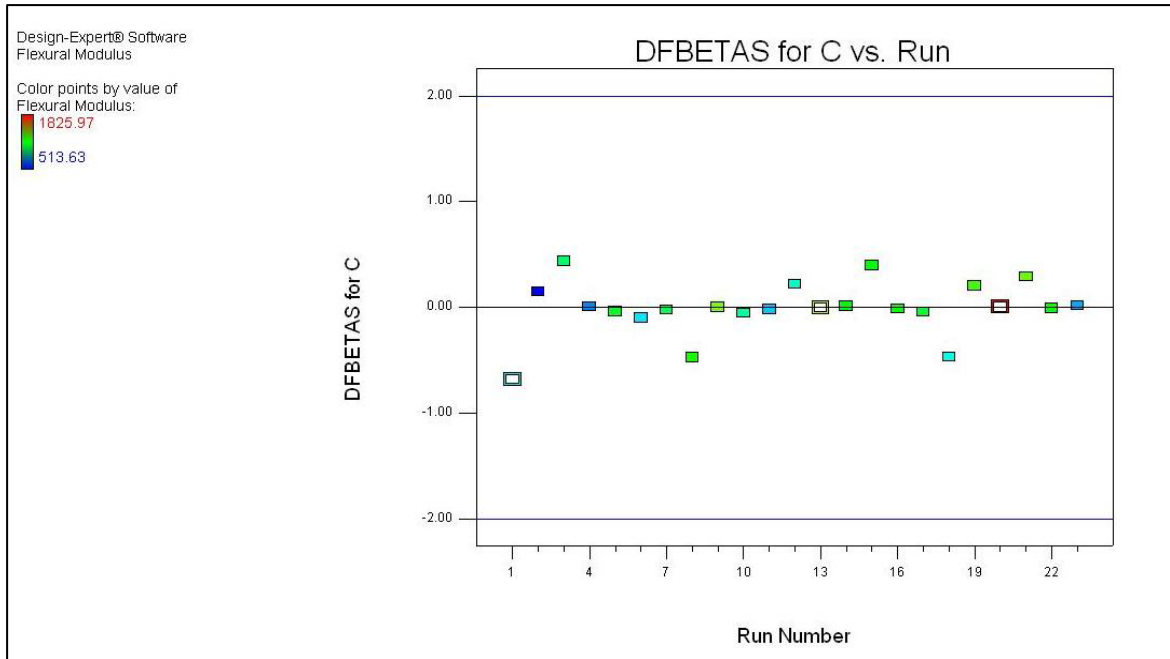
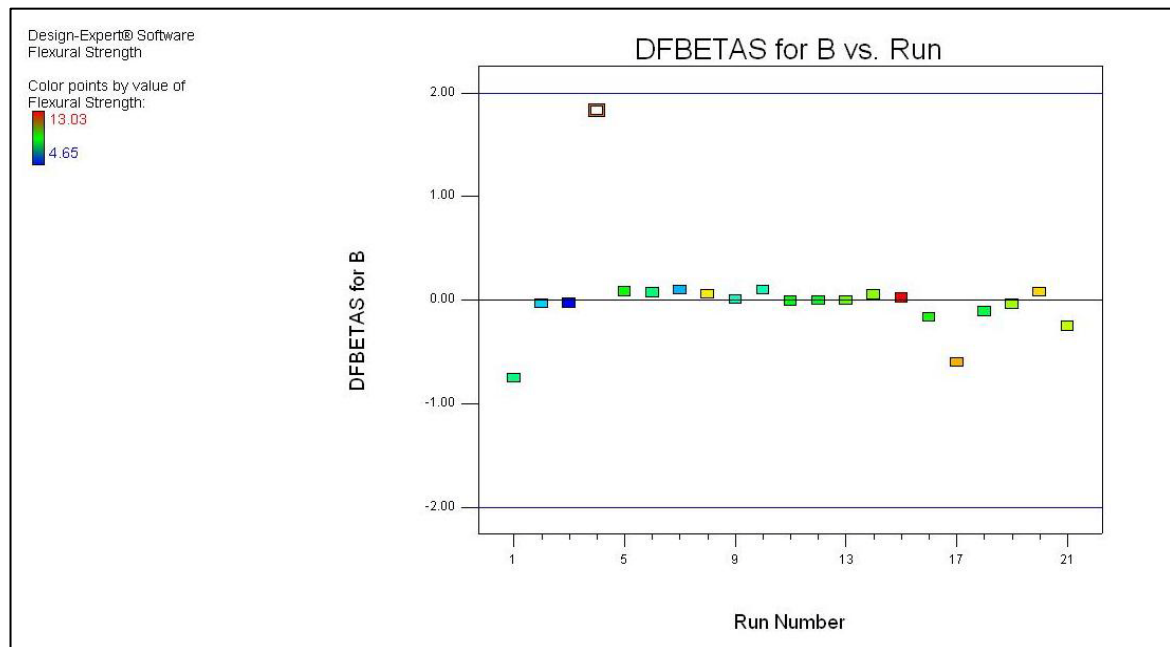