Characterization of the Effect of Recycled Polypropylene Using DOE

by

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Abstract

Polypropylene (PP) is a thermoplastic polymer and is one of the most widely used polyolefin polymer, which is a well-known semi crystalline polymer. Plastics such as polypropylene are commonly used for different applications and purposes due to their versatility; however, it should be noted that our current approaches in plastic product production, consumption and disposal are not sustainable and causes concerns for environment and human health. Hence, appropriate use and disposal, especially recycling has an essential role. There are several studies investigating the chemical and mechanical properties of polypropylene; and also there are a few studies on the effects of multiple recycling on chemical and mechanical behavior of polypropylene; however, there are relatively few investigations involving combination of virgin and recycled polypropylene. To more accurately investigate the effects of the addition of different percentage of recycled polypropylene on tensile properties of polypropylene this study was conducted. Also the effects of annealing and glass fiber additive on recycled content polypropylene are investigated in this study. Cold temperature effect on polypropylene properties also investigated (due to environmental conditions in Edmonton). Hence, using design of experiment (DOE) methods, the effects of all these factors on tensile behavior of polypropylene is carried out in detail. Also a wide angle X-ray diffraction (WAXD) system is used to investigate on crystalline structure of the recycled content polypropylene samples to better explain their tensile behavior. Furthermore, drop test simulations are performed on a sample box, using the measured tensile properties at room temperature as a case study. Based on the current experimental study, and

combination with the literature reviews, it is concluded that recycled material could be use without detrimental effect on the material properties of polypropylene.

Guidelines of this study could be useful for further studies on different materials or more combinations. The comprehensive tensile results obtained through this study are valuable for plastic industries and designers, which could be helpful to reduce industries, cost of material and also recycling is one of the most environmental friendly ways to dispose plastics. Combination of DOE methods and statistical analysis with tensile experiments and FEA simulations makes a powerful method which could be used as a guideline for further studies, other researchers, industries and designers to reduce cost and plastic production.

Preface

This thesis is an original work by Fatemeh Khademi.

A version of chapter 3 of this thesis has been submitted as F. Khademi, YS. Ma, K. Duke, K. Choi, and C. Ayranci, “Effects of Recycling on Mechanical Behavior of Polypropylene at Room Temperature through Statistical Analysis Method”, *Journal of Polymer Engineering and Science.* (Under revision)

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Technical case study and materials were provided by Drader Manufacturing industries (Edmonton, Alberta).

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Chapter 1 : Introduction and literature review

1.1 Plastics and its applications

Term "plastics" today is used for a wide range of polymers like polypropylene, polyethylene, polyvinyl chloride, polystyrene, PVC, nylon, and etc., which can be molded to make a rigid or slightly elastic part. Plastics came to human's life by using polymers since approximately 1600 BC when the ancient Mesoamericans processed natural rubber into balls, figurines and bands [1]. Since then, human relied increasingly on plastics and rubbers; starting with natural polymers, waxes, horns, natural rubbers and resins since 19th century when development of modern thermoplastics began. Development of modern plastic extended in the first 50 years of 20th century with creating at least 15 new classes of polymers. Plastics proved their versatility for being use in a range of types and forms such as natural polymers, modified natural polymers, thermoplastics, thermosetting plastics and recently more biodegradable plastics [2].

Plastics are very unique materials due to their wide range of properties and flexibilities. Plastics could have a high strength-to-weight ratio, stiffness and toughness, corrosion resistance, ductility, bio-inertness, non-toxicity, thermal and electrical insulation, they can be operated over a wide range of temperatures, high durability in compare with their low cost, and also due to their light weight they have less transportation cost [2]. Also, the relatively low melting temperature of plastic compared with metals or ceramics, makes it easy to be formed in different shapes is another unique characteristics. Hence, all these characteristics make plastics a very resource efficient material.

Plastics are playing an essential role in today's world. Plastics are being used in all industries for variety of purposes; clothing and footwear, car industries, construction, packaging, electronics, fashion, decoration, toys, and even in medical industries; the world is not even imaginable without plastics. Plastics are very unique as they can be used in a wide range of temperature and also they are chemical- and light- resistant. Plastic can be strong and tough, however it can be easily worked when it is a hot melt; this range of properties combine with its low cost drive to consume over 300 million tons of plastics annually worldwide [3]. Plastic processing is a complex procedure which provides variety of methods for production of different parts by development of thermoplastic materials, which production and processing for these materials occur at molten state [4]. There are lots of limitations in traditional plastic part design and manufacturing. Because of the complexity and high cost of this procedure it is important to be precise in preliminary stages rather than doing troubleshooting at the end.

Virgin plastic polymers are rarely being used in productions. Typically, plastic polymers are mixed with different additives- such as: carbon and silica for reinforcing the material, flame retardants, thermal and ultraviolet stabilizer, plasticizers to render the material pliable, and color in substantial quantities- to improve plastics functionality [5].

As mentioned, due to strong durability which plastics have these days, they are considerably beneficial in human life; however, it should be consider that our current approaches in plastic product production, consumption and disposal are not sustainable and causes concerns for environment and human health. Hence, appropriate use and disposal, especially recycling is essential [6].

1.1.1 Engineering plastics and Polymers

Engineering plastic is a synthetic polymer with mechanical properties to be use in a form of load-bearing product. Polymers are the major portion of engineering plastic. Polymers are made up of tremendously large molecules and from polymerization of different monomers. Therefore, the basic structure of polymers affects the properties the manufactured plastic [7]. The interesting part about polymers is that it is possible to design a desired polymer with specific properties by selecting specific atoms and understanding of their forces behavior. Polymer properties are mainly based on the polymer structure, which is subjective to basic chemical composition, morphology, and processing. Therefore, a polymer scientist can do this custom polymerization process to make a plastic which meets the specific application requirements.

Engineering plastics are being use when demanding requirements are preferred for mechanical performance of applications in order to provide the most cost effective design with the optimal material and process selection.

1.1.2 Polypropylene

Polypropylene (PP) is a thermoplastic polymer which also known as polypropene which is the focus of this study. PP is one of the most widely used polyolefin polymers and also is a well-known semi crystalline polymer. The main reason which makes PP to be center of attention is that it provides a balance of strength, modulus and chemical resistance at also a relatively low cost and weight [8]. PP has a high melting temperature and chemical resistant, lower density and better mechanical properties which makes this material useful for several potential applications in different industries such as automobile, appliances and different other commercial products. Most of these applications require acceptable performance over a range of temperatures; therefore investigation on its performance over a range of temperature is useful and important.

Mechanical behavior of PP has been investigated by many researchers in recent years. For example, Hartmann et al. [9] and Duffo et al. [10] have reported the tensile behavior of polypropylene at different temperatures. Arruda et al. [11] did some other studies on tensile properties of polypropylene at various strain rates. Furthermore, other researchers have tried to find the combination effects of temperature and strain rate on this material [8,12].

1.2 Plastic applications and performances

Plastics are being used for different applications from daily usage like plastic bags and containers to specific ones like automobiles or even aircraft industries. Hence, it is noticeable that plastics have a wide range of properties which makes it essential to choose the right material for the specific application. There is variety of performances which could be required for any end-use product based on its functionality requirements. Performances can be divided in to two categories: physical and mechanical; physical performance such as transparency, flammability, electrical, moisture and chemical compatibility, and ultraviolet stability are only related to the material property; however, mechanical performance requirements are such as strength, stiffness, impact and temperature resistance which depends on both the material and design geometry [13].

What makes plastics unique is that the functionality of the end-use product is function of not only its material but also the environmental factors. Hence, to get the desired functionality from the end-used product, there are three essential factors which make a critical triangle to determine the appropriate end-use and application: material selection, processing, and design (figure 1.1) [14]. Therefore, the final functionality of the end-use product is not only based on the design of the part, but also it extremely depends on appropriate material selection and processing conditions [13, 15].

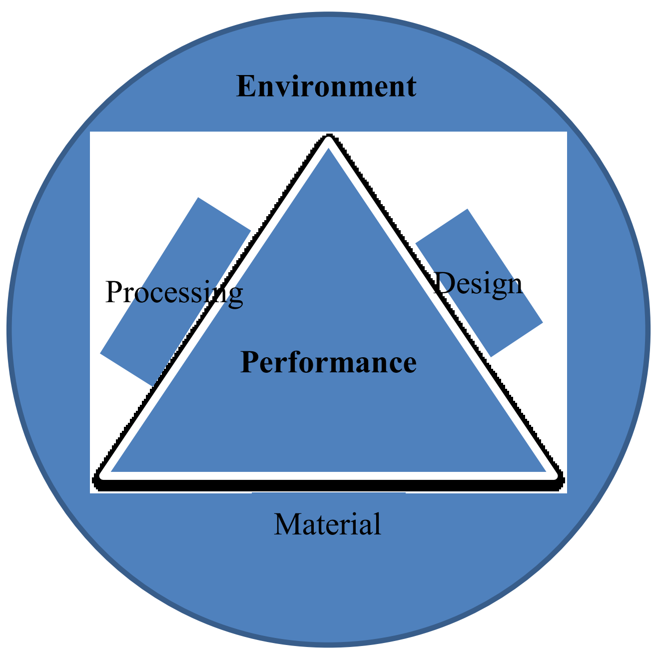

Figure 1-1: graphical representation of the factors influencing plastic part performance

1.2.1 Plastic part design

In designing any product several factors should be considered but basically designers always consider factors, namely, economically, reliability, maintenance and life cycle, and timely delivery of the product. It should be noted that integrated with named factors, functionality and performance of the end-use product is critical [16]. Hence, designers after the preliminary study of the end-use product, try to define the geometry by using sketches and drawings to gradually develop the design of the part which represents the detailed design and function of it through the product development process which represented in figure 1.2 [17]. Computer aided design (CAD) is one of the most important applications in computer aided domain which can strongly help designers in the process of product development. There are several tools and software which could help designers through this stage, some of them are mentioned in table 1.1 [17].

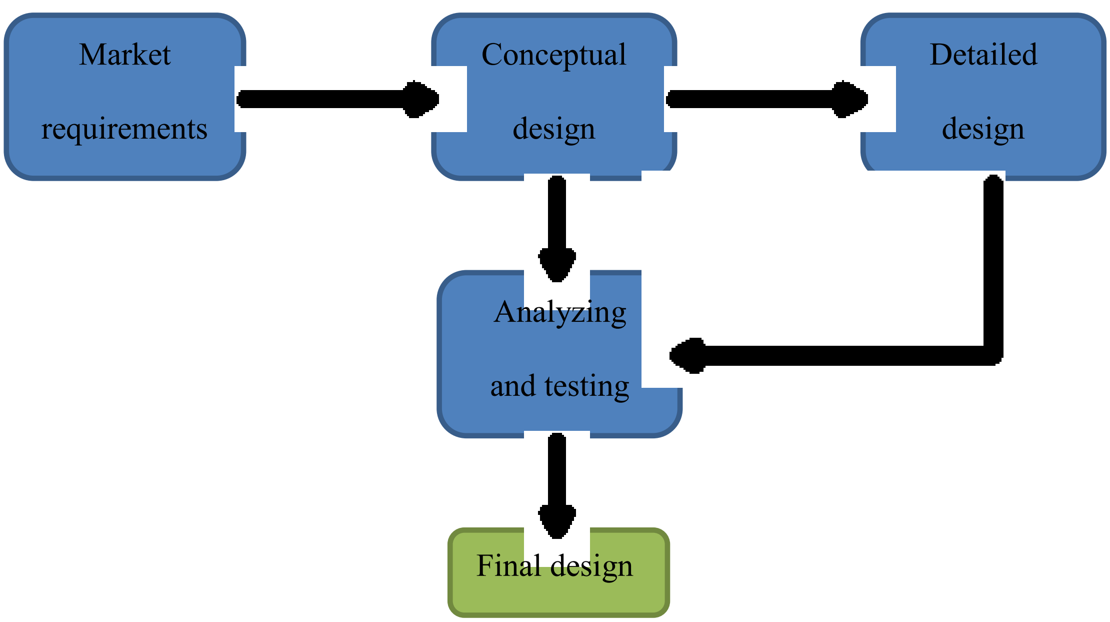

Figure 1-2: Product development process

Table 1-1: Designing tools and software

Company	Product
Autodesk	Inventor 2014
Dassault Systèmes	CATIA V6
	SolidWorks 2014
PTC	Pro/E ,Wildfire 5.0
SIEMENS	NX 10
	Solid Edge

As mentioned earlier, integrated system of design, material and process can guaranty to have a successful end-use product. Therefore, design-based material selection [18, 19] is one of the techniques to reach this goal. In this method, preliminary part design, material performance and manufacturing constraints are considered to meet the part performance requirements with the least cost.

1.2.2 Plastic part failure analysis

As mentioned earlier, there are three key factors of material selection, processing and design which work concurrently to determine the performance of the end-use plastic part. Therefore, it is always difficult to identify a single cause which affects the performance of the plastic part and different integrated factors can lead to failure of a plastic part. There are several modes of failure for plastic parts, such as fatigue, environmental stress cracking; brittle fracture, ductile fracture, and creep rupture [15]. Hence, a systematic failure analysis is needed in order to find the certain cause and mode of the failure.

Compared with other materials such as metals, failure analysis of plastics is much more complicated as plastic properties not only depend on the polymers molecular structure but also depends on the additives such as colors and fillers of the plastic [20]. Hence, it is essential to have a good knowledge of material properties of plastics to make a successful. Additionally, it can help to have a good understanding of the effects of different factors such as time, temperature and load rate on material performance which are key factors to prevent part failure [13].

1.3 Effects of different conditions on plastic properties

The following section will outline the effects of temperature, recycled material, adding fiber and annealing on the performance of plastics.

1.3.1 Effects of temperature

As mentioned earlier, Polypropylene has several potential applications in different industries such as reusable containers, laboratory equipment, automobile appliances and many different other commercial products. Most of these applications need to have acceptable functionality and appearance over a range of temperatures; therefore investigation on polypropylene performance at different temperature is useful and important. Each material has a useful working temperature range which is characterized by its glass transition temperature, T_g, and the melting temperature, T_m.

In general, for most polymers, temperature and strain rate can be considered to have a kind of equivalent effect on the material properties of the polymer. Implicate that, loading that polymer at a low temperature high strain rate could be equivalent to loading at a high strain rate and vice versa [12]. It indicates that, with increasing the temperature, the modulus of polymers normally decreases, though thermal transitions will occur which explains an affected step-change in stiffness over a quite small range of temperature [8, 12]. Polypropylene is semi-crystalline polymer, which reveals a complex combination of thermal transitions arising in the crystalline phase and also in the amorphous phase of the material. The presence of the crystalline phase can also enforce limitations on the flexibility of the amorphous phase, which complicates the ability to predict the mechanical performance of this semi-crystalline polymer at various temperatures [21].

1.3.2 Effects of recycling

Recycling is a process to reuse waste materials which is essential for waste reduction. This process of changing the waste material in to new products prevents the discarding of potentially useful materials and also reduces environmental pollution.

Usage of plastic materials has grown rapidly in recent years as they become an important part of society. As plastics are not biodegradable, recycling is a green movement which makes plastics to be one of the best candidates for recycling process in order to reduce waste. Through the process of recycling for plastic materials, a used product will be melted and then cast into a new products.

Several researchers have noted that recycling of polymers and their blends has significant effects on mechanical properties and morphology of polymer blends [22-26]. Also, in order to get intermediate material properties of the recycled material, it is usual to blend them with the same virgin material [27]. More specific studies worked on recycled polypropylene [23, 26, 27]; and they notices improvement in some of the mechanical and chemical properties of the material; increase in elastic modulus, yield stress, tensile strength, crystallization rate and crystallinity, melting temperature and also thermal stability of the material. However, elongation at break and fracture toughness were decreased by recycling [27]. However, previous researchers focused more on the effects of different times of recycling on properties of the polypropylene. From the literature it can be concluded that within 3-4 times of recycling, there is no significant changes in the material properties of polypropylene; although, by increasing the number of recycling, improvement in the material properties could be achieved [23, 24, 26].

1.3.3 Effects of Additives (Fiber)

Additives can yield substantial changes in the properties and processibility of polymers. Different additives can be used in the process of making plastics to improve the desired properties. Some additives such as colorants, antioxidants, and thermal stabilizers, are being use to change the appearance of the plastic part which will be produce; hence these kinds of additives will not affect the mechanical properties, but could influence viscosity of the plastic during processing. However, mineral fillers and glass or carbon fibers affect not only the mechanical properties of polymers but also their processibility. Mechanical properties of polymers could be significantly improved by adding fibers to them. However, polymers performance is dependent to the orientation and length of fiber [7]. The main characteristics to add fibers in polymers are high stiffness and high strength; fibers could be able to maintain these properties at high processing temperature, exposure and moisture; and these factors have significant effect on physical and chemical properties of fiber [28, 29]. Fibers by having high stiffness and strength will designate strong interatomic and intermolecular bonds and also strength limiting flaws to the material, which will affect the composite properties [29]. Fiber properties are dependent on the fiber microstructure, and the fiber microstructure is extremely related to the processing factors; therefore, processing factors should be set based on expected results for the fiber [28].

1.3.4 Effects of Annealing

As a result of inappropriate material selection, design or process, stress could be introduced in the plastic part resulting in poor performance causing warpage, twist or other dimensional changes. Annealing is a process of slowly heating and cooling plastic parts in order to relive the stress inside the material to prevent or reduce failure. Depending on the type of plastic and the part cross section the annealing procedure could be different. Basically, in annealing process plastic part is heated slowly to a temperature just below its melting temperature, kept at this temperature for a period of time and cooled down slowly to the room temperature. There is an industry standard for annealing process. It starts at room temperature; increase temperature 10°C/hr to reaches the annealing temperature, isothermal for 2 hours, then decrease temperature with the same speed until it reaches the room temperature.

Studies showed that annealing and annealing temperature have significant effect on crystalline structure and mechanical property of plastics [30- 33]. Increasing the isothermal temperature will increase the crystallinity and mechanical properties of plastics [32]. A study shows that annealing polypropylene at relatively low temperatures (80-110) enhances the material properties slightly, however annealing at high moderate temperatures (140-160) has more significant improvement [28-33]. Another study shows that fracture initiation, which is related to the degree of crystal perfection, could be enhanced by annealing close to the melting temperature and also annealing above the crystallization temperature improved the fracture propagation [32].

1.4 Methods

1.4.1 Material characterization

Material characterization which also known as material evaluation, is a basic engineering activity which is done through all stages of product life cycle from design and manufacturing to failure analysis of the final product [7]. Material characterization could start from the design phase to select an appropriate material and process which could provide the best interrelationship of material, process, shape, and function (figure 1.3) [34].

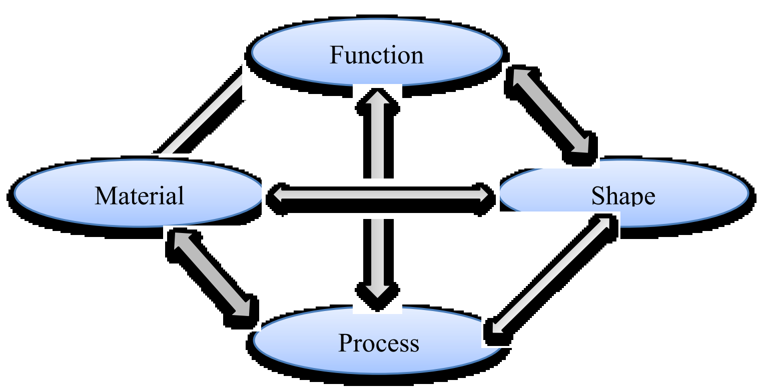

Figure 1-3: Interrelated factors involved in the design process (32)

Tensile testing is one of the most commonly performed mechanical tests use to evaluate mechanical properties of materials. The tensile test is a simple test which is also inexpensive and fully standardized. For tensile testing of polypropylene ASTM D638 suggests that a dog-bone shape specimen should be used. Tensile test works by pulling the dog-bone shape specimen until it breaks to obtain a complete tensile profile. As a result of this test, a force-displacement curve could be generated which Later on, based on specimen's dimensions, it should be converted to a Stress-strain curve (see figure 1-4). The failure point is typically called "Ultimate Strength" or UTS; according to the "Hook's Law", the linear part of this curve shows the ratio of stress to strain which is the "Module of Elasticity" or "Young's Modulus" as $E = \sigma/\varepsilon$. Tensile Strength at yield or break, Elongation results at yield and at break, Strain results, Modulus of Elasticity, and Poisson Ratio are some of the main results which could be conducted from the tensile test [35]. Considering polymers result, which are the focus of this study, they could be categorize by their mechanical properties as soft-weak, hard-brittle, soft-tough, hard-strong, and hard-tough [36].

