INCORPORATION OF WASTE POLYETHYLENE TEREPHTHALATE (PET) INTO CONCRETE USING STATISTICAL MIXTURE DESIGN

by

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ABSTRACT

This thesis investigated the incorporation of waste Polyethylene Terephthalate (PET) into concrete as a replacement for natural fine aggregate and found an optimal combination of components that produces a useful concrete product. Six components were considered: cement, water, coarse aggregate, fine aggregate, superplasticizer, and waste PET. A total of 31 mixes were prepared based on a statistical mixture design approach. The responses of these mixtures were workability, compressive strength, and splitting tensile strength.

The waste PET was first reduced in volume by shredding and then combined with the rest of the components. The responses from the experiments were statistically analyzed and a model fitted to each response. Linear models were found to fit the responses best. Using the desirability function approach, four optimal options were selected and then verified in the lab by comparing the experimental with the predicted values. Except for one, all the values fell within the 95% prediction interval. The option that best fulfilled the workability and strength requirements was found. The average response values obtained with this combination were: (1) compressive strength of 23.8 MPa; (2) slump 123 mm, and (3) splitting tensile strength of 3.33 MPa. This mix can be used in basements foundation walls or slabs, inside buildings not exposed to freezing temperatures. It is recommended that future work should consider method of mixing, time of mixing, volume of mix, curing conditions, and other responses to further understand the characteristics of incorporating waste PET into concrete.

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List of Abbreviations and Symbols

Agg/cem	Aggregate/Cement ratio
ANOVA	Analysis of Variance
ASTM	American Society for Testing Materials
D	Desirability
H_0	Null Hypothesis
H_a	Alternative Hypothesis
HRWRA	High Range Water Reducer Admixture
NRMCA	National Ready Mixed Concrete Association (USA)
OFAT	One Factor at a Time
PET	Polyethylene Terephthalate
PRESS	Prediction Error Sum of Square
Sp	Superplasticizer
WPET	Waste PET
w/c	Water/cement ratio

CHAPTER 1

INTRODUCTION

1.1 Plastic waste problem

The plastic industry is one of the largest industries worldwide. Globally, in 2013, over 299 million tons of plastic were produced. Plastic has replaced paper, cardboard, metal and glass (Andrady, 2015). This displacement is a result of several advantages that plastic has over these other materials. Plastic is low-cost, lightweight and easy to handle, it also has relatively high strength and corrosion resistance (Andradi, 2015; Ferreira et al., 2012). Because plastic products have a large presence in a variety of markets (e.g. packaging, automotive, healthcare), these markets are directly contributing to increase the volumes of plastic in the waste stream (Silva, de Brito, and Saikia, 2013).

Plastic consumption has increased dramatically worldwide. This is in contrast to the recycling rate, which has remained low (Gu and Ozbakkaloglu, 2016). In USA, the contribution of plastic to the waste stream has increased from an average of 0.39 million tons in the 1960s to 31.75 million tons in 2012. Over the span of 50 years, the recycling rate has increased only 8.8%, which makes plastic waste volume a serious issue for solid waste management (Gu and Ozbakkaloglu, 2016).

Every year in the Canadian province of Newfoundland and Labrador, approximately 4-thousand tons of plastic are consumed, which is approximately 8% of the solid waste generated in the province. This plastic waste is collected, compacted and sent to other provinces. Because of the option of sending plastic waste to other provinces, Newfoundland has not yet developed any long-term strategy for the management of this solid waste. This has drastic economic and environmental impacts (Government of Newfoundland, 2002).

1.1.1 Waste Polyethylene Terephthalate (PET) in the waste stream

Polyethylene Terephthalate (PET) is one of the main fractions of the plastic waste stream (Silva et al., 2013). PET is mostly employed as a multi-purpose plastic for bottled water, soft drinks, and as single-use packaging material (Andrady, 2015). Products made of PET are generally large in volume and can take approximately one thousand years to decompose under natural environmental conditions (Silva, et al., 2013; de Brito and Saikia, 2013).

Once being collected, the most common treatment options for waste PET are: incineration, landfilling, and recycling (Gu and Ozbakkaloglu, 2016). Incineration takes advantage of the calorific properties of the polymers, which can then be used as fuel. However, there are concerns related to the generation of gases and fly ash that would result in air pollution (Andrady, 2015). Landfilling is considered as the least desirable treatment option. Because it requires of large amount of land and quantities of waste PET, and may lead to potential environmental issues due to leachate generation and gas emission (de Brito and Saikia, 2013). Recycling, on the other hand is currently considered as the best solution for addressing the problem of waste PET. (Gu and Ozbakkaloglu, 2016; Ge, Huang, Sun, and Gao, 2014). However, the recycling and reuse of waste PET have not being correctly performed. In 2012, in USA, 4.5 million tons of PET waste was available for recycling but only 880 thousand tons were recycled. This means that 80.1% of the available PET was discarded (Gu and Ozbakkaloglu, 2016).