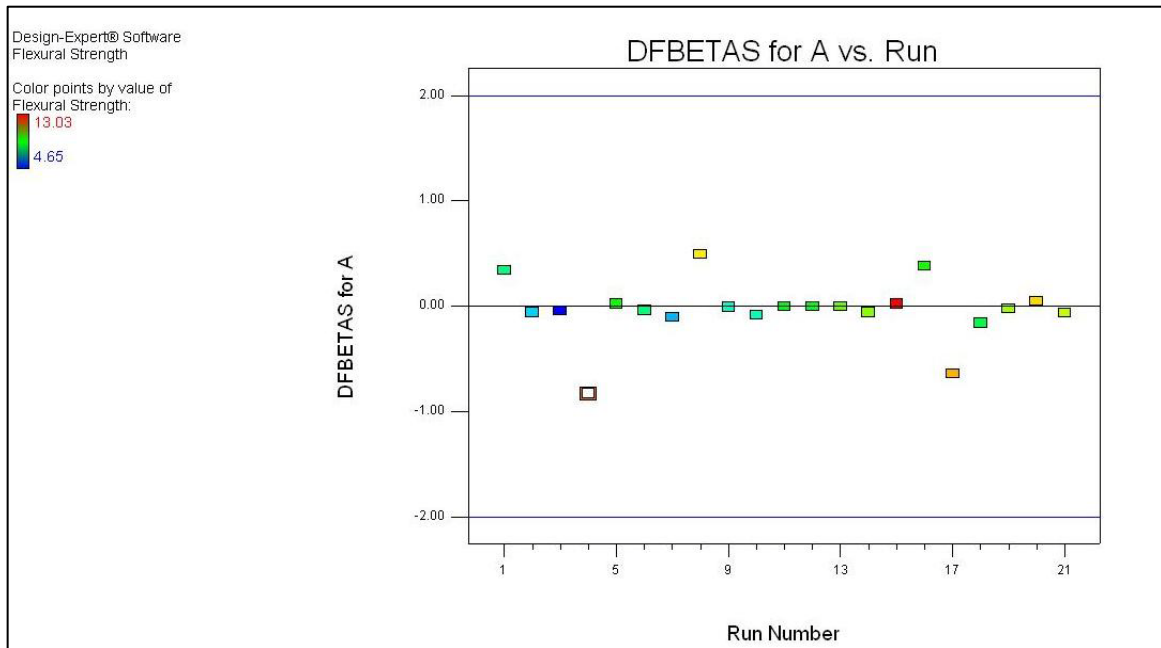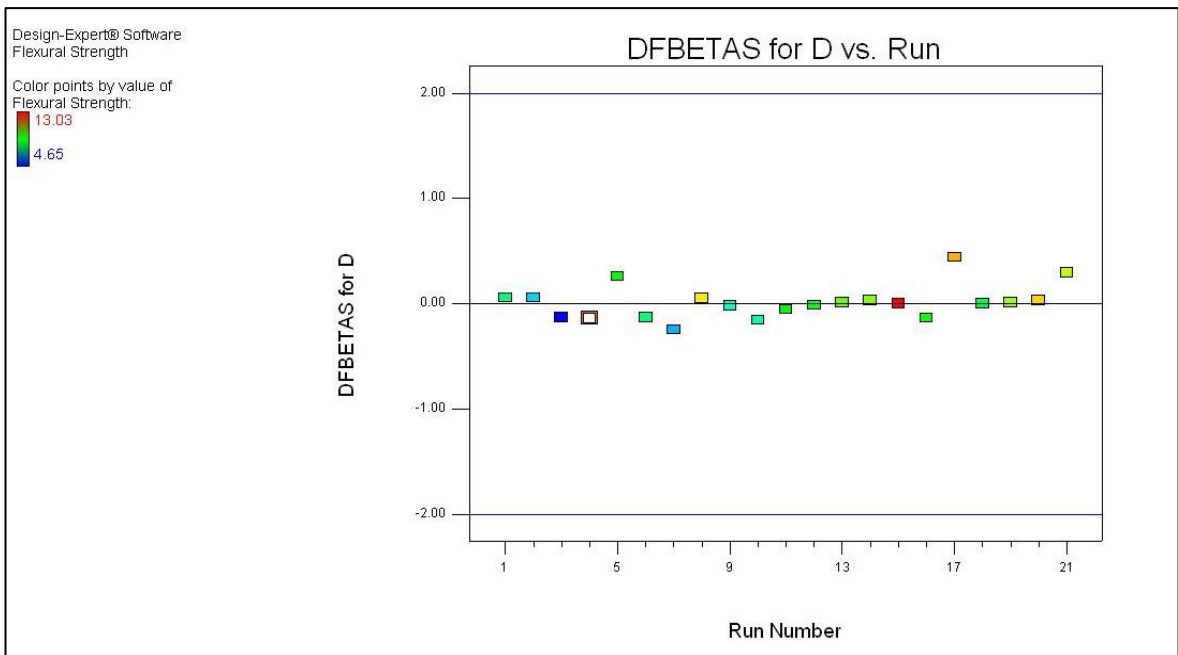# APPENDIX 10: FLEXURAL STRENGTH DFBETAS GRAPHS FOR EACH COEFFICIENT