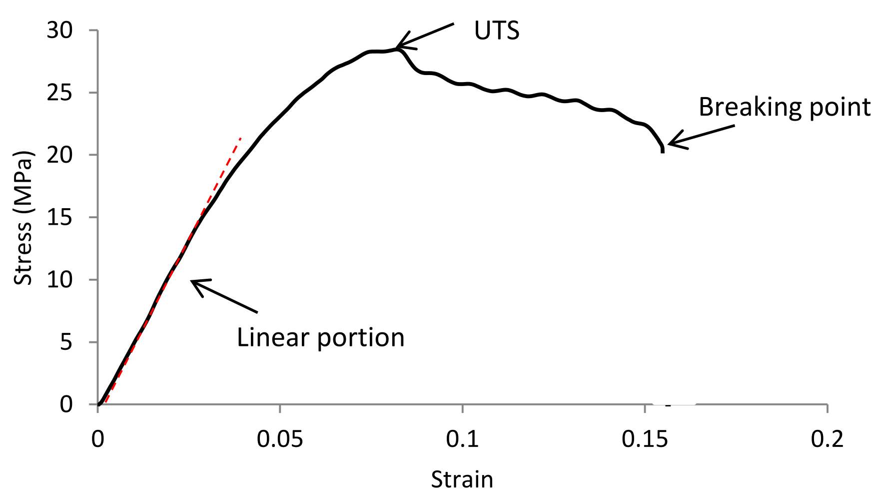

Figure 1-4: Sample stress- strain curve from our results

1.4.2 Wide Angle X-Ray Diffraction

Wide angle X-ray diffraction (WAXD) also called as wide angle X-ray scattering (WAXS) which is a non-damaging method to characterize solid materials. WAXD is a technique to determine the crystalline structure of polymers and the degree of crystallinity of polymers. When X-rays directed in solid sample they will scatter in a predictable patterns which is related to the internal structure of the material; therefore, as a result the scattering intensity versus the 2θ angle of the X-ray will be plotted and every crystalline material has a unique pattern like a “finger print” for that material [37].

1.4.3 Design of experiment (DOE)

Design of Experiment (DOE) is a tool to help engineers and scientists to optimize their experiments in design and development process. It helps by providing the optimized structure of design to optimize the usage of time, cost and effort. Use of DOE in early stages of product life cycle could significantly reduce development time and cost and also it could lead to better performance and higher reliability [38]. DOE is an essential statistical tool to solve complex problems by saving time and effort. DOE helps not only to design the experiment, but also to analyze the results by providing comparison and effect tools in multi- factor and multi-level experiments. Computer software programs are widely being used to assist in performing experimental designs. Different software can be used for this purpose; Design-expert, Minitab, STATISTICA, and JMP are some examples. In this study we are using STATISTICA software to analyze the results. Therefore, it helps to identify the key significant factors, determine their interactions, and to generate the most optimized solution.

1.4.4 Finite element analysis (Drop test simulation)

Finite element analysis (FEA) is an essential computer aided engineering (CAE) tool, which assists engineers in design and development process during the life cycle of the part. FEM helps by providing tools to predict part quality and performance without spending time, money and effort to produce it. Therefore it is very helpful in troubleshooting procedures. CAE helps by providing simulation tools to analyze the process to provide reliable and optimized reference data which not only increase the success probability, but also increase the quality of design and manufacturing. Simulation tools help to predict performance, quality, and also production process of the part in very early stages of design. To implement this method, a sample drop test simulation performed using UG NX 9.0, NASTRAN software. Drop test is one of the important tools in this package which helps to estimate the part performance in drop situation in order to make reliable and strong part according to design specifications. Therefore, the model of the part firstly should be meshed in a 2D mode and after correcting the mesh errors it should be converted to a 3D meshed model to be ready for any kind of simulations. In UG NX NASTRAN software to apply a drop test simulation, after making a correct 3D mesh, drop point and vector should be defined and constraints should be added, then the simulation could be run. In industries UG NX software is popular software for part design and simulations, also there are many other software available in the market for design and simulations. Y. Gu and D. Jin used drop test simulation and also by using DOE techniques to optimize the design of microelectronics packages [39]. Also many other researchers used drop test simulations for optimization and reliability test of designed parts [40, 41] . Hence, a three-dimensional drop test simulation performed using the material properties determined from the specimen characterization on an actual container as a case study.

1.5 Objectives

The overall objective of this study is to obtain a more reliable polypropylene material from the mechanical point of view while taking advantage of using recycled material. Consequently this will reduce plastic productions by finding more applications for recycled materials. Also, performance of the material under different temperature conditions will be investigated to find the most reliable combination of material. This main objective will be reached by the completion of following tasks:

1. Fabricating different combination of recycled and virgin polypropylene specimens.
2. Experimentally investigate tensile properties of the specimens at select temperatures.
3. Running simulations (drop test) as a case study to prove our investigation at room temperature.
4. Study the combined effect of various factors (Recycled, Annealing, Fiber, and Temperature) on mechanical properties of the selected combination of materials.
5. Provide guidelines

To reach the above goals, a systematic experimental investigation using design of experiment (DOE) methods and statistical analysis was performed to analyze the experimental results. Hence, tensile tests performed to investigate the tensile properties of the materials and the effects of factors; also a wide angle X-ray diffraction system used to see the effects of factors on crystallite structure of tested materials.

Fulfilling this study will provide useful guidelines in optimal selection of materials while taking advantage of recycled material to reduce cost and also to make a green choice which could be very beneficial for plastic industries.

The uniqueness of this study is that it is the first to simultaneously investigate the effects of multiple factors, using DOE, on the mechanical properties of polypropylene; and also investigations on the effects of blending recycled material with the same virgin material was a new study.

1.6 Thesis outline

This thesis is arranged in the following sequences:

- The first chapter gives a brief introduction and literature review of the scope of this study.
- The second chapter focuses on materials and methods which used in this study.
- The third chapter investigates the effects of considered factors on tensile behavior of polypropylene at room temperature.
- The forth chapter focus on the effects of cold temperatures on tensile and crystallite behavior of polypropylene
- Finally, in chapter five, the content of the thesis is summarized and conclusions are made.

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Chapter 2 : Materials and methods

2.1 Materials and definitions

- The polypropylene material which is the focus of this study is called “20 melt, PP CO, no-break”. PP could be bought with two different polymer chains which are PP copolymer and PP homo-polymer; in this study, PP copolymer is used which is more flexible and less stiff. This PP is also the base material for the plastic part which will be analyzed as a case study. “20 melt” refers to melt rate of the material from standardized melt flow test [1] which higher number basically shows that material flows more easily in melted condition under pressure. Finally, no-break is another rating method based on IZOD impact test [2] which basically means that the material did not split during the impact tests.
- Virgin PP is from virgin polypropylene pellets which comes from a material supplier and have never before been molded into parts. Therefore, virgin PP specimens are specimens that have been molded from virgin PP pellets.
- Regrind material refers to chunks, bits, particles and dust made from grinded used parts through granulator. This material is in odd shapes and sizes and does not process uniformly.
- Reprocessed material is regrind material that has been processed into uniform pellets before being molded again and this extra step is sometimes taken to ensure uniform process control.

- Recycled parts are parts molded from either regrind material or reprocessed material. Figure 2.1 shows the process of making recycled part more clearly in a graphical view [3].
- Recycled material which used in this study made from reprocessing of "20 melt, PP CO, no-break" parts at "Drader manufacturing ltd".

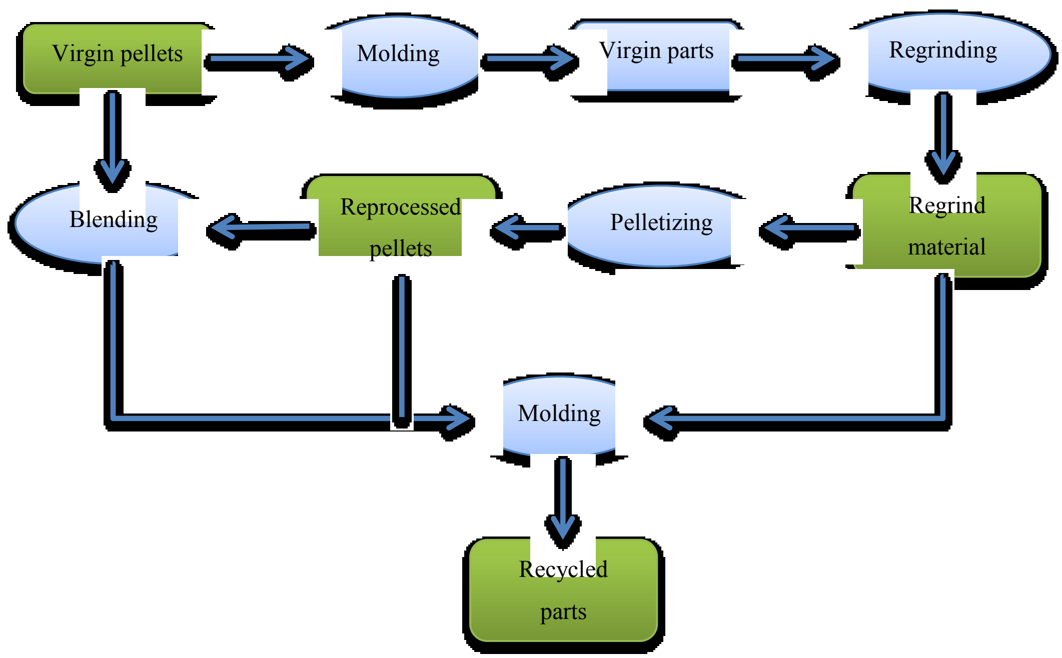

Figure 2-1:The procedure of making recycled parts (3)

2.2 Sample preparation

2.2.1 Specimens' recipes

In this study the focus is to investigate the effects of adding recycled material to the virgin material on mechanical properties of the recycled unreinforced and reinforced polypropylene material. The virgin polypropylene which used in our specimens bought from "Chase Plastics Thermoplastic Distributer Industry" (North and Central America) and the recycled material produced at "Drader manufacturing ltd" (Edmonton, Alberta, Canada).

It is essential to find the optimized amount of recycled material usage, to produce a useful material. Hence, 5 different levels of recycled material percentage were considered, namely, 0%, 25%, 50%, 75%, and 100% (table 2.1).

Table 2-1: percentage of recycled pp in each sample

Sample	Recycled polypropylene	Virgin polypropylene
1	0%	100%
2	25%	75%
3	50%	50%
4	75%	25%
5	100%	0%

In next phase glass fibers are added to the specimens with the same amount of recycled material in order to investigate the effect on the mechanical properties of reinforced recycled specimens. Hence, recipes are showed in table 2.2 as follows.

Table 2-2: considered percentage of glass fiber

Sample	Recycled polypropylene	Virgin polypropylene	Glass fiber
6	25%	67.5%	7.5%
7	50%	45%	5%
8	50%	35%	15%
9	75%	17.5%	7.5%

2.2.2 Injection molding

Injection molding is a manufacturing process of producing products by injecting molten material into a mold. Injection molding is a very common and economical production process for mass production of thermoplastics and thermosetting polymers. In this procedure, molten plastic is pushed through the mold cavity to shape the final product, then it cools down to solidify the plastics and finally the mold will be opened and the part will be ejected. One advantage with injection molding is that multiple products could be made at same time depending on the mold design. In this study, polypropylene samples are made through injection molding process at Drader Manufacturing Industries Ltd. In this procedure a mold was designed to produce 4 dog-bone samples simultaneously. Following ASTM D638-10 [4], type V specimens were made through injection molding process; dimensions and schematic view of the specimen showed in figure 2.2.

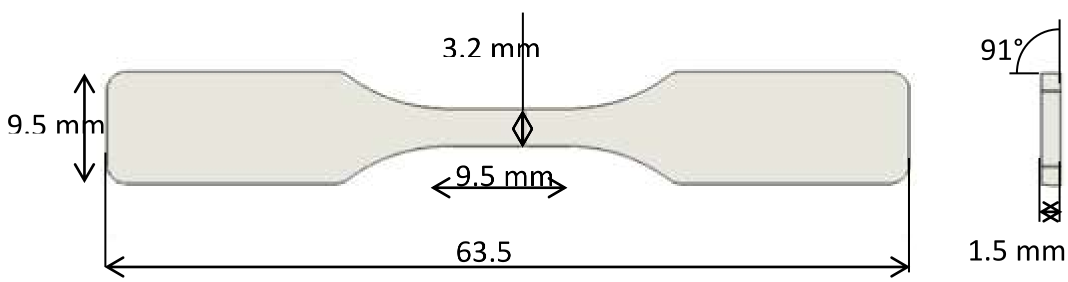

Figure 2-2: Type V dog-bone sample dimensions

Following the selected sample recipes mentioned in the previous section, recycled, virgin and fiber resins blended with each other and then vacuumed to the injection molding system to produce samples. To have consistency, extra specimens made for future studies; about 100 for each recipe. For the injection molding process of the specimens following specifications used, namely: barrel temperature of 227°C; melting temperature of about 210°C; injection time sets to 5 second which ensures the part is fully filled; cooling time was, about 35 seconds to ensure that the specimens will have minimal shape change after cooling outside of the mold; injection pressure was 103 MPa; packing (holding) pressure was 98 MPa and for three seconds [3]. All samples were packed and stored in the same place before and during the experiments for consistency.

2.2.3 Annealing procedure

In the annealing process samples were slowly heated and cooled down to relive the stress inside the specimens. This process helps to prevent and reduce failure by affecting the crystalline structure and mechanical property of the specimens [5]. The industry standard for annealing procedure is to start at room temperature, increase temperature 10°C/hr to reaches the specific temperature, isothermal for 2 hours, and then decrease temperature with the same rate to reach the room temperature. In this study, 150°C was selected as the annealing temperature. According to literature reviews [5, 6] annealing in arrange of 140°C to 160°C were suggested which compared to the melting temperature of polypropylene (190°C), this range for the annealing temperature is a high-moderate temperature which could more significantly improve the specimens properties [17]. However, in parallel another study is ongoing to investigate the effects of annealing at 140°C and 160°C. Therefore half of each level of samples (each recipe) was annealed to compare the results with non-annealed specimens. The whole view of the prepared specimens shown in table 2.3 resulted in 18 different categories of specimens. For this study the annealing procedure took about 32 hours. As samples are small, it was possible to put multiple samples at the same time on a tray in the oven to be annealed which made this procedure more efficient.

2.3 Design of Experiment (DOE)

In this study by taking advantage of DOE, an optimized balanced amount of experiments were conducted which could also results in a more reliable findings by analyzing the interrelationship between considered factors. The considered factors are: percentage of recycled polypropylene (0w%, 25w%, 50w%, 75w%, 100w%), percentage of glass fiber (0w% and 7.5w%), annealing condition (non-annealed and annealed at 150 °C), and the testing temperature (room temperature, 0°C, -20°C). These considered factors are named as manipulated factors in this study which summarized briefly in table 2.4.

Table 2-3: Whole view of considered samples

	Recycled PP	Virgin PP	Glass fiber	Annealing
1	0%	100%	0%	Non-annealed
2	0%	100%	0%	Annealed
3	25%	75%	0%	Non-annealed
4	25%	75%	0%	Annealed
5	25%	67.5%	7.5%	Non-annealed
6	25%	67.5%	7.5%	Annealed
7	50%	50%	0%	Non-annealed
8	50%	50%	0%	Annealed
9	50%	45%	7.5%	Non-annealed
10	50%	45%	7.5%	Annealed
13	75%	25%	0%	Non-annealed
14	75%	25%	0%	Annealed
15	75%	17.5%	7.5%	Non-annealed

16	75%	17.5%	7.5%	Annealed
17	100%	0%	0%	Non-annealed
18	100%	0%	0%	Annealed

Table 2-4: Considered factors which could affect the tensile properties

Recycled pp percentage	Fiber percentage	Annealing condition	Test temperature
0%	0%	Non-annealed	Room temperature
25%	7.5%	Annealed at 150°C	0°C
50%			-10°C
75%			
100%			

Therefore it will result in 5×2×2×3=60 experiments for a full factorial design; which includes all possible theoretical combinations and also according to ASTM D638-10 [4] five specimen should be tested for each sample which results in 60×5=300 experiments including five replicates for each sample; which is a huge number of tests. Taking advantage of DOE it is not necessary to test all the possible combinations when we are considering four different factors [7]; also because of the infeasibility of some combinations non-factorial fraction factorial design were used which resulted in 280 experiments including replicates. Table 2.3 shows the all possible combination of materials which are considered in this study. However, we will analyze the effect of each considered factor individually first and next the analysis on their combination will be discussed.

It should also be considered that 280 experiments is also a huge number of tests which could also be reduce to less using fractional factorial DOE techniques, however as mentioned, this is all the possible feasible combination of factors. In order to reduce the number of experiments, the experiments are divided in two phases. In the first phase, we just focused on the room temperature test. After finishing all the room temperature tests, results were analyzed to see the standard deviation of results for the five replicates of each sample. If the results show a tight standard deviation, in phase the second phase, for the colder temperature tests, as also they are more time consuming, it could be possible to reduce the number of replicates to three instead of five; which will result in 100(room temperature tests including five replicates) + 108 (colder temperature tests including three replicates) = 208 total experiments. The whole picture of the design of experiment of this study is shown in following table without considering the replicates.

Table 2-5: design of experiment of the study

	Recycled %	Annealing	Temperature	Fiber %
1	0	23	23	0
2	0	150	23	0
3	25	23	23	0
4	25	23	23	7.5
5	25	150	23	0
6	25	150	23	7.5
7	50	23	23	0
8	50	23	23	7.5
9	50	150	23	0
10	50	150	23	7.5
11	75	23	23	0
12	75	23	23	7.5
13	75	150	23	0
14	75	150	23	7.5
15	100	23	23	0

16	100	150	23	0
17	0	23	0	0
18	0	150	0	0
19	25	23	0	0
20	25	23	0	7.5
21	25	150	0	0
22	25	150	0	7.5
23	50	23	0	0
24	50	23	0	7.5
25	50	150	0	0
26	50	150	0	7.5
27	75	23	0	0
28	75	23	0	7.5
29	75	150	0	0
30	75	150	0	7.5
31	100	23	0	0
32	100	150	0	0
33	0	23	-10	0
34	0	150	-10	0
35	25	23	-10	0
36	25	23	-10	7.5
37	25	150	-10	0
38	25	150	-10	7.5
39	50	23	-10	0
40	50	23	-10	7.5
41	50	150	-10	0
42	50	150	-10	7.5
43	75	23	-10	0
44	75	23	-10	7.5
45	75	150	-10	0
46	75	150	-10	7.5
47	100	23	-10	0
48	100	150	-10	0

2.4 Experimental method

2.4.1 Tensile test

Tensile tests were performed according to ASTM D638-10 on Bose 3200 test system (accuracy exceeds ASTM E-2309, Class A; ± 0.5% of readings) ASTM at crosshead speed of 10 mm/min for room temperature, zero degree, and -10℃. An in house built optical extensometer was used to process samples profile for obtaining the Young Modulus (E), this method was previously used by other researchers [8-11]; It ensures more accurate results to calculate the Young Modulus (E), instead of just using the reading of Bose displacement. Hence, in this study optical extensometer is used for the linear portion of the stress-strain curve to obtain the module of elasticity (E) precisely; for the rest of the curve Bose test system output used.

As a result of the tensile test, Bose testing machine provides three outputs, namely, time, force, and displacement. Also the optical extensometer provides strain outputs for the elastic region of the specimens. Therefore, integration of these two outputs could provide a precise stress-strain curve for each specimen.

2.4.2 Wide angle X-ray diffraction (WAXD)

To investigate the crystalline structure of the PP samples and the effects of recycling, annealing and fiber glass addition, Wide Angle X-ray Diffraction (WAXD) was carried out using the “Rigaku Ultimate IV Multipurpose X-ray Diffraction System Diffractometer” with Cu as the anode at 40 kV and 44 mA. Diffraction angle was from 10˚ to 110˚ by radiation wave length of λ=1.54 Å.

Therefore, all sample types were tested to see the crystalline structure of the samples and the effects of recycling on their crystalline structure. Hence, the crystalline fraction (X_c) was calculated using:

$$X_c = \frac{\sum A\ crystalline}{\sum A\ crystalline + \sum A\ amorphous} \times 100\% \ [25];$$

which $A_{amorphous}$ is the area under the amorphous background curve and $A_{crystalline}$ is the area under the first few main crystalline peaks and above the background curve.

2.5 Measurements and calculations

According to the ASTM D638-10 [4], injection molded specimens were used. Specimens' dimensions were determined by taking actual measurements of only one specimen out of each sample. Therefore, width and thickness of the center part of specimens were measured and recorded for future calculations.

Stress and strain values were determined using the measured load, displacement, and also the original specimen length (L_0) and cross-sectional area (A_0) as:

$$\text{(Stress)}\ \sigma = \frac{F}{A_0} \qquad , \text{(Strain)}\ \varepsilon = \frac{\delta}{L0}$$

As a result of plotting stress ($\boldsymbol{\sigma}$) against strain (ε), an engineering stress-strain curve is obtained. From the stress-strain curve specific information about each sample could be determined. Figure 2.3 shows a sample stress strain curve. Beginning of the curve starts with a linear approach (A); this linear portion of the curve obeys Hooke's law and the slope of this region called module of elasticity or Young's modulus (E), at this region, material is in elastic region and any deformation is not permanent. As strain increased, the stress-strain curve approach non-linearity; this phase change point called proportional limit which is the start point of the plastic region of the curve (B) and after this point any deformation will be permanent. Continuing, the curve reaches a peak which called UTS point (C) and at this point necking begins and the actual cross section area is reduced, so the true UTS value cannot be calculated, so the engineering stress value is lower than the true stress and the load begins to decrease until the specimen breaks at fracture point (D).