1.1.2 Options for waste PET recycling

Recycling waste PET is a challenge that needs to be addressed. Waste PET management and recycling has become an increasing concern worldwide. Research on innovative approaches to recycle waste PET is currently being done (Andrady, 2015). Incorporating waste PET into the concrete industry has been regarded as one of these innovative approaches (Gu and Ozbakkaloglu, 2016; Ge et at., 2014; de Brito and Saikia, 2013). In the concrete industry waste PET can be used for 1) the production of polymeric concrete, 2) as concrete reinforcement, and 3) as concrete aggregate (Gu and Ozbakkaloglu, 2016; Ge et at., 2014; de Brito and Saikia, 2013).

The production of polymeric concrete involves chemical depolymerisation of waste PET bottles into unsaturated polyester resin (Ge et al., 2014). This process results in a quality polymer concrete, with high resistance to compression and flexion. However, in order to be economically feasible, PET consumption should be large and steady in a given area (Gu and Ozbakkaloglu, 2016; Ge et at., 2014). The incorporation of waste PET as fiber concrete-reinforcement consists of first shredding the waste PET into flakes and then melting the flakes into monofilaments (Gu and Ozbakkaloglu, 2016). This end product efficiently controls shrinkage. Unfortunately since a thermal process is involved, this is a very expensive approach that is generally not affordable (Gu and Ozbakkaloglu, 2016; Siddique, Kathib, and Kaur, 2008)

Finally, the incorporation of waste PET as an aggregate replacement into concrete only requires shredding the waste PET into small particles and incorporate the shredded particles into the aggregate mixture (sand, gravel or crushed stone) (Gu and Ozbakkaloglu, 2016; de Brito and Saikia, 2013). Using waste PET as an aggregate replacement has two important benefits: the waste PET products that occupy an enormous volume in the waste stream can be dramatically reduced by shredding and disposed of; and natural aggregate can be partially replaced, reducing the impact to natural resource availability. Thus, an important reduction in environmental impact of waste disposal while saving natural resources and energy consumption can be achieved (Gu and Ozbakkaloglu, 2016; Frigione, 2010).

1.2 Shortage in natural aggregates and concrete sustainability

In 2004, 14 tons of aggregates were consumed per person in Central Canada (Aïtcin and Mindess, 2011). In 2009, 10 to 11 billion tons of natural aggregates (sand, stone, gravel) were used worldwide for building and construction purposes (de Brito and Saikia, 2013). With the increasing demand on construction and infrastructure, natural aggregate resources are becoming scarce (de Brito and Saikia, 2013). Considering the future scarcity of natural aggregates, unconventional aggregates need to be investigated and tested for future use (de Brito and Saikia, 2013; Alexander and Mindness, 2011).

Sustainability is the consumption of natural resources without compromising the availability of these resources for the future generations (Andrady, 2015). According to Mehta and Monteiro (2014) there are strong trends that govern the current state of the world. These trends include: massive population growth, technology advances, rapid urbanization, and increasing environmental awareness. Mehta and Monteiro (2014) state that the influence of those trends strongly compromises the survival of future generations. Improving sustainable practices requires research on strategies and alternatives that diminish the environmental impact caused by humans (Andrady, 2015). Changes in our consumption behavior and adoption of sustainable policies and practices are necessary (Andrady, 2015).

Globally, concrete is one of the most utilized materials for building purposes (Mehta and Monteiro, 2014). Additionally, one the most demanded natural materials are aggregates for the construction industry (Andrady, 2015). A large amount of new buildings and building rehabilitations are expected to take place in the upcoming years. Concrete is versatile and can be combined with different materials (Mehta and Monteiro, 2014). Sustainability in the concrete industry can be achieved by the use of waste materials from other industries into concrete (de Brito and Saikia, 2013). Additionally, as stated by Mehta and Monteiro (2014), Portland cement is able to safely incorporate materials from other industries as raw material.

1.3 Design of experiments-mixture design approach

Some experimenters use the one-factor-at-time approach (OFAT) of experimentation to interpret the outcome data. In this method, the factors or variables are varied one at time, when one factor is changed the other factors are held constant (Myers and Montgomery, 2009). Thus, the responses are analyzed each time that a single variable is modified independently. This approach has long been outdated, inefficient, and does not consider interactions or influences among factors that can affect the outcome of the experiment (Myers and Montgomery, 2009).