DFBETAS for C vs. Run

Design-Expert® Software
Flexural Strength

Color points by value of
Flexural Strength:
13.03
4.65

DFBETAS for D vs. Run

Design-Expert® Software
Flexural Strength

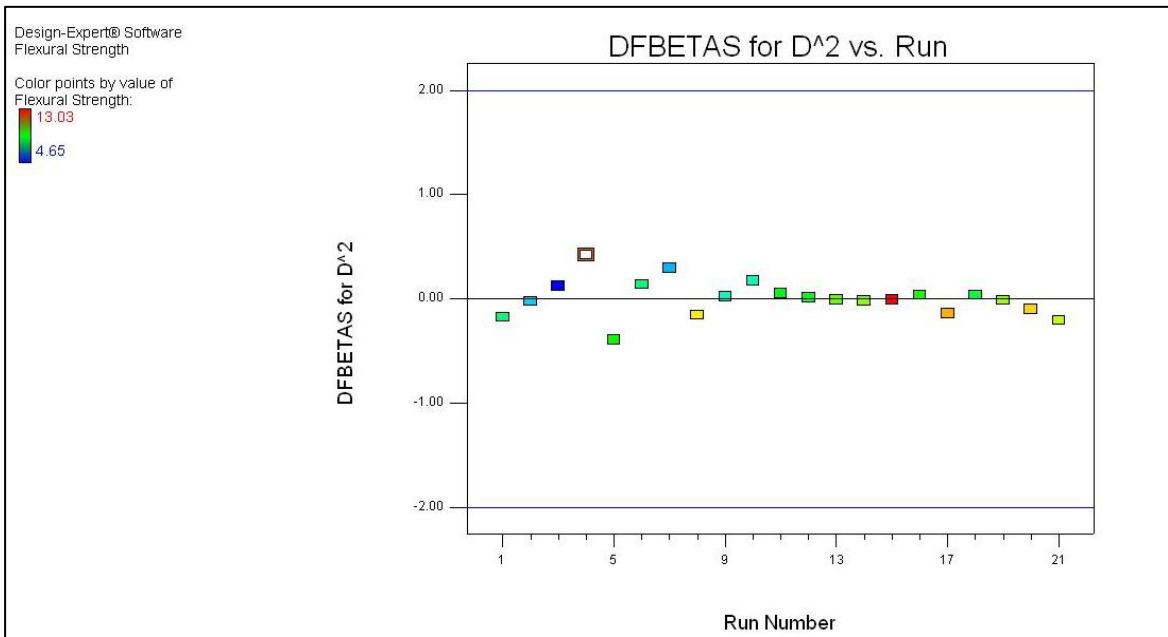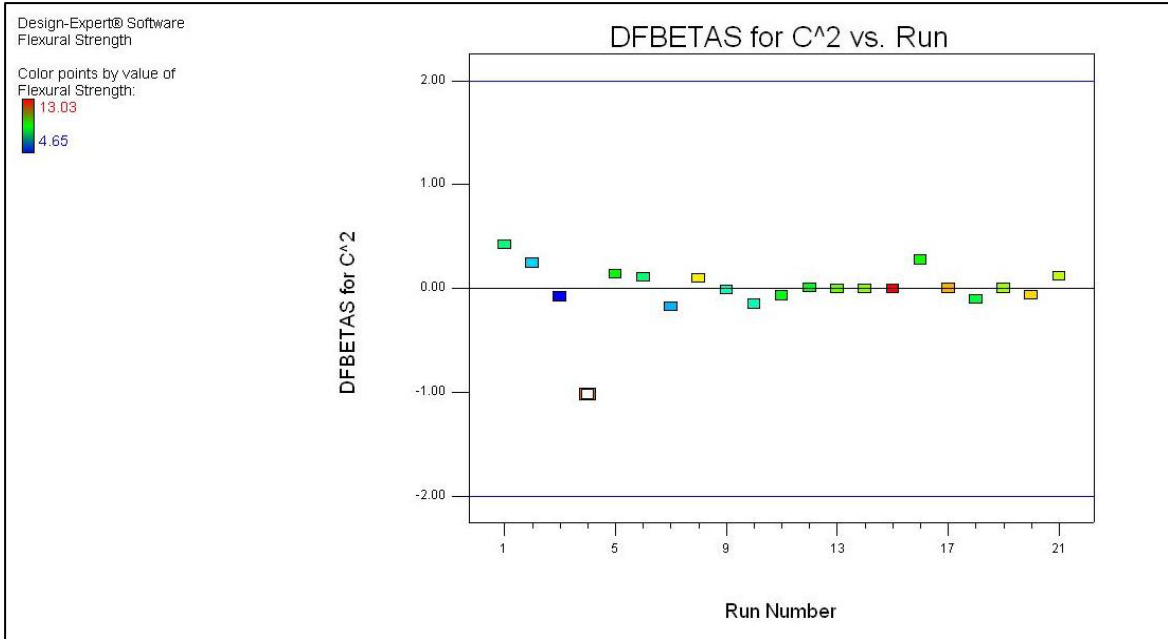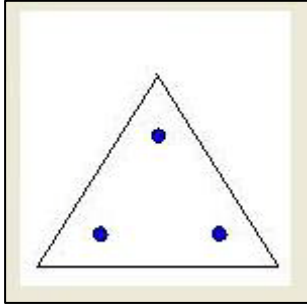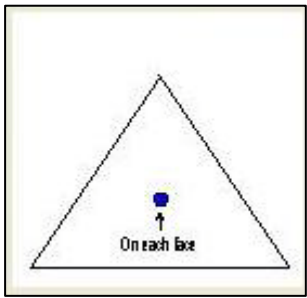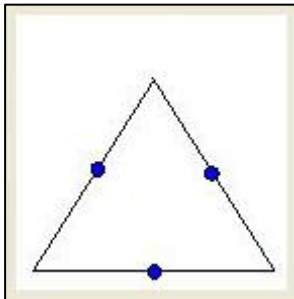Color points by value of
Flexural Strength:
13.03
4.65