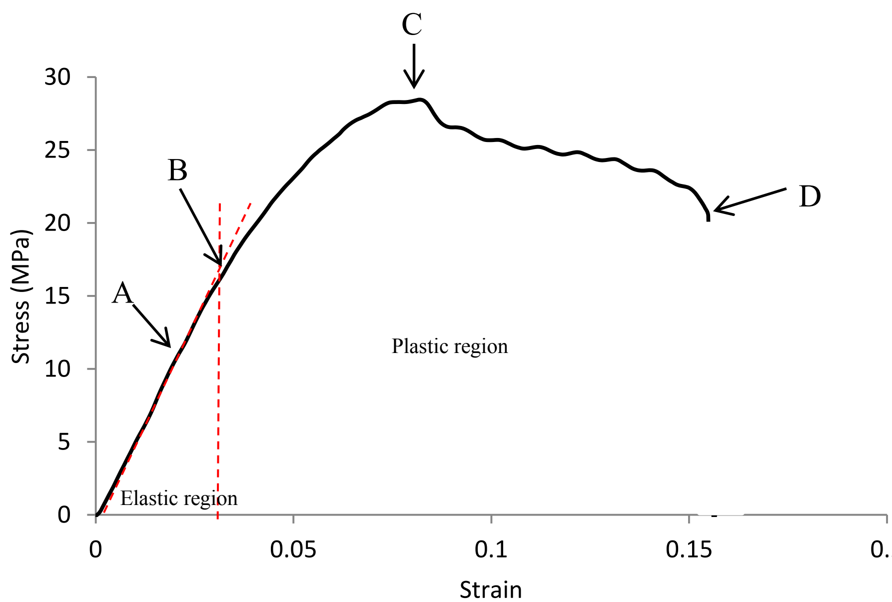

Figure 2-3: Sample stress-strain curve

After conducting tests for each series of samples the average value calculated was reported as the property of that series of samples. Also, standard deviation calculated and reported as follows: $s = \frac{\sqrt{(\sum X^2 - n\bar{X}^2)}}{n-1}$

Where:

S = estimated standard deviation

X = results of one sample experiment

n = number of repeated tests, and

$\bar{X}$ = Average value of a series of tests.

Finally the results are compared and analyzed using statistical analysis tools by STATISTICA software. Effects and correlation between different factors of the involved factors on mechanical properties of tested materials studied using different tools such as ANOVA, normal distribution, Pareto chart, and means plots.

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Chapter 3 : Effects of Recycling on Mechanical Behavior of Polypropylene at Room Temperature through Statistical Analysis Method

3.1 Introduction

Polypropylene (PP) is a thermoplastic polymer which is also known as polypropene. PP is one of the most widely used polyolefin polymers. PP is a commonly used semi crystalline polymer material as it provides a balance of strength, modulus and chemical resistance at also a relatively low cost and weight [1]. Mechanical behavior of PP has been investigated by many researchers over the years. Hartmann et al. [2] tested machined dog-bone specimens from a polypropylene sheet and Duffo et al. [3] tested machined specimens out of cylindrical extruded rods of polypropylene; they have reported the tensile yield behavior of polypropylene at a range of 20°C to 150°C temperatures. Arruda et al. [4] evaluated deformation of polypropylene at various strain rates. Furthermore, other researchers tried to find the combination effects of temperature and strain rate of injection molded unfilled and talc-filled homopolymer polypropylene using uniaxial tensile tests [1-5]. Zhou et al have used experimental methods and constitutive equations to evaluate and predict the combination effects of temperature and strain rate of polypropylene [1,5].

In today’s world, plastics are playing an essential role and they become involved in all aspects of human life; although plastics are very useful due to their wide range of properties and flexibilities, it should be noted that current approaches in plastic product production, consumption and disposal are not sustainable and causes concerns for environment and human health [6]. Hence, appropriate use and disposal, and especially recycling, is essential. Several researchers investigated the effects of recycling on the microstructure and the mechanical properties of polypropylene [7-11]. V.A. González et al studied on chemical and molecular weight changes of polypropylene through multiple extrusions and they found no changes in the polypropylene chemical structure [8]; Y. Long et al investigate on mechanical properties of recycled polypropylene and they revealed that recycling decreased the impact strength however it has no significant effect on tensile properties [9]; From the literature [10,11] it can be concluded that within 3 times of recycling, no significant changes are seen in the material properties of polypropylene; although, by increasing the times of recycling, improvement in the material properties could be achieved due to higher crystallinity of recycled materials [10].

In this article, firstly the effect of recycling (reprocessing) on the mechanical behavior of polypropylene is investigated at room temperature. Secondly, the effect of post-manufacturing thermal treatment (will be referred to as annealing from this point forward) was investigated as another factor which could affect the mechanical properties of the material. Finally, the effects of adding fiber to recycled polypropylene were studied. Main test methodology used in this work was uniaxial tensile testing. Tensile testing is one of the most commonly used material testing technique to evaluate mechanical properties of materials, which is also inexpensive and fully standardized [12].In order to investigate the main effects and the interactions of these variables Design of Experiments (DoE) was implemented. At the end, a sample drop test simulation was performed by UG NX NASTRAN software on a box as a case study.

3.2 Materials and Methods

3.2.1 Materials

The Polypropylene material which is used in this study called "PP CO, no-break"; which means polypropylene copolymer and has a melt rate of 12-16 based on standardized melt flow test [13]; no-break refers to the material IZOD impact test [14] results with no breaking. The PP material was supplied by "Chase Plastics Redefining Resin Distribution" company. Virgin polypropylene pellets come from the material supplier and have never before been molded into parts. Therefore, virgin PP specimens are specimens that have been molded with virgin PP pellets material. Recycled PP made from two times reprocessing of parts which is made out of "20 melt, PP CO, no-break" material, into uniform pellets at Drader manufacturing ltd, (Edmonton, Canada) before molding to our specimens [15].

3.2.2 Sample preparation

Sample types: Five different weight percentages of recycled PP (0 w%, 25 w%, 50 w%, 75 w%, and 100 w %) were blended with the virgin PP. The other factor considered in this study was the effect of adding fiber to the recycled materials, but only one level of 7.5 w% of glass fiber was added to the recycled materials. Reinforced pellets were purchased from "Chase Plastics Redefining Resin Distribution" company with 15 w% glass fiber in PP pellets. This limited different combinations of "recycled+ virgin+ fiber" samples. Also, it was important to have a consistent percentage of recycled materials to have controlled comparable experiments. Consequently 8 different types of specimens were prepared for this study. Table 3.1 shows all different samples and their blending combinations used in this study.

Injection molding: According to ASTM D638 [12], type V specimens were made through an injection molding process, Fig. 3.1 shows a schematic view of the type V specimens. Polypropylene samples were injection molded at Drader manufacturing industries Ltd. As specimens are small a mold was designed to produce 4 dog-bone samples simultaneously and also a 1 degree taper was designed to prevent sticking in the mold. Virgin, recycled, and fiber resins of polypropylene were blended and then vacuumed to the injection molding system to produce samples based on mentioned samples recipes. For consistency, the same injection molding process specifications were followed to make all the specimens; namely, barrel temperature of 227°C; melting temperature of ~ 210°C; injection time sets to 5 seconds to ensure that the part is fully evenly filled; cooling time was 35 seconds to ensure that the specimens will have minimal shape change after cooling outside of the mold; injection pressure was 103 MPa; and packing (holding) pressure was 98 MPa and for three seconds [15]. All samples were packed and stored in the same place before and during the experiments for consistency.

Table 3-1: Polypropylene sample recipes

Recipes	Recycled PP	Virgin PP	Glass fiber
1	0%	100%	0%
2	25%	75%	0%
3	25%	67.50%	7.50%
4	50%	50%	0%
5	50%	42.5%	7.5%
6	75%	25%	0%
7	75%	17.50%	7.50%
8	100%	0%	0%

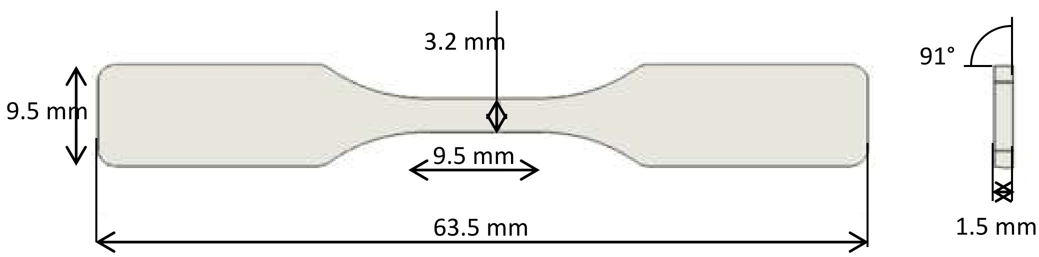


Figure 3-1: Schematic view of the dog-bone tensile specimens.

Annealing: It is a heat treatment process which relives the stress inside the specimens by slowly heating and cooling the samples. The industry standard for annealing is to start at room temperature, increase the temperature by 10°C/hr up to the specific isothermal temperature, isothermal for 2 hours, and finally decrease the temperature with the same rate to reach the room temperature. In this study samples were annealed at 150°C; based on previous research where a range of 140°C to 160°C were suggested [16,17] when compared to the melting temperature of polypropylene (190°C), this annealing temperature is a high-moderate temperature which could significantly improve the specimens properties [17]. However, in parallel another colleague is also investigating the effects of annealing at 140°C and 160°C. Therefore out of each type of produced samples, half of them annealed and the rest stored at the room temperature; therefore, comparison between the same type of annealed and not annealed samples could be carried out. Hence, 8 (type of samples)× 2 (annealed and not annealed) =16 group of samples should be tested in total.

3.2.3 Experimental method (Tensile test):

As mentioned in the previous section, 16 different groups of samples, including annealed and not-annealed, were prepared for the tensile test. For each group of samples, five replicates of tensile test performed, resulting in 80 tensile tests. As samples are injection molded, specimen's dimensions were determined by taking actual measurements of one specimen out of each batch (5 specimens were tested for each batch) [12]. Therefore, width (3.3 mm), thickness (1.6 mm), and gauge length (7.62 mm) of the specimens measured and used for stress and strain calculations. Tensile tests were performed according to ASTM D638 on a Bose 3200 test system. A crosshead speed of 10 mm/min was used and the specimens were tested at room temperature (~20°C). An in-house built optical extensometer was used to process samples for the linear portion of the stress-strain curve to obtain the module of elasticity (E).

Statistical analysis was used to have a better understanding of the effects of considered factors (recycled material percentage, annealing condition, and glass fiber additive percentage) on mechanical behaviour of polypropylene copolymer. Specifically, ANOVA analysis and means plots, and surface plots were conducted.

3.2.4 Drop test simulations (Case study)

Canada post container (CPC) is a box to store and carry mail. CPC is being produce in Drader manufacturing industries Ltd (Drader), in Edmonton, AB. Based on material properties from the tensile test results, drop test simulations were performed using UG NX Nastran software to simulate the drop performance of the part; hence, a tree-dimensional (3D) drop test simulation were used.Drop test simulations were only performed on not-annealed, non-reinforced samples (due to company's interest) by simulating the dropping process in different points and vectors (see Fig. 3-2). Material properties for each simulation used from the tensile test results. The simulation for each material performed for room temperature and in 5 different directions (corner point, bottom side and 3 edges). Therefor, 25 simulation processes were done in total.

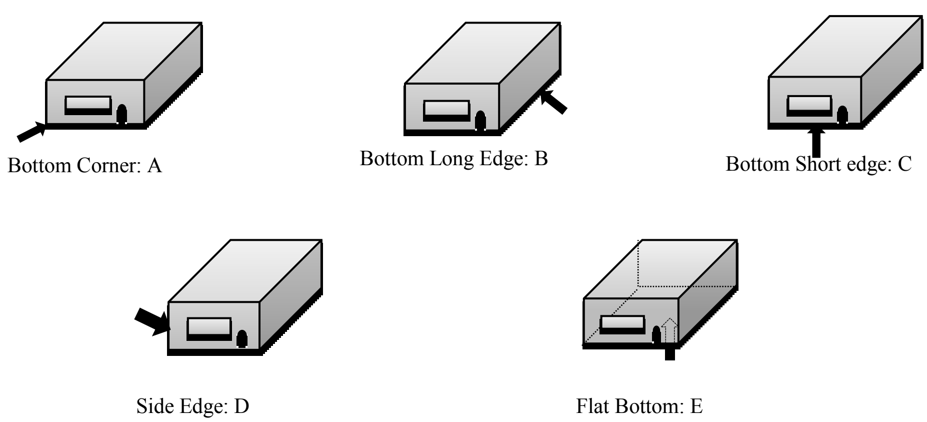
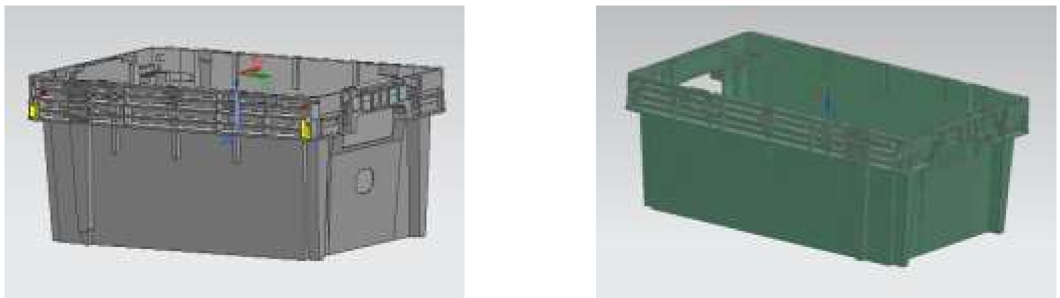


Figure 3-2: Drop orientations and vectors

To start the simulation, firstly the model was simplified to remove non-significant features such as edge blends which could cause mesh errors; then, the model was meshed and meshed errors corrected to run the simulation with fewer errors (See Fig. 3-3), the final meshed model has 101286 number of elements and 203381 number of nodes in the mesh.



a. *b.*

Figure 3-3: Simplified model (a) and meshed model (b) of the Canada post box

3.3 Results and discussion

3.3.1 Tensile test results

Tensile behavior of the virgin, reprocessed, and reinforced reprocessed polypropylene is investigated. Also the effect of annealing on polypropylene samples was studied. Tensile tests performed at room temperature (~20°C) based on ASTM D-638 standard methods [12]. A characteristic stress-strain curve of the specimens is shown in Figure. 3.4. The curves starts with a clear linear region up to proportional limit to reach the yield point; then deformation starts by necking, next material shows post stress-whitening up to UTS point and finally fracture occurs.

Specimens with no reinforcement had an average± standard deviation of UTS and E of 24.21± 0.2 (MPa) and 792.66± 31.53 (MPa) at the not annealed condition, respectively. The elastic modulus as a function of recycled PP percentage is plotted in Fig. 3.5-a. The elastic modulus shows a slight improvement (about 10%) by using more percentage of recycled material which could be due to higher crystalline structure and stiffness of recycled PP at room temperature [10]. As the Young's modulus, ultimate tensile strength plot also shows the same trend (less than 10% improvement) (see Fig. 3.5-b). The results for the yield strength (σ_y) and elongation at break (ε_b) also indicate about 5% change by using more recycled PP (see Fig. 3.5-C & D). Therefore it could be concluded that using more recycled material has slight positive effect on mechanical properties of polypropylene which could be very useful for industries to reduce the cost by using more recycled material and also it is an environmental friendly solution.

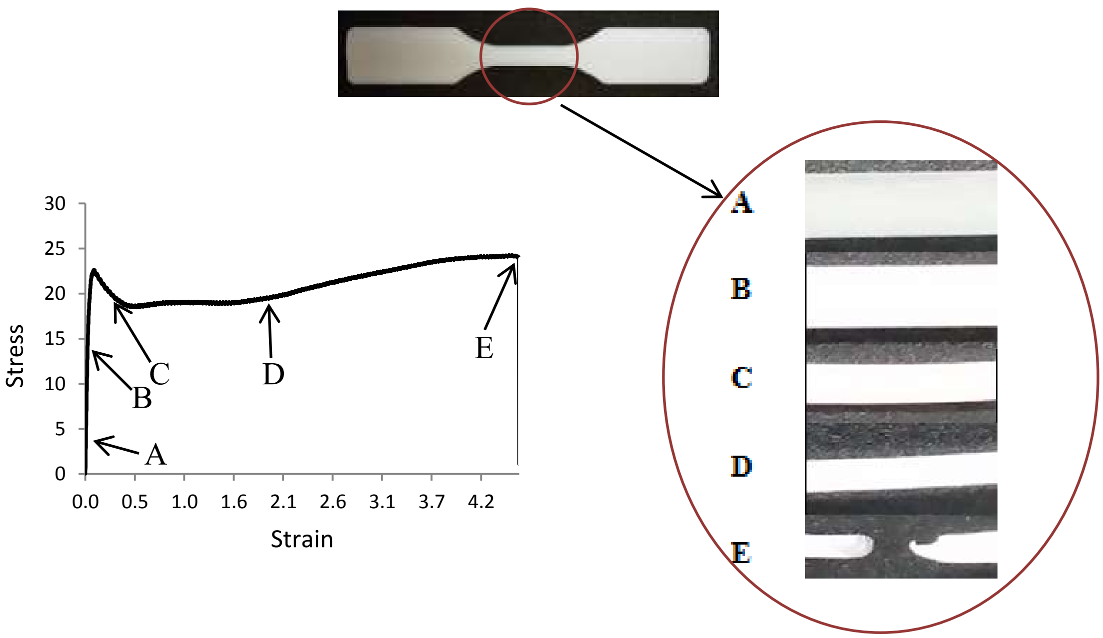


Figure 3-4: Stress-strain curve and sample deformation process through different times of the tensile test; elastic deformation (A), proportional limit (B), necking (C), stress-whitening (D), fracture (E) (12).

UTS of 24.49± 0.4 (MPa) and E of 989.66± 161.2 (MPa) recorded as average± standard deviation for the non-reinforced samples after annealing. Hence, to show the effects of annealing investigated on "recycled+ virgin non-reinforced PP" samples, Fig. 3.6 compares the stress-strain curve of the materials at annealed and non-annealed conditions. From this figure it is observed that annealing has rigidity improvement on the mechanical properties of polypropylene in a way which changed the category of materials from a soft and tough polymer to a hard and tough category which means the material became more rigid by improvement of the young modulus [18]. To be more specific, Fig. 3.7-A & B indicate that annealing the samples at 150°C increased the Young's modulus (E) less than 50% and the yield stress (σ_y) by more than 10 %; which is due to enhancement of material crystallinity and crystal size by heat treatment of the samples [16]. Fig. 3.7-A also indicates that annealing made more improvement in young's modulus of samples with more recycled percentage (~ 50% improvement) compared with virgin samples (~10% improvement); however, annealing had comparatively same improvement on yield stress of all "recycled+ virgin PP" samples by almost 10% (See Fig. 3.7-B).

UTS of 31.3± 4.3 (MPa) and 31.11± 3 (MPa); and Young's modulus of 1136.± 94.6 (MPa) and 1782± 238.3 (MPa) observed for the reinforced "recycled+ virgin PP" samples at not annealed and annealed conditions, respectively. Effects of adding fiber on mechanical properties of "recycled+ virgin PP" samples plotted in Fig. 3.8; it revealed that adding fiber will increase the elastic modulus and yield stress significantly. It can be seen that the results for the "recycled+ virgin+ fiber" specimens has more variability compared with the non-fiber samples; also, this variability was more significant in Young's modulus (E) results (see Fig. 3.10-A). Fig. 3.8-B also indicates a decreasing trend in yield strength (σ_y) by increasing the usage of recycled material percentage. Effects of annealing on glass fiber reinforced samples are shown in Fig. 3.9; it indicates that elastic modulus of the "recycled+ virgin+ fiber PP" samples increased by more than 50% after annealing the samples, although the effect on the yield stress was not significant.

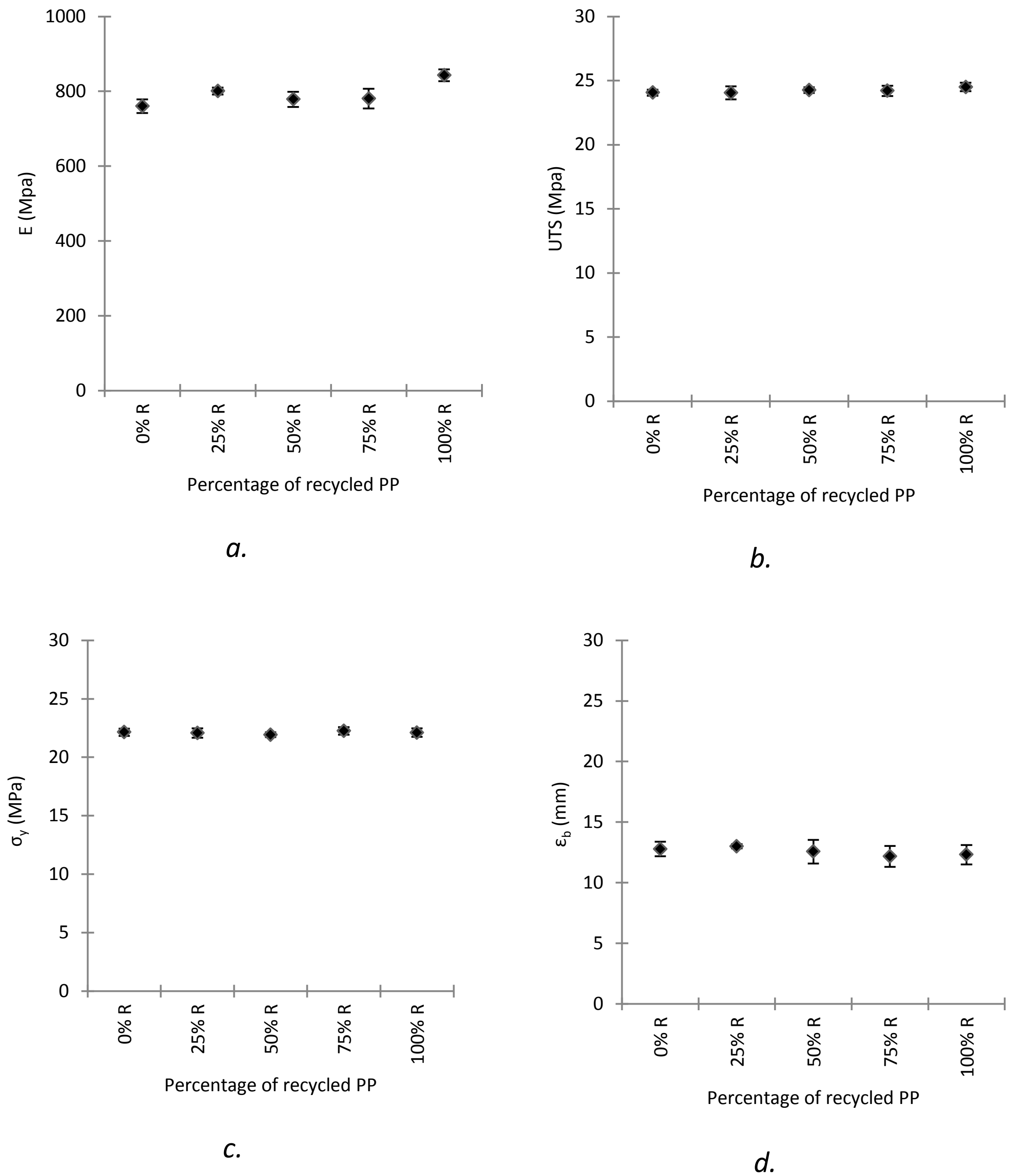


Figure 3-5: Effects of recycled material percentage on the mechanical properties of "recycled+ virgin PP" samples; Elastic modulus vs. percentage of recycled PP (a); Ultimate tensile strength vs. percentage of recycled PP (b); Yield stress vs. percentage of recycled PP (c); Elongation at break vs. percentage of recycled PP (d).

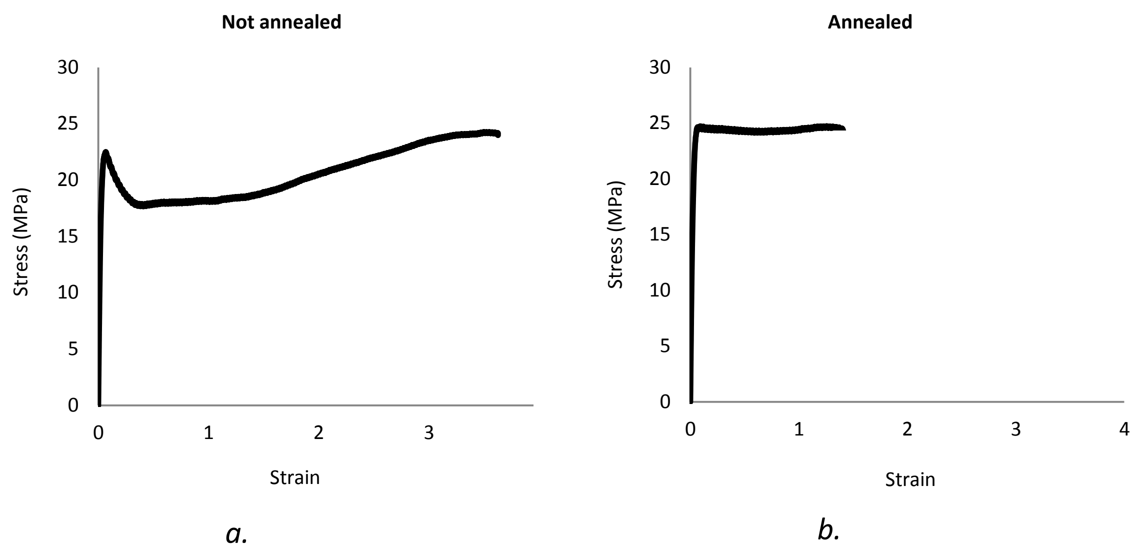


Figure 3-6*: Effects of annealing on a sample stress-strain curve; not annealed PP sample (a), annealed PP sample (b).*

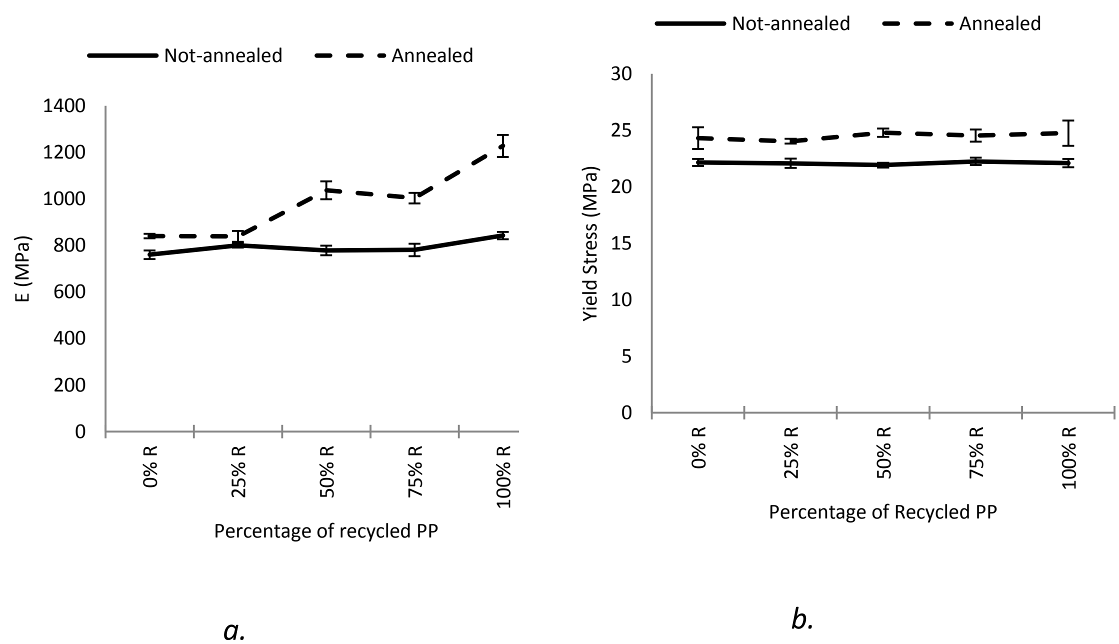


Figure 3-7*: Effects of annealing on "recycle+ Virgin PP"; Elastic modulus (E) vs. percentage of recycled PP (A); Yield stress (σ_y) vs. percentage of recycled PP (B).*

Table 3-2: Mechanical properties of different percentage of "recycled+ virgin+ Fiber" PP samples (average ± standard deviation)

Polymer	UTS (Mpa)		E (Mpa)		Elongation at break (mm)	
	not-annealed	Annealed	not-annealed	Annealed	not-annealed	Annealed
0% R PP	24.06± 0.23	24.38± 0.89	760.24± 18.6	840.47± 9.35	12.78± 0.6	6.31± 3.14
25% R PP	24.04± 0.51	24.06± 0.23	800.49± 9.35	839.68± 23.5	12.57± 0.26	6.53± 1.1
50% R PP	24.26± 0.22	24.96± 0.61	778.72± 20.4	1036.58± 38.5	12.57± 0.8	6.53± 3.4
75% R PP	24.21± 0.4	24.78± 0.77	780.86± 26.4	1003.71± 22.4	12.37± 0.67	8.88± 3.2
100% R PP	24.50± 0.34	24.29± 1.8	842.97± 16	1227.88± 47.4	12.41± 0.7	5.14± 2.45
75% R PP+ 7.5% F	26.72± 2	27.70± 0.43	1238.84± 360	1916.62± 540	1.16± .01	1.56± 0.17
50% R PP+ 7.5% F	31.82± 2	32.36± 0.66	1119.27± 440	1922.37± 376	4.95± 1.11	2.97± 1.18
25% R PP+ 7.5% F	35.32± 0.27	33.27± 1.3	1052.03± 350	1506.73± 415	6.21± 1.44	3.65± 1.55

Finally, the effects of all considered factors; namely, Recycled PP percentage, annealing, and fiber; summarized schematically and numerically in Fig. 3.10 and Table 3.4. The "recycled+ virgin+ fiber" annealed PP samples had the highest Young's modulus (E) compared with "recycled+ virgin+ fiber" not-annealed, "recycled+ virgin" annealed, and "recycled+ virgin" not-annealed PP samples; however they had similar yield strength results compared to the same not annealed samples. Also, it can be seen that more usage of recycled material only results in changes less than 10% in mechanical properties of non-reinforced, fiber reinforced, annealed or not- annealed PP samples. It also revealed that interaction of annealing and fiber reinforcing has the most significant effect on Young's modulus improvement (See Fig. 3.10-A); however, fiber reinforcing has the highest effect to improve the yield stress of PP samples (See Fig. 3.10-B).

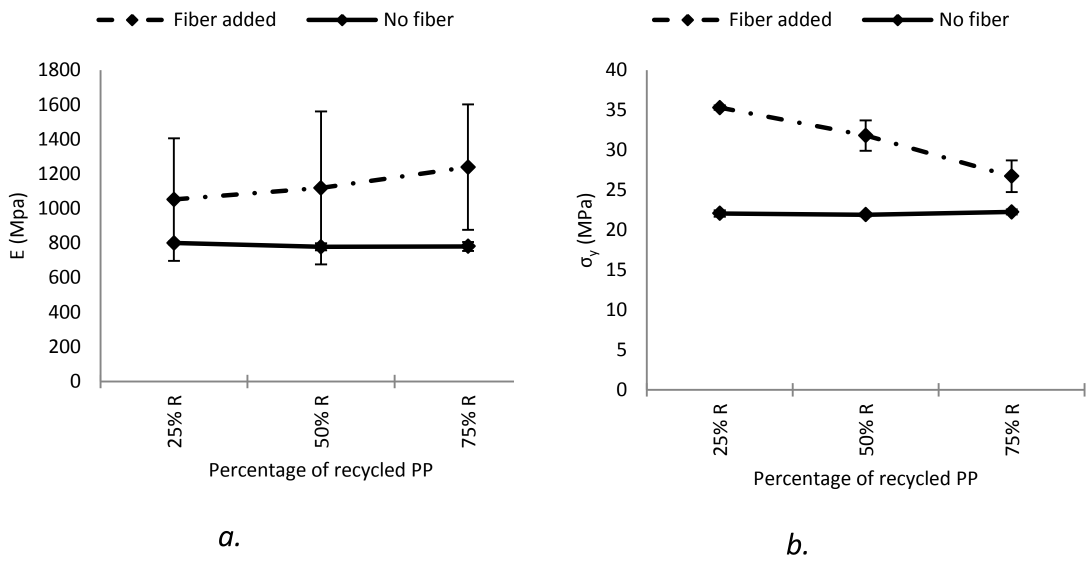


Figure 3-8: Effects of adding fiber on "recycled+ virgin PP"; Elastic modulus (E) vs. percentage of recycled PP (a); Yield stress (σy) vs. percentage of recycled PP (b).

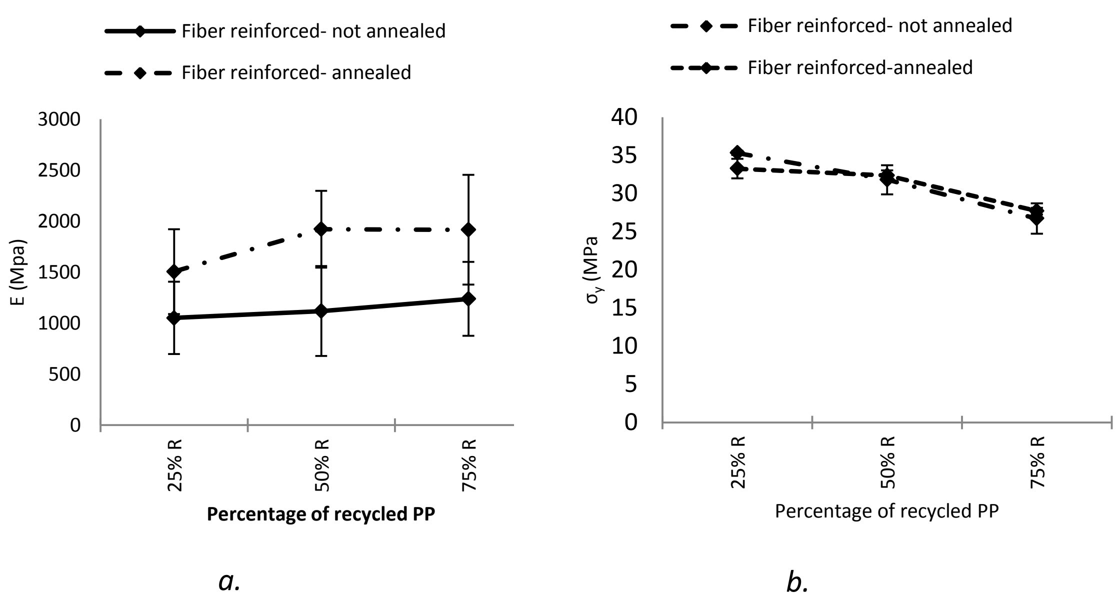


Figure 3-9: Effects of annealing on "recycle+ virgin+ fiber PP"; Elastic modulus (E) vs. percentage of recycled PP (a); Yield stress (σy) vs. percentage of recycled PP (b).

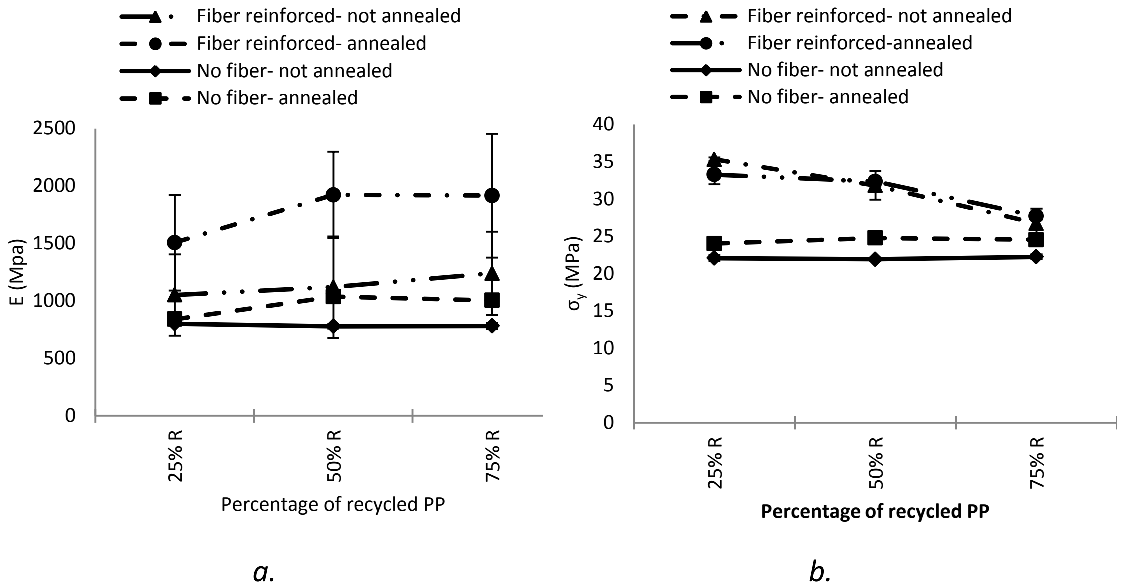


Figure 3-10: Summery of all effects; Elastic modulus (E) vs. percentage of recycled PP (a); Yield stress (σy) vs. percentage of recycled PP (b).

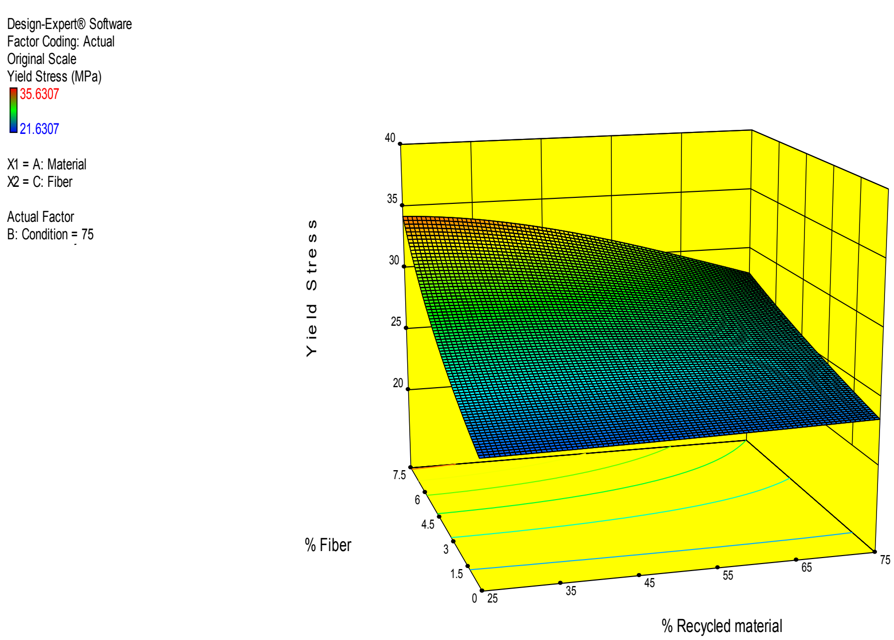


Figure 3-11: Surface plots of effects; interaction effects of fiber and recycled PP% on yield stress

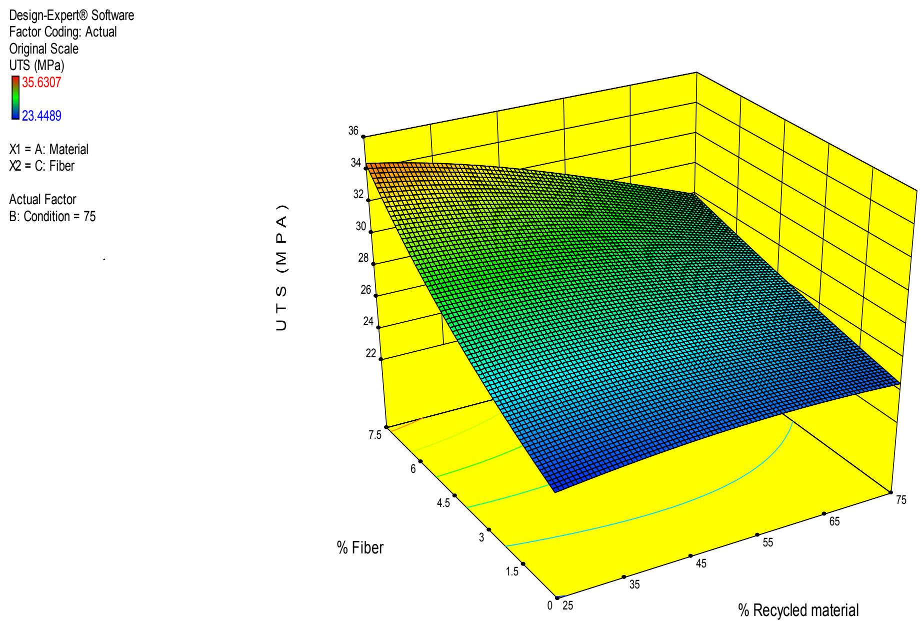


Figure 3-12: Surface plots of effects; interaction effects of fiber and recycled PP% on UTS

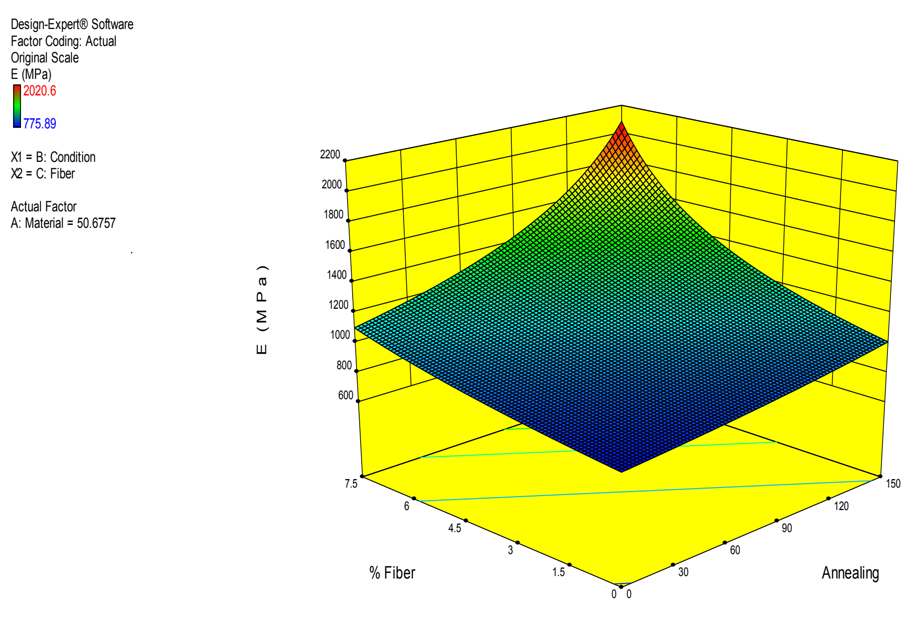


Figure 3-13: Surface plots of effects; effects of annealing and fiber on E

Additionally, ANOVA analysis was conducted using "Design Expert 9" software, on transformed data (for respective responses) for the "recycled+ virgin+ fiber" PP samples at annealed and non-annealed condition. According to the ANOVA results, effects of factors on response variables were significantly fit into a "response surface reduced cubic" model; which was recommended for all considered responses.