126
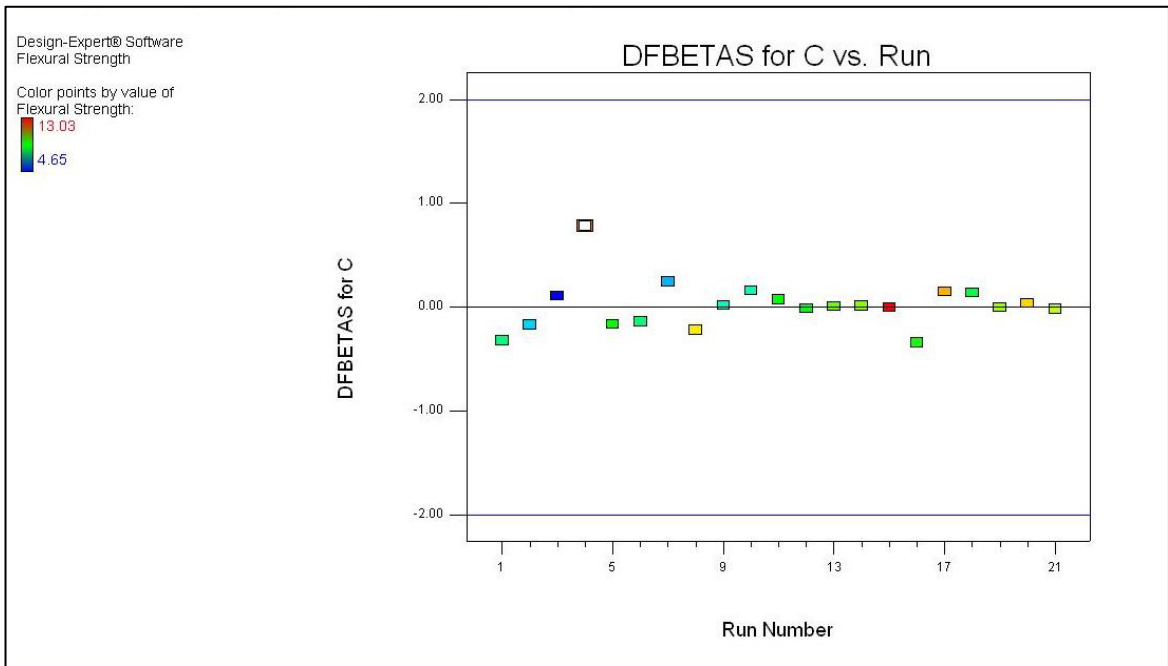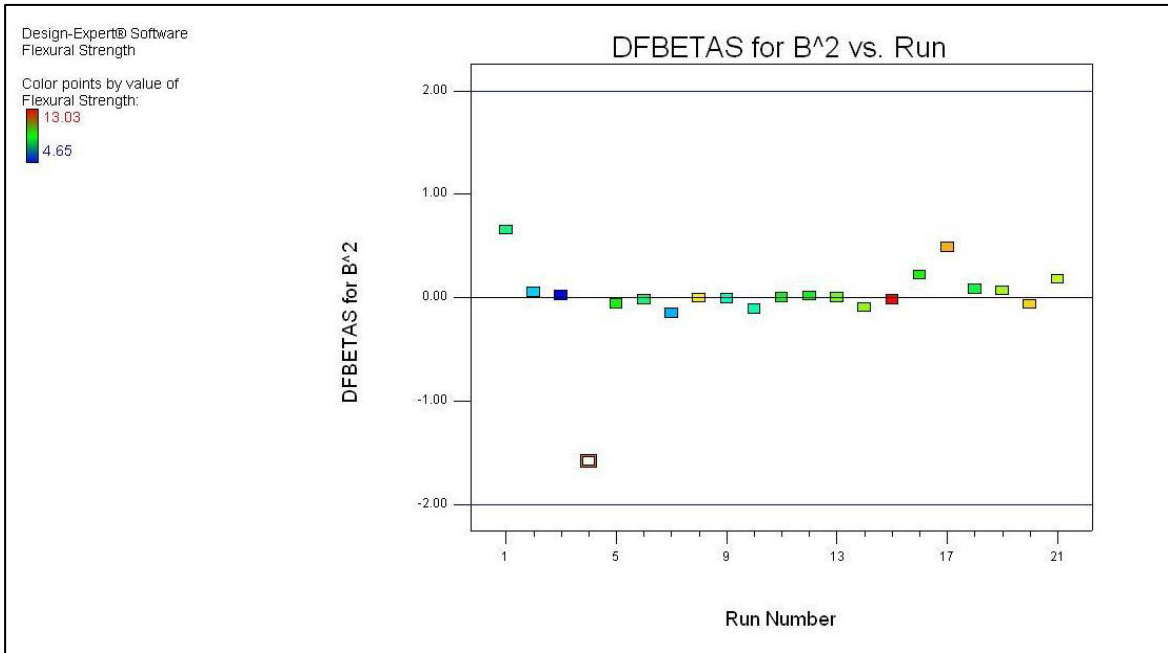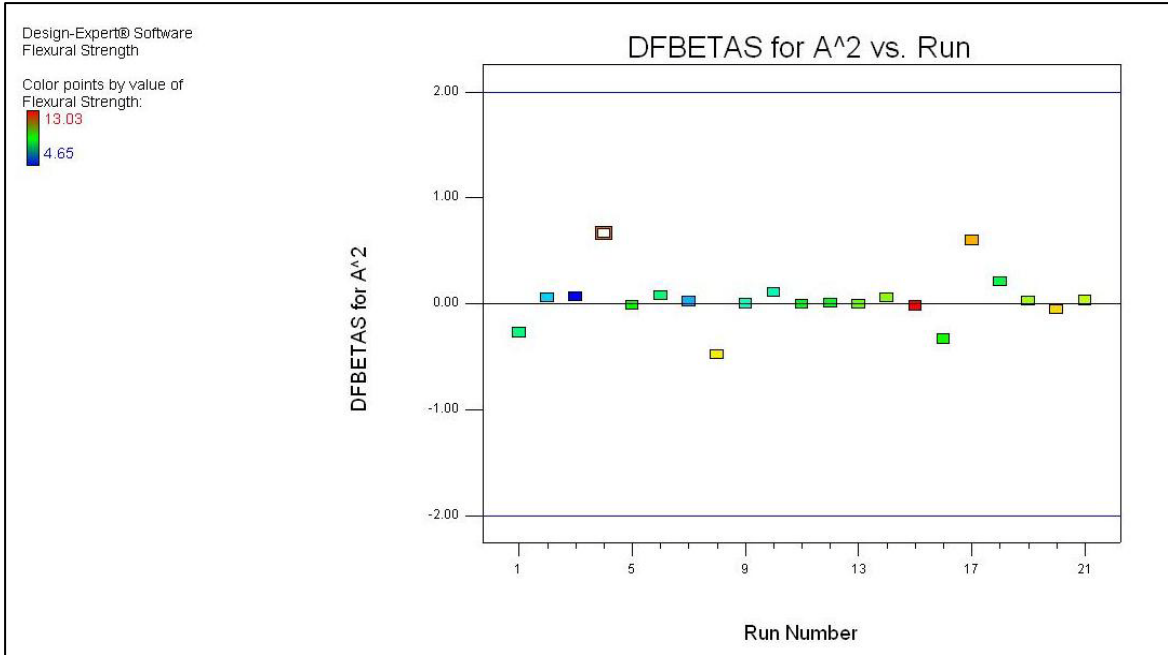
APPENDIX 11: GRAPHICAL DISPLAY FOR CANDIDATE POINTS OF A DESIGN ON ILLUSTRATIVE TRIANGULAR SHAPES
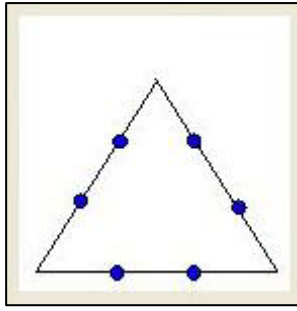
Axial Check Blends

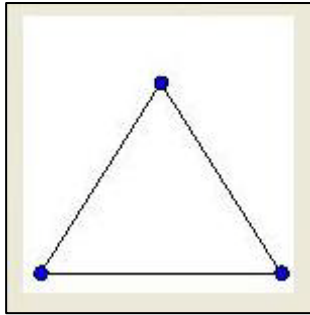Constraint plane centroids (on each face)

Centers of Edges
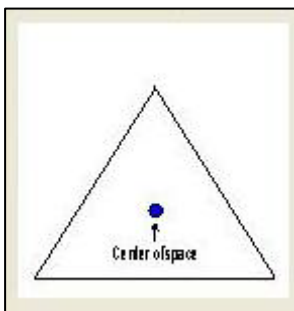
Overall Centroid (center of space)

Thirds of edges

Vertices