For the yield stress results, ANOVA analysis performed on power transformed data by λ=-2.36 which recommended by Box-Cox plot. It determined that all considered factors (w% recycled material, annealing, and fiber) has significant effect ($p< .05$) on yield stress results. Also, it revealed that interaction effects of fiber with the recycled PP w% is significant (see Fig. 3.11); and only % recycled material has significant quadratic effect on yield stress results of "recycled+ virgin+ fiber" PP samples.

ANOVA analysis was also performed for the UTS results of the "recycled+ virgin+ fiber" PP samples; power transformation by λ=-1.48 recommended by Box-Cox plot. It revealed that all considered factors have significant effect on UTS; however, quadratic effects of material and interaction effects of material and fiber are significant (see Fig. 3.12).

The same analysis performed for E results using power transformation of λ=-1.9 fitted to the same model as other responses; it observed that all considered factors have significant effect on E; Also, quadratic effect of material, interaction effect of annealing and fiber are significant (See Fig. 3.13).

To check the validity of the fitted “response surface reduced cubic” model, actual measured values compared with the predicted values by this model and it revealed that the error was 3, 0.7, and 11 percent for yield stress, UTS, and E respectively which indicates an appropriate fit between the model and the actual results.

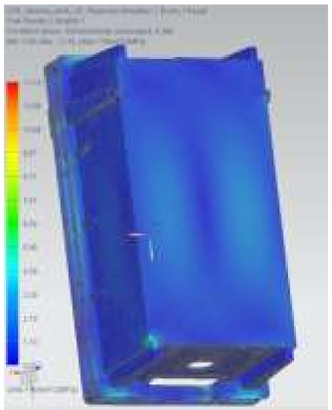
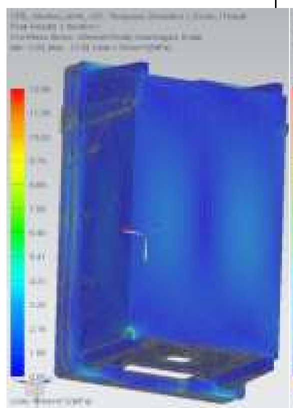
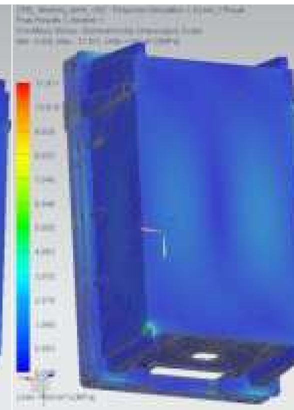
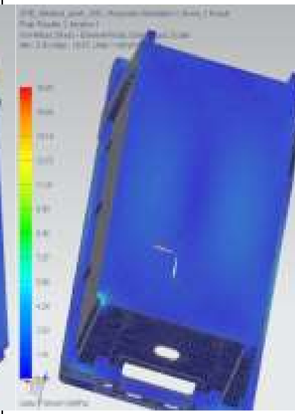
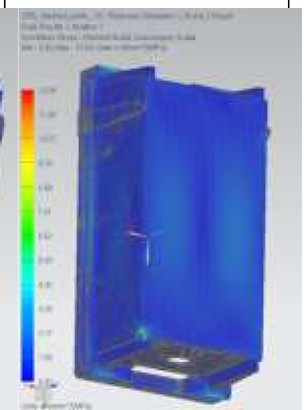
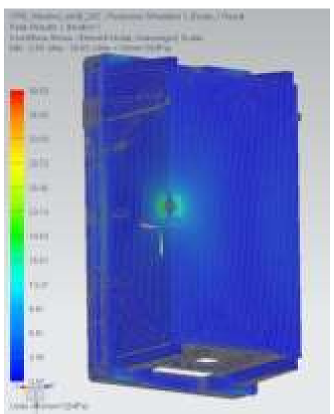
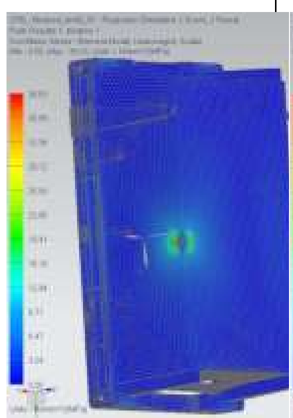
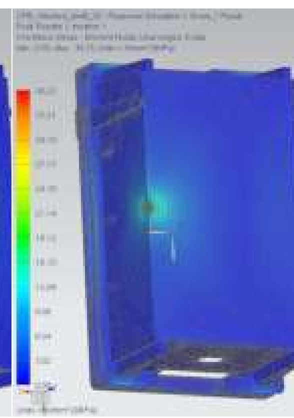
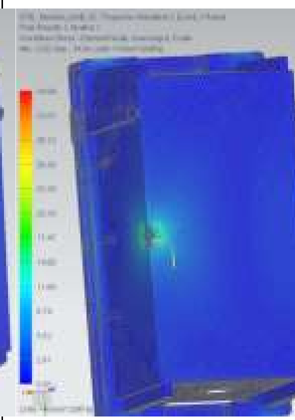
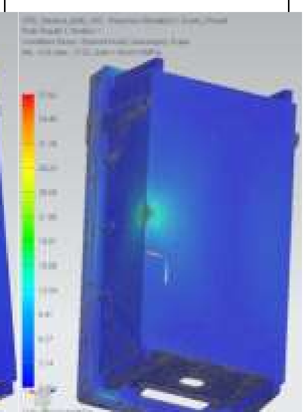
3.3.2 Drop test results

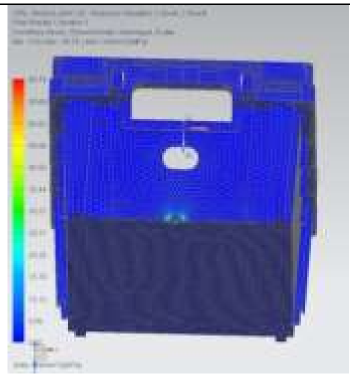
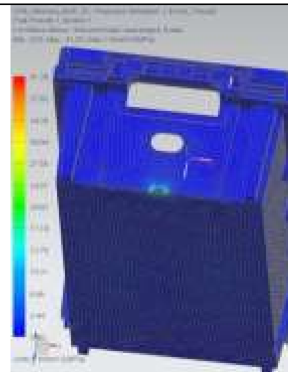
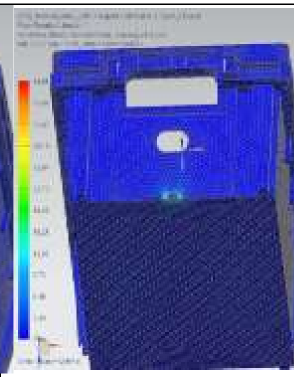
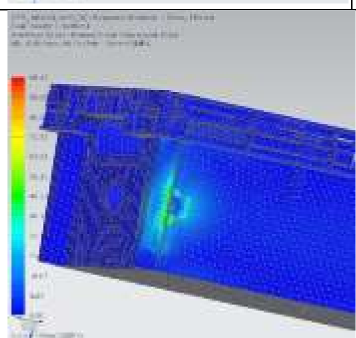
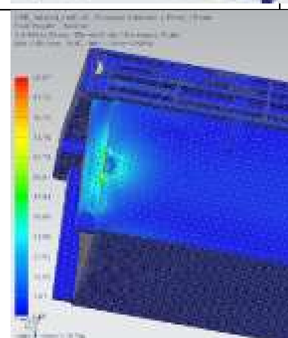
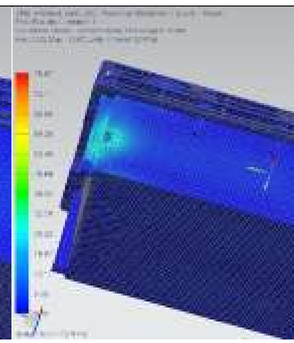
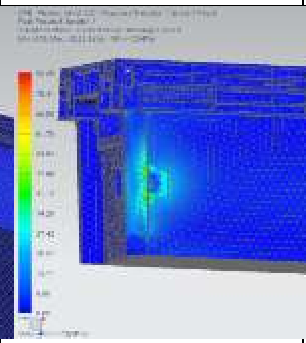
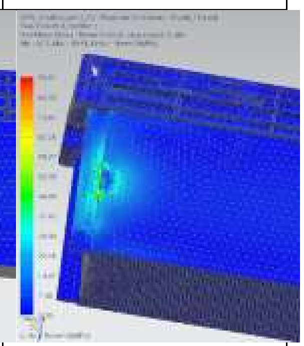
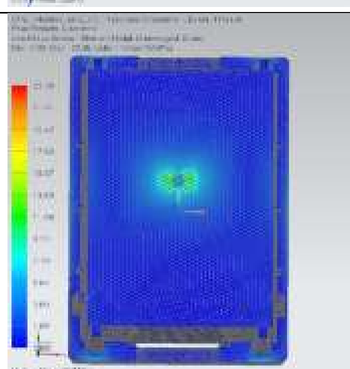
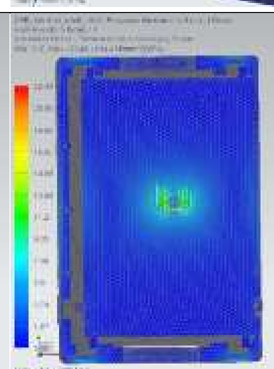
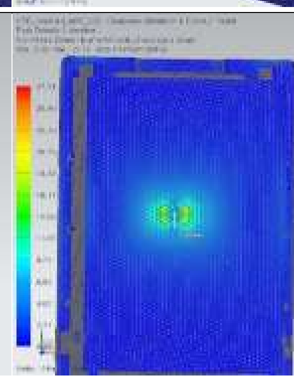
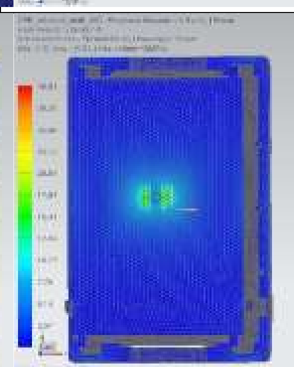
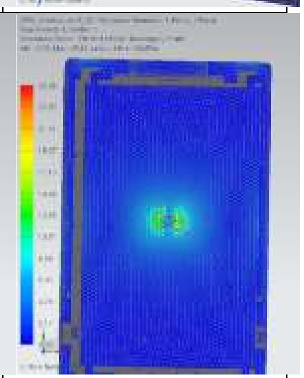
The drop test simulation results are shown in table 3.3 and 3.4 numerically and schematically, respectively. It represents that the 50w% recycled material has the minimum average for the maximum stress applied to the box as a result of drop impact. However, looking at the average values of different drop directions, it seen that it has more significant effect on the max stress values; therefore, changes in the design of the part would be suggested.

Table 3-3: Summery of the maximum stress applied to the box (MPa)

	Bottom corner (A)	Bottom long edge (B)	Bottom short edge (C)	Side edge (D)	Flat bottom (E)	**Average**
0w% Recycle	13.16	39.63	60.75	94.43	23.36	46.27
25w% Recycle	12.99	38.83	41.26	95.67	22.43	42.24
50w% Recycle	11.911	36.23	29.98	78.67	27.71	36.90
75w% Recycle	16.97	34.94	38.91	82.26	30.81	40.78
100w% Recycle	13.04	37.62	65.19	89.81	25.69	46.27
Average	13.61	37.45	47.22	88.17	26	13.61

Table 3-4: Schematic view of the drop test simulations

	0w% Recycle	25w% Recycle	50w% Recycle	25w% Recycle	0w% Recycle
A					
B					

C					
D					
E					

3.4 Conclusion

Tensile behavior of the virgin, reprocessed and reinforced reprocessed polypropylene at annealed and not-annealed condition allowed mechanical properties to be investigated. The effects of adding different percentage of recycled polypropylene to the virgin polypropylene, the effects of glass fiber reinforcement on reprocessed polypropylene and annealing were investigated. Tensile behavior was studied at the room temperature (~20°C) using ASTM standard D638 [12].

Mechanical testing of properties for “recycled+ virgin” samples revealed that greater usage of recycled PP in the specimens does not significantly improve the UTS and E (less than 10 %); however, the yield stress (σ_y) results had a more constant trend. Investigations on the effects of annealing on “recycled+ virgin PP” samples indicates that annealing improves the material performance by making PP samples harder and stronger as it improved E by about 50%,and σ_y by more than 10%. Effects of adding fiber to “recycled+ virgin PP” samples shows a significant improvement in mechanical properties of the PP samples by about 50%; however it has a higher variability compared to non-reinforced PP samples. Furthermore, “recycled+ virgin+ fiber PP” samples were annealed to see the effects of annealing on reinforced reprocessed PP samples; it revealed that annealing has significant effect on Young’s modulus of reinforced samples (more than 50% improvement) compared with non-reinforced PP samples (10-40 % improvement).

Finally it is concluded that, using higher percentage of recycled material will not have a significant effect on mechanical properties of materials, therefore, to minimize the cost, more recycled material could be used without significant reduction in material performance. Adding fiber had the greatest effect in improving the young modulus and UTS of the specimens. Annealing also had some correlation with the recycled percentage used in the specimens. Finally, the drop test simulation results revealed that the 50w% recycled content PP sample has the minimum average of drop impact stress at all five tested drop points, with the average maximum impact stress of 36.90 MPa; however, greater variability in impact stress was observed depending on how the CPC was dropped.

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Chapter 4 : Effects of Recycling on Mechanical Behavior of Polypropylene at Cold Temperatures through Statistical Analysis Method

4.1 Introduction

During the past several years, plastic material usage has grown and plastics are involved in all aspects of human life. Rapid development of plastics is due to their versatile properties and flexibilities, although it increases environmental awareness which leads to appropriate use and disposal. These days recycling, is an essential environmentally acceptable green movement which is commonly considered in industries [1].

Polypropylene (PP), the focus of this article, is a well-known semi crystalline polyolefin thermoplastic polymer. PP is widely used for different applications because of its low cost and weight in combination with a balance of strength, modulus and chemical resistance [2]. Several researchers have reported the mechanical and chemical behavior of PP in the past [2- 9]; some researchers investigated the tensile behaviour of polypropylene at different temperatures (20°C up to the melting point) and strain rates; they obtained the strain rate and temperature sensitivity of PP [2- 5]. Other researchers investigated on the effects of recycling on chemical and mechanical behaviour of polypropylene [11, 7, 8]. V.A. González et al. [9] investigated on chemical and molecular weight changes of PP through multiple extrusions which resulted in no changes in PP chemical structure; Y. Long et al. determined that recycling decreased the impact strength, while it made no significant changes on tensile properties [10]. Other researchers concluded that up to 3 times of recycling, no significant changes are seen for material properties of PP [11, 12]; however, increasing the recycling cycles, improved the material properties, as recycled materials are more crystalline [11]. Several researchers used wide angle x-ray diffraction (WAXD) method to investigate on crystalline structure of polypropylene to find a clue for its behavior [13- 16], and the crystalline fraction is reported.

In this article, the tensile behaviour of polypropylene is studied by considering the effects of cold temperatures (-10°C, 0°C, and room temperature) on the tensile behavior of recycled (reprocessed) content polypropylene samples. Moreover, the effects of annealing and adding glass fiber on the mechanical properties are considered. Additionally, the effects of recycling on crystalline structure of recycle content polypropylene samples are investigated using wide angle x-ray diffraction (WXRD). Design of Experiment was implemented to inspect the main effects and the interactions of these variables. The purpose of this article is to investigate the tensile behaviour of various polypropylene specimens at different temperatures.

4.2 Experiments

4.2.1 Materials

16 different groups of recycled content polypropylene samples which mentioned in previous chapter in detail were also used for the tests in cold temperatures.

4.2.2 Tensile test

Tensile tests were performed according to the ASTM-D638 standard [17] on a Bose 3200 test system with a cold chamber at crosshead speed of 10 mm/min at three different temperatures (room temperature, 0°C, and -10°C). Using a custom made optical extensometer; the module of elasticity (E) was obtained for the linear portion of the stress-strain curve. For the room temperature tests, 5 replicates were performed to ensure the repeatability of the results [17, 20]; as standard deviation of the results were tight for the room temperature results, 3 replicates were performed for the colder temperatures. The cold tests were conducted in a Bose hot/cold chamber. Samples were first tightened in the jaws in the chamber and then conditioned at the test temperature for about 10-15 minutes. Based on ASTM-D638 [17] and as samples are injection molded, samples' dimensions were founded by actual measurements of one specimen out of each group of samples. A stress-strain curve was obtained as a result of each tensile test which was further analyzed to extract the mechanical properties. Average values were reported and standard deviations were used to show the errors.

As mentioned in the previous section, 16 different groups of samples were tested at three different temperatures (room temperature, 0°C, and -10°C) and each test was repeated 3 times which was resulted in 16× 3× 3= 144 tensile tests in total for a full factorial design.

4.2.3 Statistical analysis

Statistical analysis was performed by "Design Expert 9" Software to analyze the mechanical behaviour sensitivity of polypropylene copolymer to the considered factors (temperature, recycled material percentage, annealing condition, and glass fiber additive percentage). ANOVA analysis, means plots, and Pareto charts were conducted to analyze and show the effects of the considered factors.

4.2.4 Wide Angle X-ray Diffraction (WXRD)

To investigate the crystalline structure of the PP samples and the effects of recycling, annealing and fiber glass addition, Wide Angle X-ray Diffraction (WAXD) was carried out using the "Rigaku Ultimate IV Multipurpose X-ray Diffraction System Diffractometer" with Cu as the anode at 40 kV and 44 mA. Diffraction angle was in a range of 10˚ to 110˚ by radiation wave length of λ=1.54 Å. Therefore, 16 (one from each group of samples) square pieces (1 by 1 cm) of dog-bone samples were tested using WAXD method to see the crystalline structure of the samples and the effects of recycling on crystalline structure. Hence, the crystalline fraction (X_c) was calculated using:

$X_c = \frac{\sum A\ crystalline}{\sum A\ crystalline + \sum A\ amorphous} \times 100\%$ [25], which $A_{amorphous}$ is the area under the amorphous background curve and $A_{crystalline}$ is the area under the first few main crystalline peaks and above the background curve.

4.3 Results

Tensile behavior of non-reinforced and reinforced recycled content polypropylene at room temperature was reported in detail in the previous chapter [21]; this article brings the comparison between the room temperature results and the colder temperatures through statistical analysis methods with a focus on different combination of recycled PP. Increase in elastic modulus and UTS by decreasing the temperature on non-reprocessed PP has previously been reported [23, 24]. In addition the crystalline structure is reported.

The maximum responses for the ultimate tensile stress (UTS), and Young's modulus (E) recorded for 25w% recycled PP+ 7.5w% GF at -10°C at 49± 0.2 and 2560± 300 MPa, consecutively; at 0°C the maximum responses achieved at 45± 0.08 MPa for UTS and 2400± 300 MPa as elastic modulus also for the 25w% recycled PP+ 7.5w% GF, although annealing had no significant improvement on these both responses at -10°C and 0°C. Also, based on the room temperature results, 25w% recycled PP+ 7.5w% GF had the highest responses compared to the other combination of factors; however annealing had a more significant effect on room temperature tensile test specimens which is described in detail in the previous chapter.

ANOVA analysis performed on transformed data for each respective response based on response surface model for a full factorial design with 144 runs. Hence, for each responses different transformation performed based on the nature of the results using Design Expert 9 software. For the UTS response, effects of factors were significantly fit to response surface reduced quartic model for the inverse transformed results which was suggested by the software using Box-Cox plot. Therefore, Pareto chart was prepared based on ANOVA analysis to show the significant effect of factors on the UTS. As shown in Fig. 4-1 testing temperature has the most significant effect on the UTS results and next with a big difference, fiber also has significant effect on the UTS results. Also it could be concluded that recycled PPw% (material) and annealing has no significant effect on UTS. Similar to UTS, ANOVA analysis also applied to the yield stress results and the Pareto charts are shown in Fig. 4-2; the results for the yield stress is similar to UTS, and temperature and fiber have the most significant effect, however the effects of fiber is more compared to the UTS results. Next, ANOVA analysis applied for the Young's modulus results using power transformation by λ=-1.7 fitted to the response surface reduced fifth model; it observed that the recycled material percentage have the least significant effect on E; however, material has interaction effect with annealing and temperature factors. Also Pareto chart of the effect of factors was conducted based on ANOVA analysis for the E results (see Fig. 4-3), which determines that again temperature and fiber individually and also their interactions have significant effect on the E results of the tested PP samples. Based on ANOVA analysis to check the validity of the fitted models, actual conducted values compared with the predicted values by the model (see Fig. 4-4 to 4-6); it revealed that for the UTS, yield stress and E, the error of prediction by the model was only 1.07%, 1.16%, and 13.2% respectively; which indicates significant fit between the model and the actual observations.

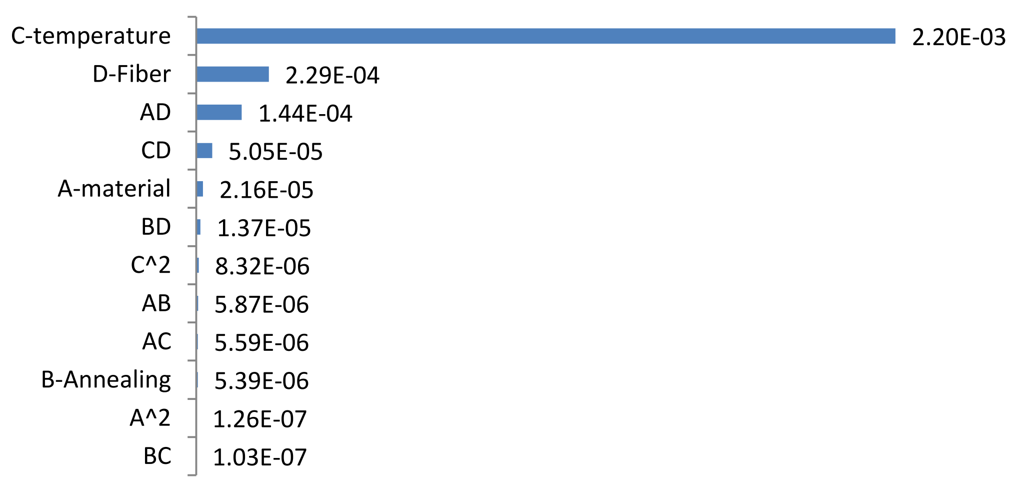


Figure 4-1: Pareto chart of the effects of factors on the ultimate tensile stress of the PP samples.

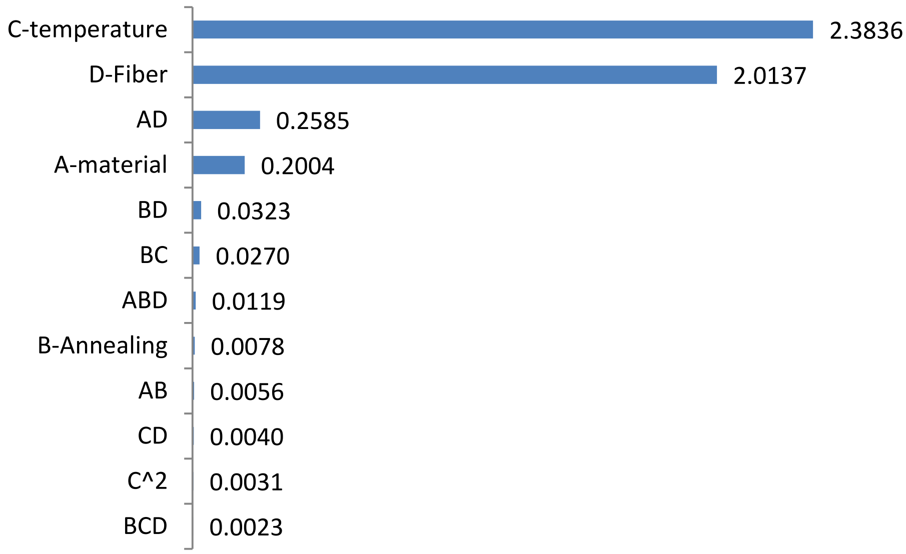


Figure 4-2: Pareto chart of the effects of factors on the yield stress of the PP samples.

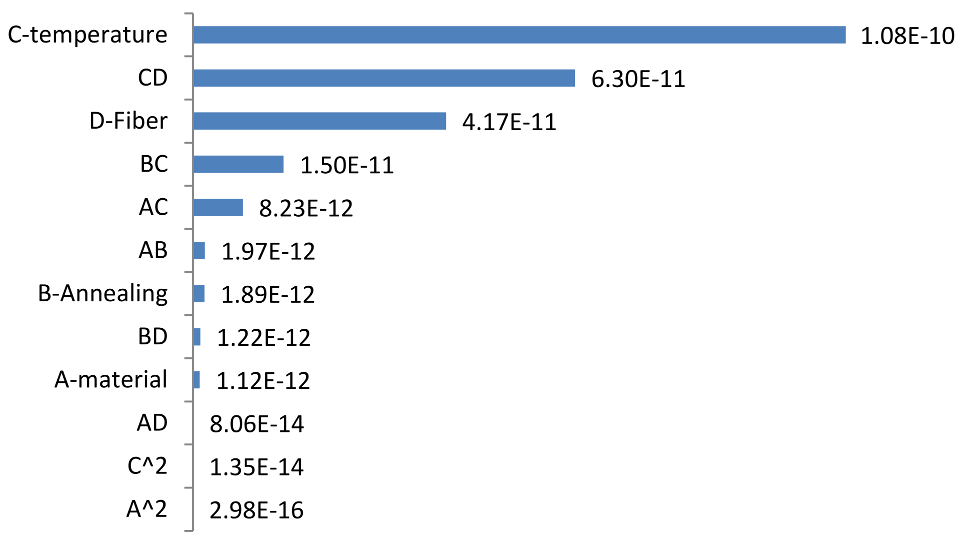


Figure 4-3: Pareto chart of the effects of factors on the Young's modulus (E) for the tested PP samples.

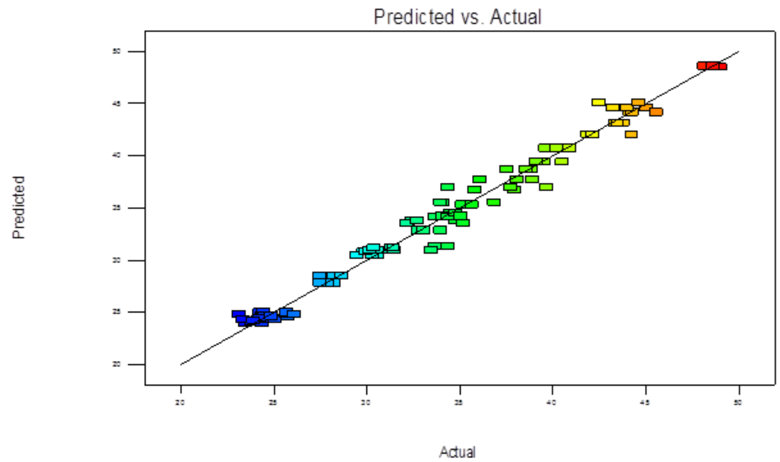


Figure 4-4: The predicted vs. actual graph of the UTS results.

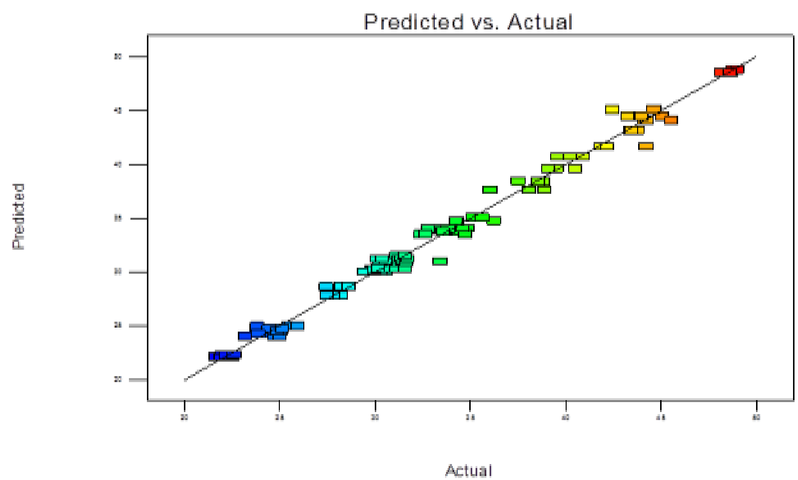


Figure 4-5: The predicted vs. actual graph of the yield stress results.

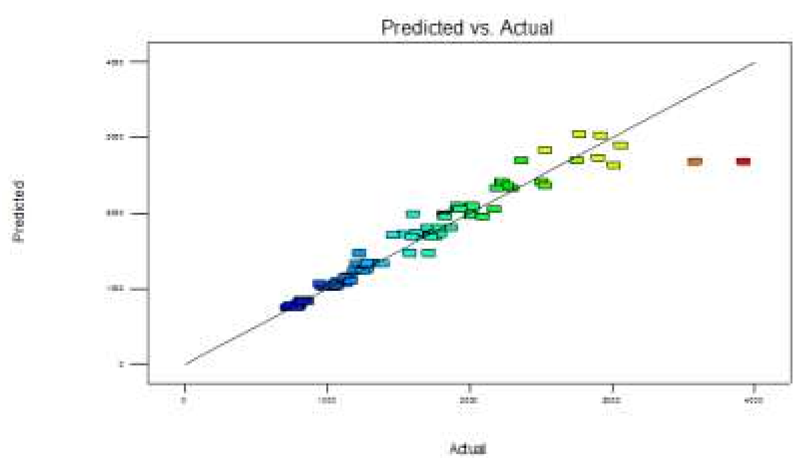
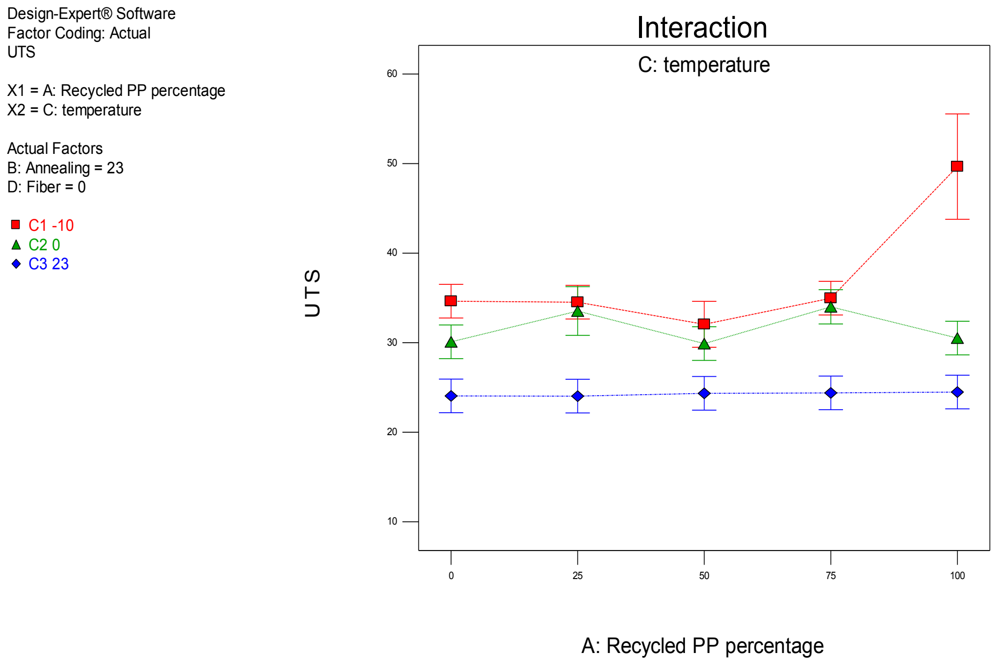
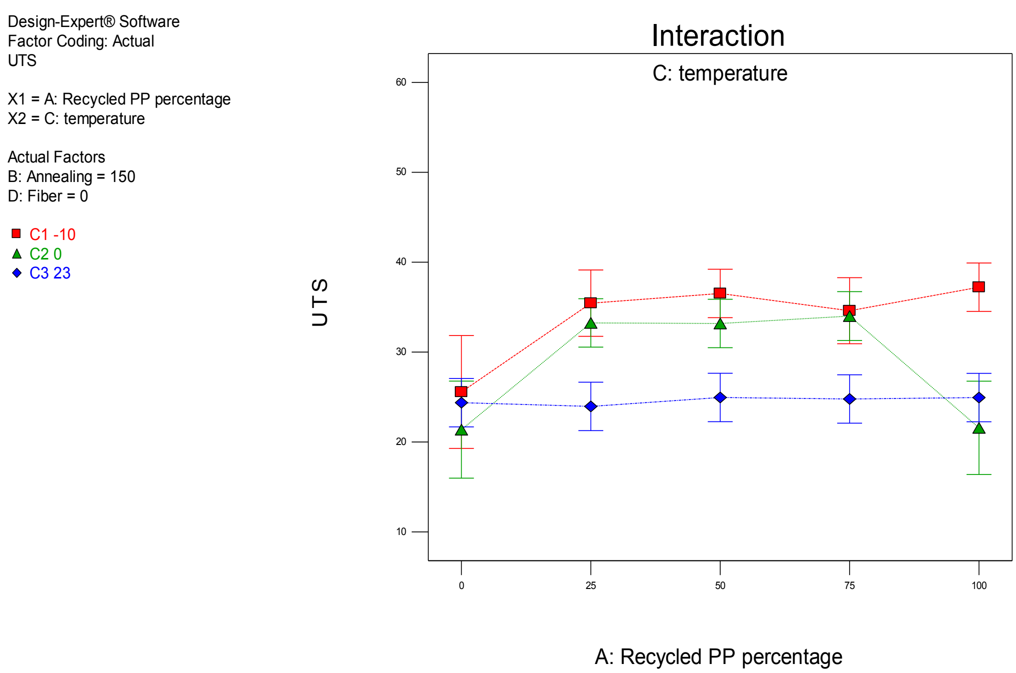


Figure 4-6: The predicted vs. actual graph of the E results.

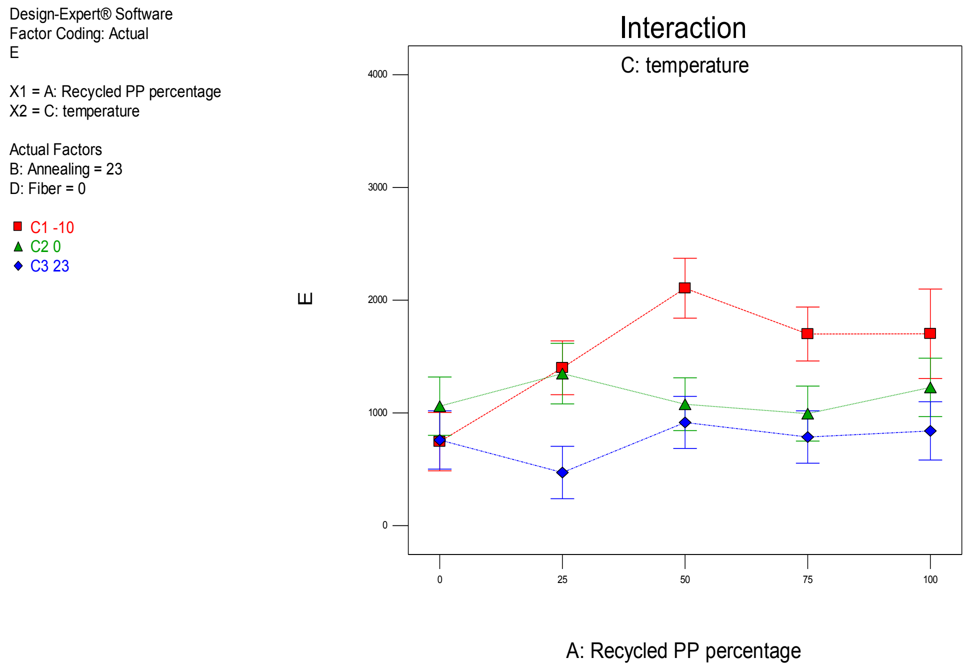


a: not annealed

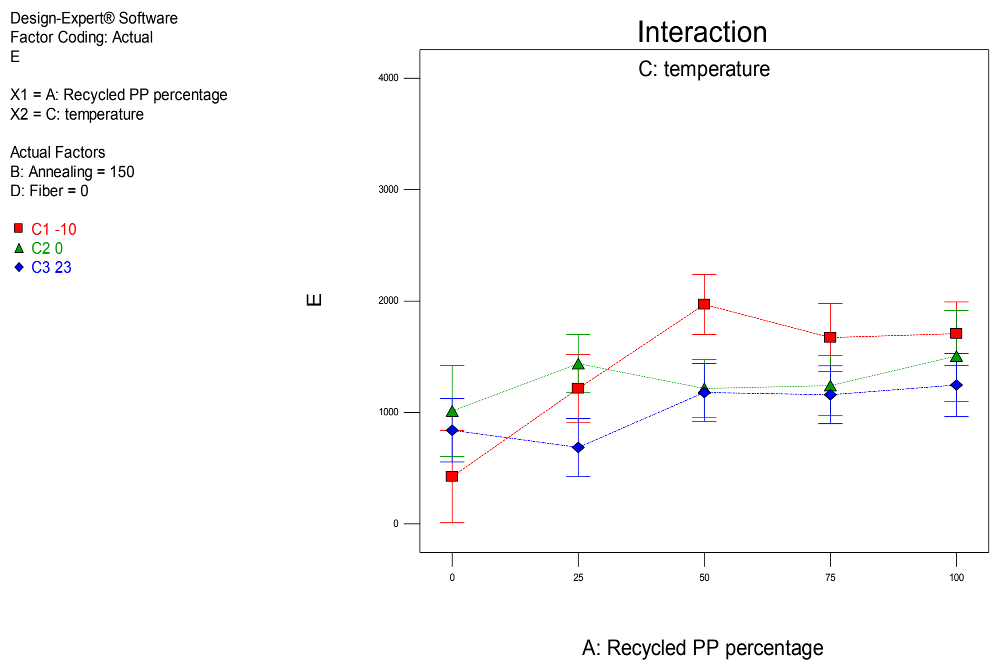


b: annealed at 150°C

Figure 4-7: Effects of temperature on UTS for the non-reinforced PP samples at annealed (A), and not annealed (B) condition



a: not annealed



b: annealed at 150°C

Figure 4-8: Effects of temperature on E for the non-reinforced PP samples at not annealed (a), and annealed (b) condition

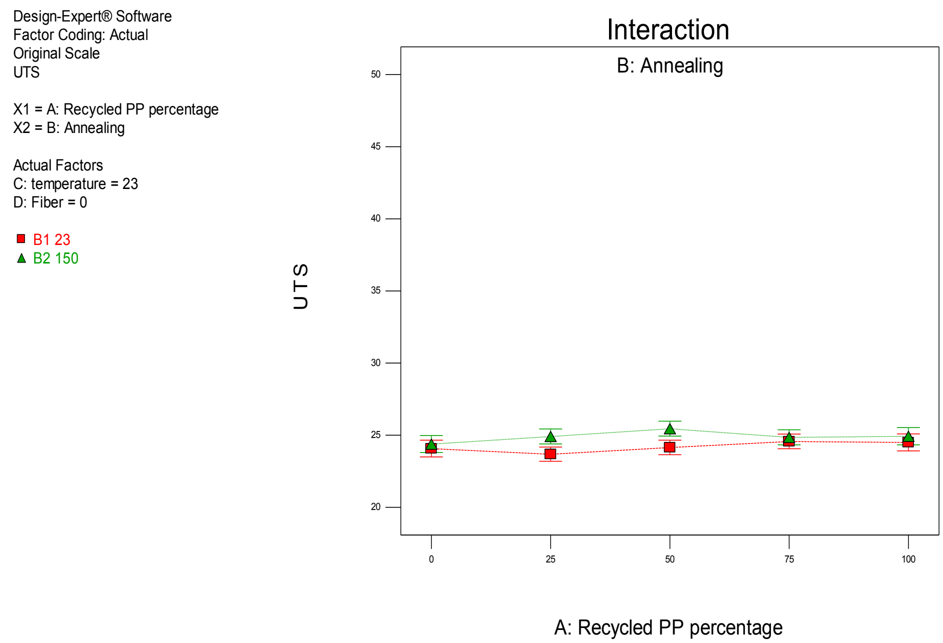


Figure 4-9: Effects of annealing on the UTS of non-reinforced PP samples at room temperature

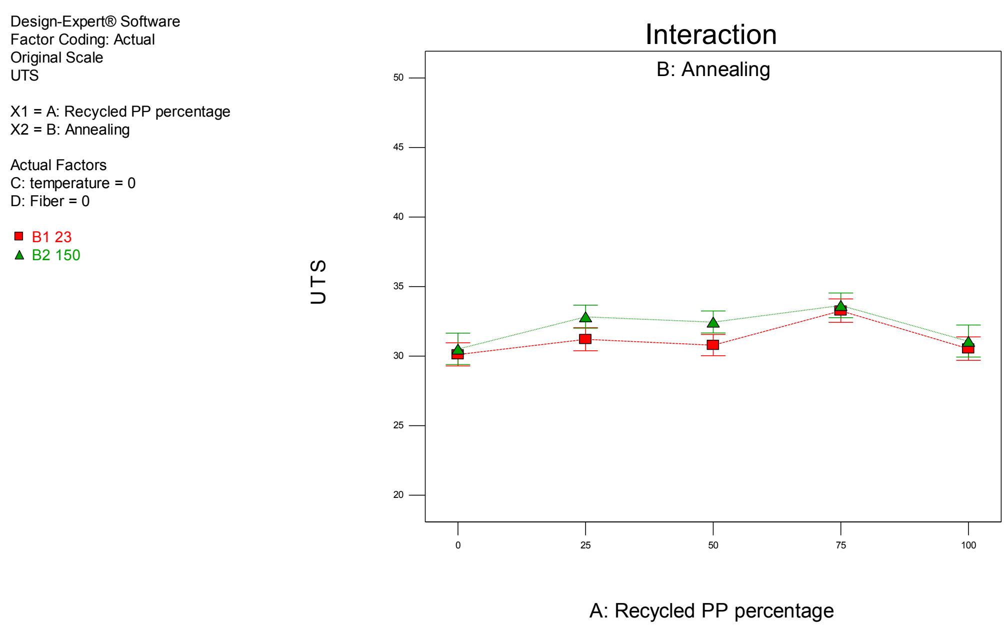


Figure 4-10: Effects of annealing on the UTS of non-reinforced PP samples at 0°C.

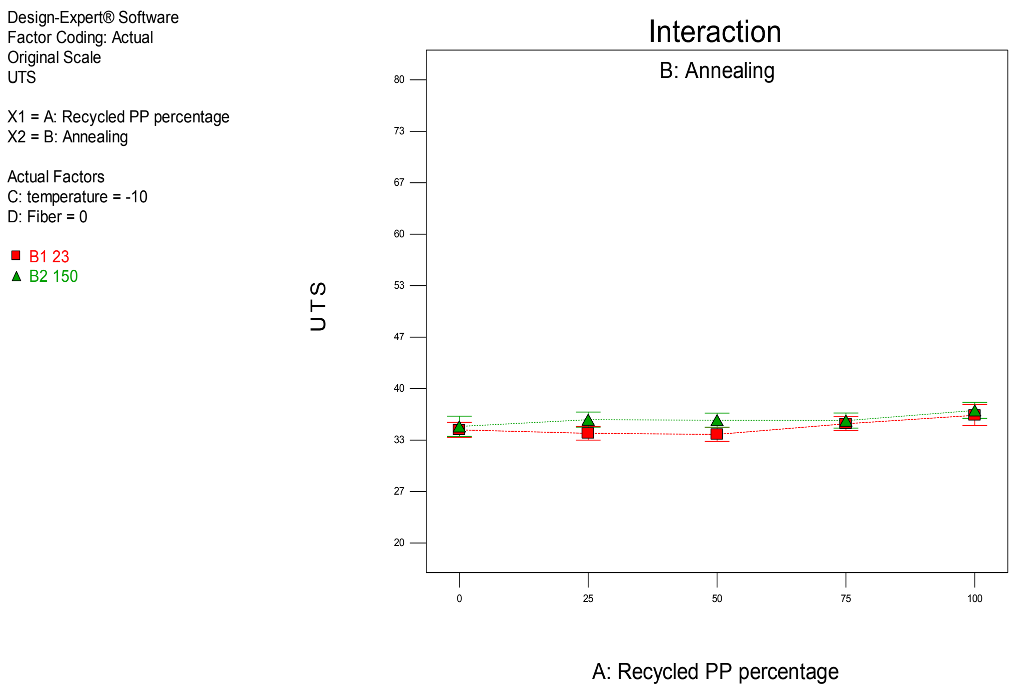


Figure 4-11: Effects of annealing on the UTS of non-reinforced PP samples at -10°C.

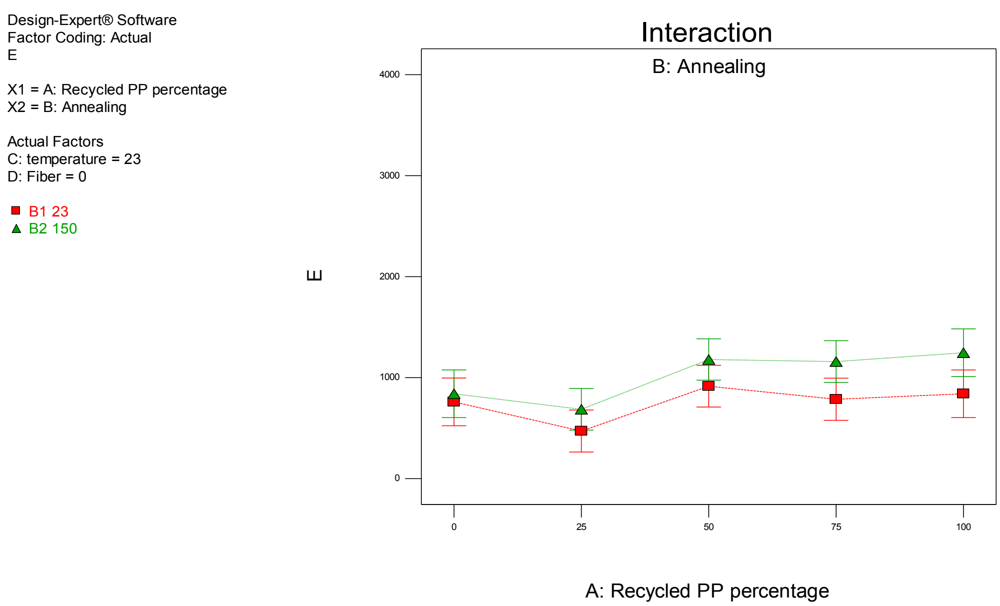


Figure 4-12: Effects of annealing on the E of non-reinforced PP samples at room temperature.

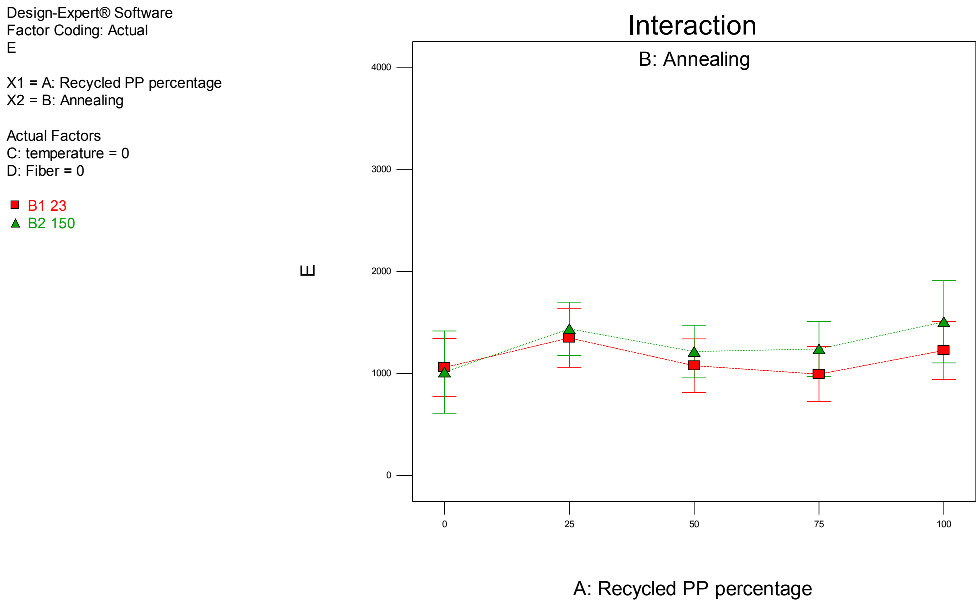


Figure 4-13: Effects of annealing on the UTS of non-reinforced PP samples at 0°C.

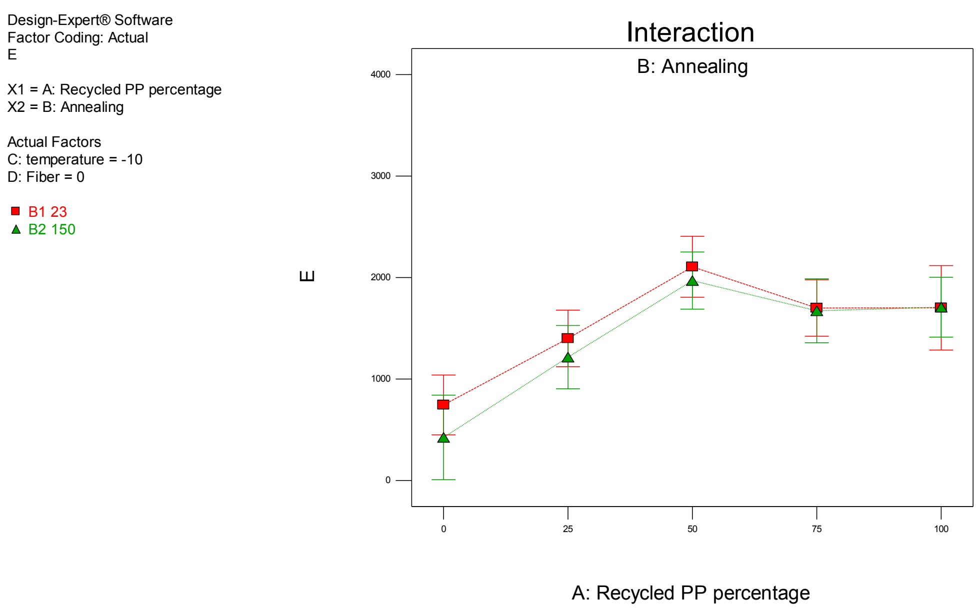


Figure 4-14: Effects of annealing on the UTS of non-reinforced PP samples at -10°C.

To visualize the effects of other factors (Recycled material %, annealing, and temperature) in more detail, the non-GF content PP samples were analyzed individually as well; therefore, means plots were provided for the responses UTS, and E; means plot in Fig. 4.7 and 4.9 show the interaction between the recycled material percentage and temperature for not annealed (a) and annealed (b) samples on UTS (Fig. 4.7) and E (Fig. 4.8). As shown in Fig. 4.7, PP sample materials have higher UTS in colder temperatures; from room temperature to zero °C UTS increased by almost 26% and from zero °C to -10°C, UTS increased by 8%; therefore in total from room temperature to -10°C the UTS increased by about 36%. Almost the same increase is observed for the Young's modulus results as 23%, 7.5% and 29% for room temperature, 0°C, and -10°C, respectively. Figures 4.9 to 4.14 show that there is no interaction between recycled material percentage and annealing, at room temperature, 0°C, and -10°C. Based on Figures 4.9 to 4.11, annealing only has about 1% improvement on the UTS of PP samples at room temperature (Fig. 4.9) and this improvement increase in colder temperature to 10% at 0°C (Fig. 4-10), and 20% at -10°C (Fig. 4-11). However, this trend was backward for the Young modulus results (See Fig. 4.11- 4.14) as annealing made 25%, 3.5%, and -25% changes at room temperature, 0°C, and -10°C.

Glass fiber has a significant effect on the mechanical properties of the tested PP samples; effects of fiber reinforcement on UTS and E are shown in Fig. 4.15 to 4-17 for the room temperature, 0°C, and -10°C; it revealed that usage of fiber improved the tensile properties, however it has more effect on the 0w% recycled PP samples.

Finally, a summery of the results at tested temperatures are presented in table 4.1, 4.2, and 4.3 for the UTS, Yield stress, and E, respectively.

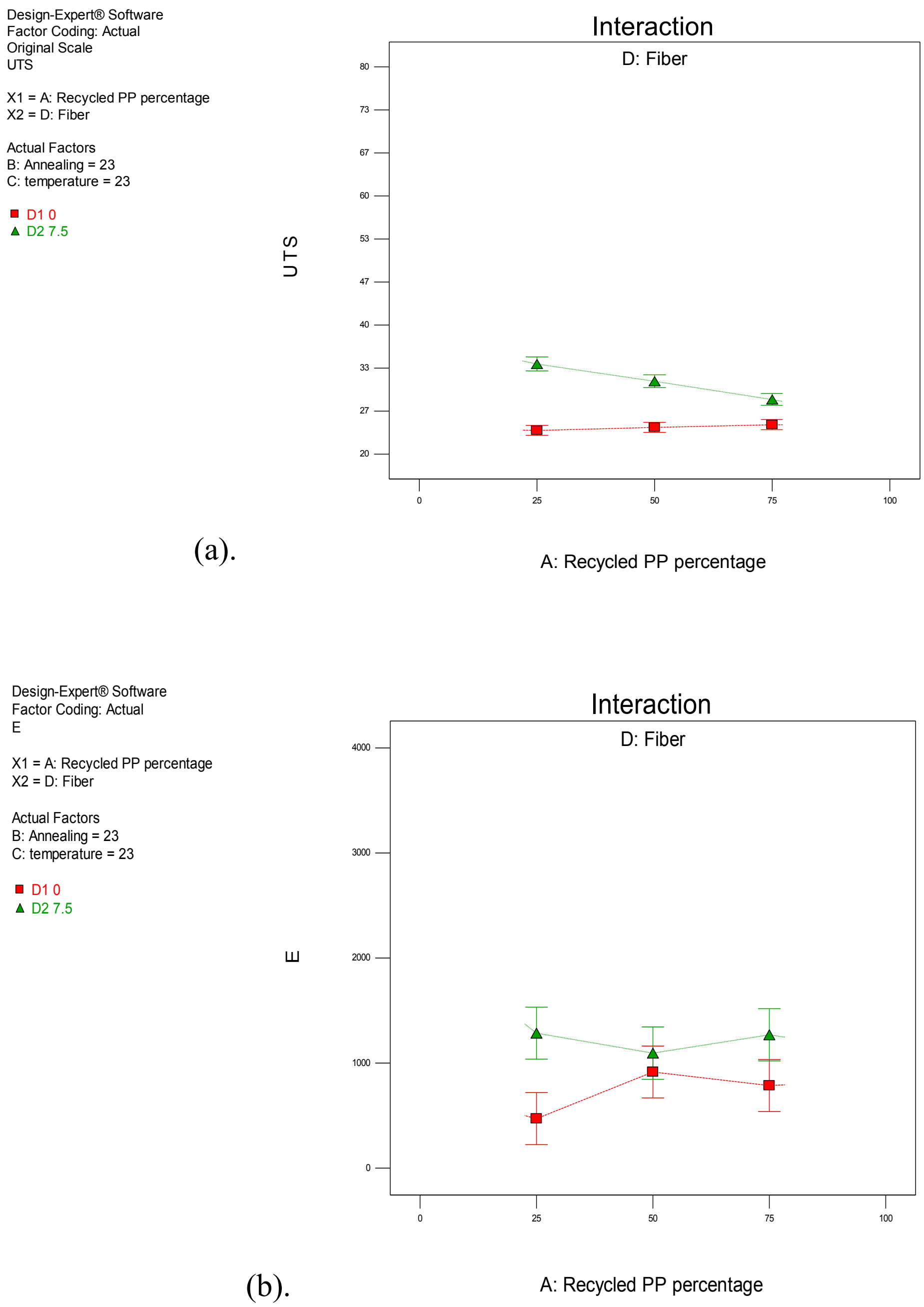
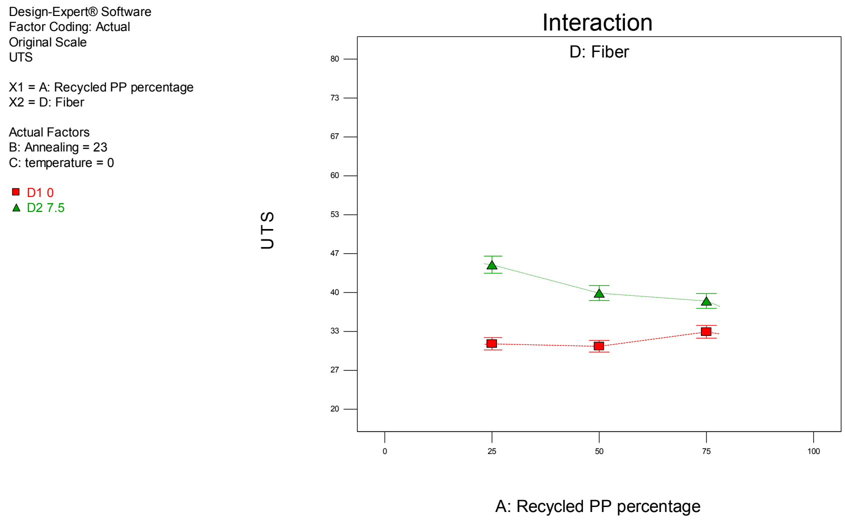
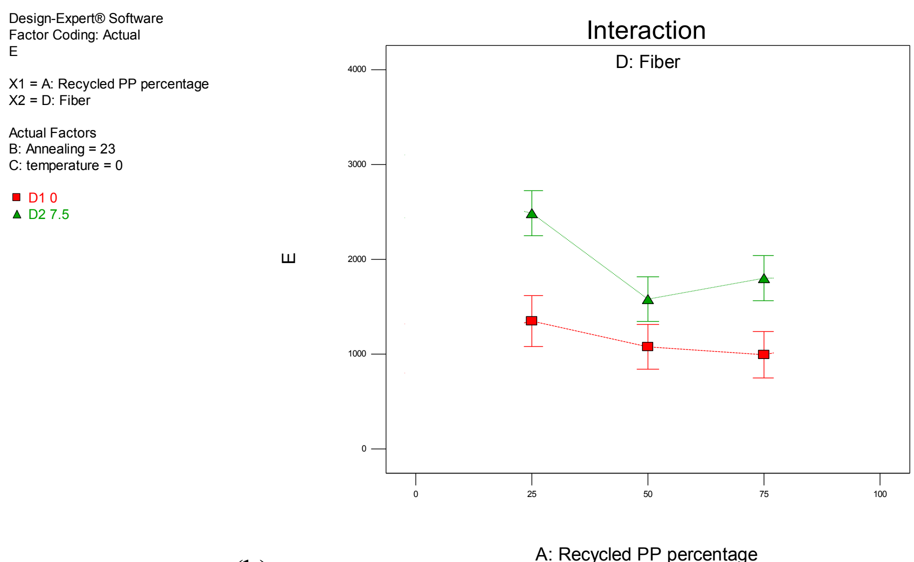


Figure 4-15: Effects of fiber on UTS (a) and E (b) of PP samples at room temperature.

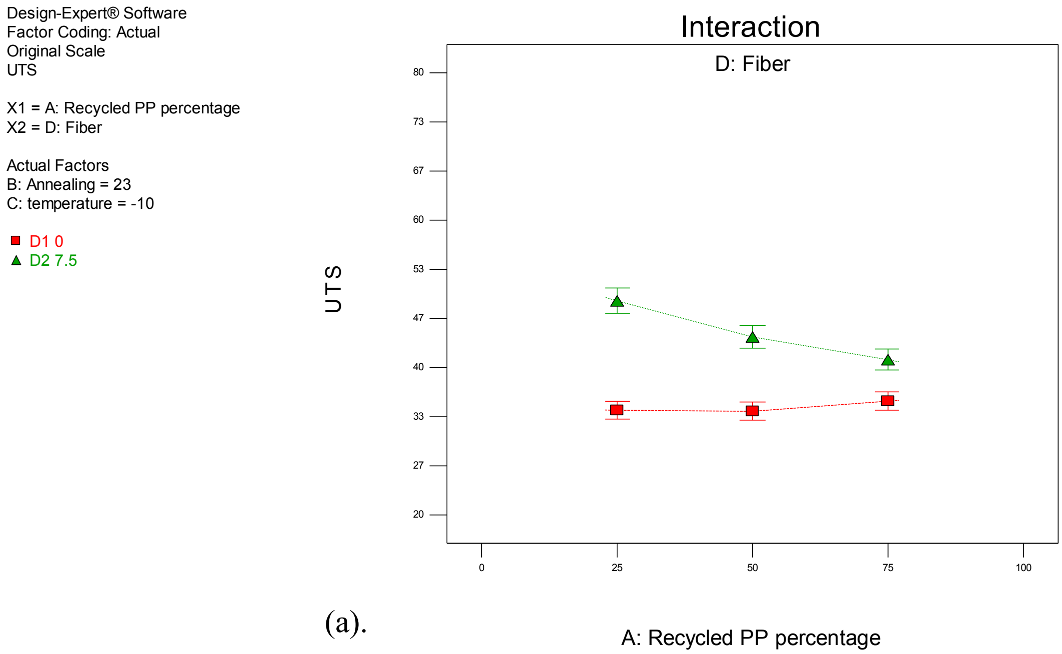


(a).

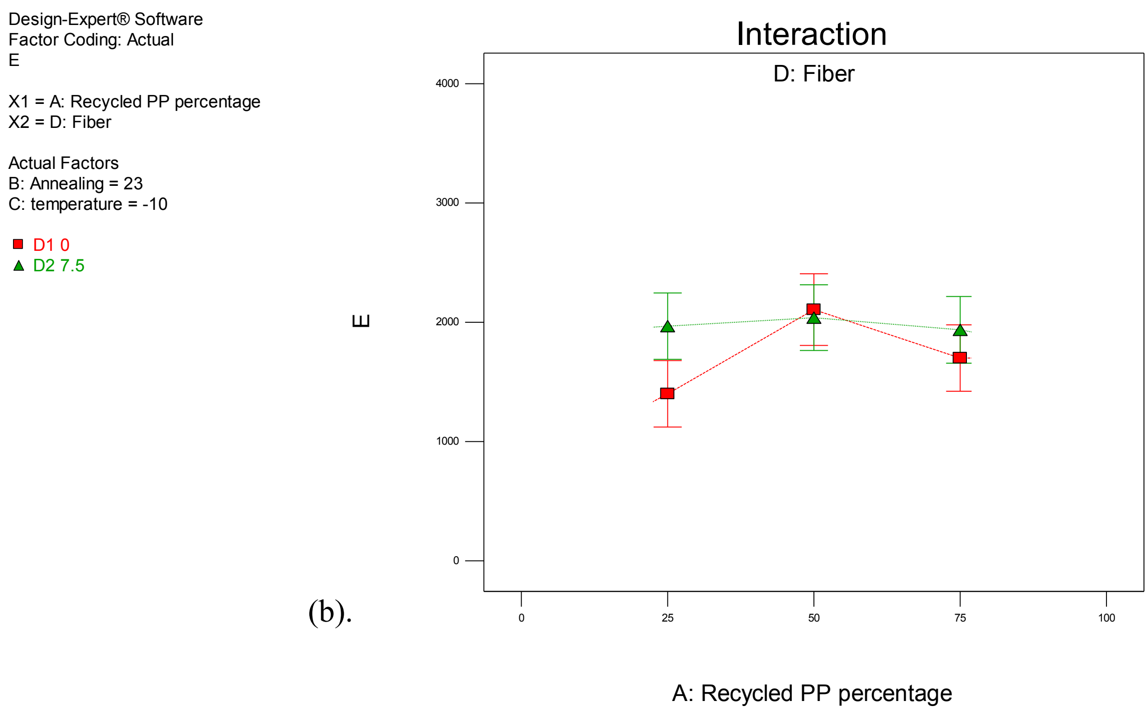


(b).

Figure 4-16: Effects of fiber on UTS (a) and E (b) of PP samples at 0°C.



(a).



(b).

Figure 4-17: Effects of fiber on UTS (a) and E (b) of PP samples at 0°C.

Table 4-1: Summery of the Yield stress (Mpa) results at three tested temperatures (NA: not annealed, A: annealed)

		100% Recycled		75% R				50% R				25% R				0% R	
		No fiber		No fiber		With fiber		No fiber		With fiber		No fiber		With fiber		No fiber	
		NA	A	NA	A	NA	A	NA	A	NA	A	NA	A	NA	A	NA	A
20°C	Ave	22.31	24.96	22.24	24.72	28.64	27.81	22.18	24.49	30.98	31.24	22.13	24.27	33.51	35.08	22.07	24.04
	Std	0.52	0.57	0.51	0.57	0.66	0.64	0.51	0.56	0.72	0.72	0.51	0.56	0.77	0.81	0.51	0.56
0°C	Ave	30.37	31.82	30.28	31.52	37.66	35.28	30.21	31.23	40.74	39.62	30.13	30.93	44.07	44.50	30.04	30.64
	Std	0.70	0.73	0.70	0.73	0.87	0.81	0.70	0.72	0.94	0.92	0.7	0.70	1.02	1.03	0.69	0.71
- 10°C	Ave	34.12	34.74	34.03	34.42	41.68	38.44	33.94	34.09	45.09	43.17	33.85	33.77	48.78	48.48	33.76	33.45
	Std	0.79	0.80	0.78	0.79	0.96	0.89	0.78	0.79	1.04	1.00	0.78	0.78	1.13	1.12	0.78	0.77

Table 4-2: Summery of the UTS (Mpa) results at three tested temperatures (NA: not annealed, A: annealed)

		100% Recycled		75% R				50% R				25% R				0% R	
		No fiber		No fiber		With fiber		No fiber		With fiber		No fiber		With fiber		No fiber	
		NA	A	NA	A	NA	A	NA	A	NA	A	NA	A	NA	A	NA	A
20°C	Ave	24.45	24.80	24.65	24.99	28.47	27.83	24.30	24.62	30.92	31.15	23.94	24.22	33.78	35.31	24.04	24.32
	Std	0.58	0.60	0.59	0.61	0.79	0.76	0.58	0.59	0.93	0.95	0.56	0.57	1.11	1.22	0.56	0.58
0°C	Ave	30.93	33.75	31.31	34.16	37.71	35.22	30.79	33.52	40.72	39.40	30.24	32.84	44.16	44.60	30.44	33.05
	Std	0.94	1.12	0.96	1.14	1.39	1.22	0.93	1.10	1.62	1.50	0.90	1.05	1.91	1.95	0.91	1.07
- 10°C	Ave	35.02	36.95	35.52	37.48	42.04	38.70	34.89	36.73	45.08	43.14	34.20	35.94	48.47	48.59	34.49	36.22
	Std	1.20	1.34	1.24	1.37	1.73	1.47	1.19	1.32	1.99	1.82	1.14	1.26	2.30	2.31	1.16	1.28

Table 4-3: Summery of the E (Mpa) results at three tested temperatures (NA: not annealed, A: annealed)

		100% Recycled		75% R				50% R				25% R				0% R	
		No fiber		No fiber		With fiber		No fiber		With fiber		No fiber		With fiber		No fiber	
		NA	A	NA	A	NA	A	NA	A	NA	A	NA	A	NA	A	NA	A
20°C	Ave	838.64	1253.02	774.47	1010.30	1334.40	1967.33	776.12	1012.90	1093.42	2093.91	798.83	831.94	1059.56	1466.40	760.22	840.88
	Std	1.58	9.12	1.12	3.57	11.96	61.54	1.12	3.61	5.05	79.29	1.30	1.53	4.40	17.94	1.03	1.60
0°C	Ave	1239.39	2647.59	1130.66	1140.95	1684.00	1926.57	1163.76	1227.21	1711.23	1949.40	1221.81	1323.21	2361.62	2682.12	1064.50	1253.14
	Std	8.69	197.60	5.84	6.07	32.26	56.48	6.62	8.30	34.50	59.28	8.17	11.54	127.70	207.37	4.50	11.94
- 10°C	Ave	4006.90	1721.25	1744.80	1236.06	1730.71	2337.49	1314.96	2410.88	1991.29	1807.42	1025.26	1152.63	2697.60	2052.61	744.55	820.32
	Std	597.15	35.36	37.44	8.59	36.19	122.70	11.23	138.44	64.66	43.37	3.81	15.48	211.84	73.15	0.94	15.45

X- ray diffraction was used to determine the crystalline structure of the different specimens. A sample X-ray diffraction result is shown in Fig. 4.19; the first three strong peaks observed at 2θ= 14.6°, 2θ= 17.5°, and 2θ= 18.9°; and the next three weaker peaks seen at 2θ= 22.1°, 2θ=26.1°, and 2θ= 29.2°. It is observed that changes in recycled material percentages, addition of glass fiber, and annealing will not have an effect on position of the peaks; however the area of the amorphous background would change. Fig. 4.20 and Table 4.4 represent the results of crystalline fraction of the 16 different groups of tested samples; it observed that crystalline fraction has a non-linear behavior by increasing the usage of recycled material and overall the 50w% recycled material has the highest crystalline fraction between all the tested categories of annealed and not- annealed of non-reinforced or reinforced samples. It revealed that annealing improves the crystalline fraction; however it has a more significant effect on reinforced samples (~36%) compared to the non-reinforced samples (~15%). The WXRD results confirm the tensile test results; as more usage of recycled material slightly increases the tensile properties due to higher crystallinity of recycled PP; also it approves the effects of annealing on reinforced and non-reinforced polypropylene samples.

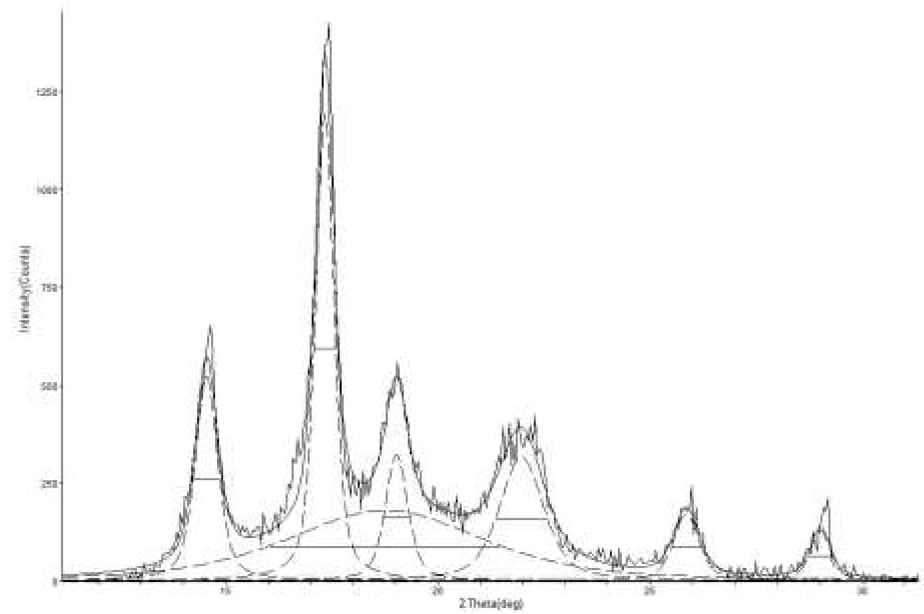


Figure 4-18: The X-ray diffraction of PP

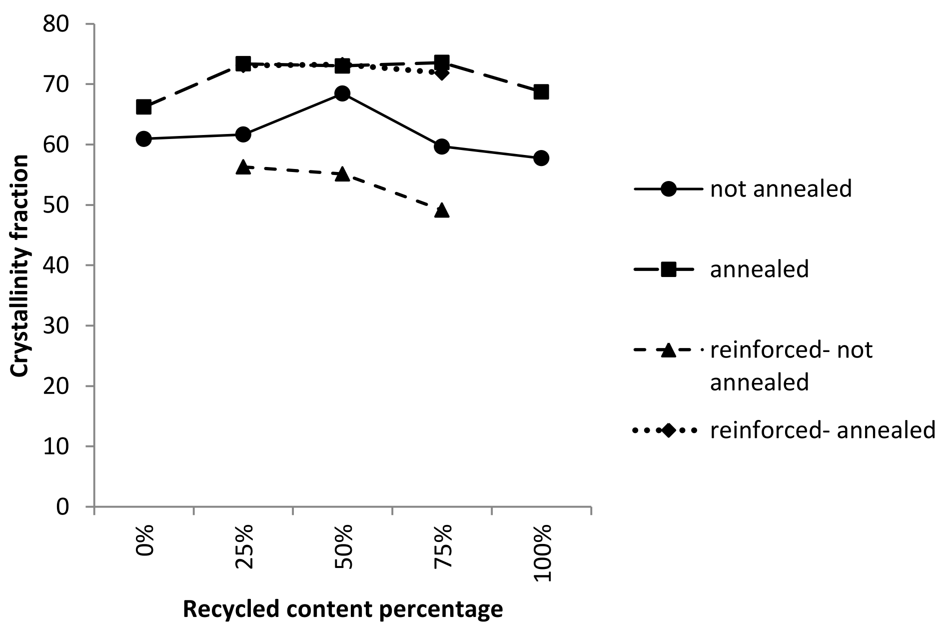


Figure 4-19: Crystallinity fraction of the tested samples

Table 4-4: Crystallinity fraction results (%)

	No fiber		With fiber	
	Not annealed	Annealed	Not annealed	Annealed
0% Recycle	60.93	66.2		
25% Recycle	61.66	73.36	56.3	73.1
50% Recycle	68.43	73.03	55.14	73.23
75% Recycle	59.67	73.58	49.13	71.86
100% Recycle	57.72	68.72		

4.4 Discussion

Others have investigated on the effects of multiple processing on microstructure and mechanical properties of polypropylene [18-12]; They reported that 1-4 times of recycling has no significant effect on mechanical properties of polypropylene, however by increasing the number of recycling above 4 times improvement in material properties could be expected due to increase in the material crystallinity [8, 11, 12], and also it could be due to the reduction of molecular weight by reprocessing [26]. In this study the one time reprocessed polypropylene were used, hence no significant improvement in the tensile properties were observed and also the changes in the crystallinity of the samples investigated by WAXD technique; hence the literature also confirm the obtained results.

Based on research analyzing the effects of annealing on crystalline structure of polypropylene, it reported that annealing improves the crystalline structure [13-15,20] and in this study it also observed that annealing enhanced the crystalline structure of PP samples using by WAXD method. In those studies improvement in the material tensile behaviour by annealing was reported [13-15,20]; however, in this study the effects of annealing was not significant. It should be noted that our materials were annealed only at 150˚C and potentially a change in material properties may be observed at higher temperatures. Current work is ongoing looking at the effects of annealing at 140 ˚C, and 160 ˚C.

The main reason to add fibers in polymers is because of their high stiffness and high strength; therefore, fibers by having high stiffness and strength will designate strong interatomic and intermolecular bonds in the composite [27]. The effects of fiber reinforcement on material properties of polypropylene showed significant improvement in the tensile behaviour of our tested samples. Fiber properties are dependent on the fiber microstructure, and the fiber microstructure is strongly related to the processing factors [28]. Fiber properties are dependent to the type, orientation and the crystallinity percentage; annealing as a heat treatment process can rearrange the molecular chains which can affect the crystallinity and the orientation of fibers which could greatly affect the composite properties [29]. From the results of annealing on reinforced samples it observed that annealing has more effect on reinforced materials.

Other studies on the effects of temperature on mechanical properties of polypropylene concluded that by increasing the temperature (between 20°C and 120°C) tensile properties will decrease in approximately linear trend [2, 6, 30]; also, another study on the mechanical properties of polypropylene composite tapes (at -40°C to 140°C), indicate that polypropylene composites also obey the same trend at cold temperatures [24]. From our investigations, we also observed the same trend for the cold temperature tensile behaviour of non- reinforced and reinforced PP samples; with decreasing testing temperature, UTS and E for the PP samples increased which is related to the molecular mobility decrease within the crystalline phase [24] and also the increase of shear strength of crystals at colder temperatures [31].

4.5 Conclusion

Tensile behavior of polypropylene is investigated at cold temperatures by also considering other different factors which could have an effect on mechanical properties. Therefore, 5 different recycled PP content (0% R, 25% R, 50% R, 75% R, and 100% R) samples were tested at three different temperatures (room temperature (~20°C), 0°C, and -10°C) at annealed (at 150°C) and not-annealed condition; also the effects of adding fiber on mechanical behaviour of recycled content PP samples was investigated by adding 7.5% GF to the samples. A full factorial design of experiment followed with three replicates resulting in 168 runs. Hence, based on DOE analysis using "Design Expert 9" software, ANOVA analysis, Pareto charts, and means plots provided to express the results. From the Pareto charts, based on ANOVA analysis, it concluded that temperature and fiber have the most significant effect on tensile properties of the PP samples; and also their interaction was significant for the modulus of elasticity. Additionally, greater usage of recycled material will not have significant changes (less than +5%) on mechanical properties of the PP samples. Finally, investigations on annealing revealed that it has a slight linear improvement on UTS results by decreasing the temperature and vice versa for the E results, however as the effect is not significant and it is a timely and costly process it will not be recommended. Fiber after temperature has the most significant effect on tensile behaviour of the PP samples and the highest responses obtained for the 25% R PP samples with the 7.5% GF content at -10°C with the UTS of 49± 0.2 and the E of 2560± 300 MPa. WAXD results show that the crystalline diffraction improves slightly with greater usage of recycled material.

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Chapter 5 : Conclusions and future works

5.1 Conclusions

The main objective of this research was to investigate on the effects of recycling polypropylene. PP is one of the most commonly used polymers, as an environmental friendly green action. Hence different percentage of recycled material blended with virgin material and tested using tensile test and WAXD methods. Therefore it is observed that based on WAXD results, usage of recycled material slightly increased the crystallinity fraction which also resulted in slight improvement in the tensile behavior of tested PP samples. Also it should be noted that in room temperature the tensile behavior of different recycled percentage content samples has a negligible difference which could be concluded that for the room temperature applications, more usage of recycled material is recommended without damaging effect on the material properties.

Also the effects of annealing were examined in this research; based on room temperaturetensile tests and WAXD results, it observed that annealing improves the tensile properties due to improvement of crystalline structure. Also, it revealed that annealing has more significant effect on reinforced PP samples in compare with the non-reinforced PP samples and also the improvement of tensile properties is more noticeable at colder temperatures.

Another objective of this study was to investigate the effects of cold temperature on tensile properties of the Polypropylene sample. It is observed that in colder temperature polypropylene samples are more rigid; therefore they have higher ultimate tensile stress (UTS) and elastic modulus.

Additionally, statistical analysis was carried out using ANOVA method to find the factors which have significant effects on tensile properties of tested polypropylene samples and it revealed that temperature and addition of glass fiber were the only factors with a significant effect.

Also, a case study simulating a Canada post container at room temperature was presented; as using fiber and annealing was not in the interest of the case provider company (Drader manufacturing), only the non-reinforced material properties were used. Drop test simulation results showed that 50w% recycled material has the least maximum impact stress as a result of drop impact.

The main contribution of this work is that it is the first to simultaneously look at 4 different parameters (specifically, the percentage of recycled material, temperature, annealing, and fiber reinforcement) and their effect on tensile properties of polypropylene. Also, the concept of blending virgin material with the same recycled material is a new study; and showed that it has no detrimental effect on the material properties; which could be very useful for industries. No scientific study has been previously performed on crystalline structure and tensile properties of these combinations of materials.

5.2 Limitation

Although this research has reached its objectives, there were some unavoidable limitations and shortcomings which will be suggested as a future work in coming section.

First of all because of time limit, this research main focus was on recycled material percentage and cold temperature; however other studied factors (annealing and fiber) are also very important factors which could be studied more in detail and with more levels.

Second, the recycled material which used in this study was prepared by Drader manufacturing (Edmonton- Alberta) from one-time reprocessing of polypropylene, and there are some literatures on multiple reprocessing which shows more improvement in the material properties; hence studies on multiple reprocessing could be useful.

5.3 Suggestions for future work

The focus of this study was on different content percentage of recycled polypropylene and its effects on tensile properties. Additionally the effects of cold temperature, reinforcement and annealing were studied. One-time reprocessed material used to make the recycled content specimens; study on multiple reprocessed materials could be an interesting topic for the future work. Annealing was only studied at one temperature (150˚C); however the melt temperature is 190˚C, so future work could investigate annealing at high temperature. Similarly, reinforcement was only observer at one percentage, therefore future studies on different percentages or different fiber materials. This would improve our understanding of the effects of annealing and reinforcement on tensile behavior of polypropylene. Therefore different annealing temperatures could be tested to find the most ideal annealing temperature to improve the tensile properties; also different glass fiber addition percentages could be tested to see the effects of fiber on polypropylene crystalline structure and tensile properties. Because the effect of fiber was significant future work should focus in that area prior to investigating annealing. Different scanning calorimetry could also be used to investigate the melt temperature of the materials. Also FEA at colder temperature could be simulated as another future work. Another area that could be investigated in the future is the effect of aging and UV exposure on the mechanical properties of PP.

Regardless of all the future opportunities available for research, this work was the first to look at the percentage of recycled material, testing at cold temperature and simultaneously looking at annealing and fiber reinforcement.