In proper design of experiments methodology, the factors are modified together to evaluate interactions and examine their influence in the measured response (Cornell, 2002). Additionally, the experiments are evaluated in order to obtain an objective conclusion from fewer experiments (Lye, 2002). A special approach called mixture design is employed for concrete mixtures. In this design, the responses (desired properties) depend directly on the proportions of the components in the mixture (Cornell, 2002). Thus, the desired properties can be achieved by optimizing the combination of the components (Myers and Montgomery, 2009). However, no studies were found regarding the incorporation of waste PET into concrete using statistical mixture design.

1.4 Thesis scope

This thesis aims to incorporate waste PET into concrete as natural aggregate replacement and find an optimal combination of components that generates a useful concrete product using statistical mixture design. Six components were included in the mixture: cement, water, coarse aggregate, fine aggregate, waste PET and superplasticizer. The usability of the concrete was determined through the evaluation of workability, compressive strength, and splitting tensile strength. The waste PET was first reduced in volume by shredding and then combined with the rest of the components. The shredding process and the incorporation of waste PET into the concrete mixture were maintained as simple and cost-effective as possible. Additionally, the cement fraction of the mixture was kept as low as possible considering the important environmental impact of cement production. A total of 31 mixtures were prepared and tested. Workability was tested with fresh concrete, and compressive strength and splitting tensile strength were tested according to the standards at 28 days. Based on the test results and the statistical analysis, the prediction models were selected for each property and the combination of components that met the required properties was optimized. It entails the following tasks in the thesis: (1) To determine the best alternative of incorporation of waste PET into concrete as a natural aggregate replacement. Based on past research, decide important factors on waste PET incorporation such as: size, shape, gradation, and percentage of replacement; and (2) To use statistical mixture design for achieving an optimal combination of components that generates a useful concrete product, through:

- Defining the minimal workability and compressive strength required for a practical use of concrete.

- Identifying the appropriate range of variability for each component according to previous research.
- Determining influential factors of the properties such as water/cement ratio, aggregate/cement ratio, and curing conditions
- Testing the concrete specimens for workability, compressive strength and splitting tensile strength according to the standards.
- Analysing the responses and determine by regression analysis the prediction model that best fits for each property.
- Determining through multi-objective optimization the combination of components that optimizes the various desired concrete properties.
- Verifying through additional laboratory tests that the best combination of components obtained by multi-objective optimization are indeed accurate and robust.

1.5. Outline of Thesis

The thesis has 6 chapters, a list of references, and 3 Appendices. Chapter 1 introduces the research problem, provides a brief description of the approach taken, the scope, and objectives of the thesis. Chapter 2 reviews and discusses the previous work on the field of the research. It includes the requirements to evaluate the applicability of the present research, as well as the main factors that influence the responses. Chapter 3 describes in detail the procedures and methodology used in the research; and defines the components of the mixture and their ranges. It also discusses the statistical analysis and the selection of a predictive model for each response. Chapter 4 presents the outcome of the research and the findings. It also presents the graphical interpretation of the results Chapter 5 presents the numerical optimization of the components and presents the experimental validation of the optimized mixtures. Finally, Chapter 6 discusses the conclusions of the study and provides recommendations for future work. Appendix A presents the physical and chemical properties of the Portland cement used, Appendix B presents in a table form the literature reviewed, and Appendix presents all the data collected from the experiments.

CHAPTER 2

LITERATURE REVIEW

This chapter reviews the pertinent available information on waste PET recycling, waste PET incorporation into concrete, and the mixture design approach. In the first section of this chapter, the current methods for waste PET recycling are reviewed and the most economical and appropriate method for this study is given special attention. In the following section, past studies on the incorporation of waste PET into concrete are reviewed. Specific aspects that influence the outcome of the mixes, the ranges of proportions of the components, and the measured responses are discussed. Finally, the aspects related with mixture design approach and the utility in concrete mixtures is discussed.

2.1. Recycling options of waste PET

There are two main treatment options for waste PET, especially for waste PET bottles: close loop and open loop. In the close loop treatment, the recycled waste PET bottles are used in combination with virgin material to manufacture new PET bottles. In the open loop process, the waste PET bottles become raw material for other products (Andrady, 2015).

The close loop treatment requires exhaustive waste PET cleaning through the use of chemicals and technology that assure the absence of contaminants and impurities. The process consists of a thermal treatment at temperatures ranging 180 to 230 °C, inert gas stripping and re-extrusion at temperatures from 280 °C to 290° (Welle, 2011). The following stage can be executed in two different ways: 1) through a hydrolytic depolymerisation at a temperature of 150 °C prior to washing, degassing and re-extrusion of the material, or 2) through glycosylation with ethylene glycol that produces a partial depolymerisation of PET into oligomers that can be a replacement for the prepolymer (Welle, 2008; Andrady, 2015).

In the open loop treatment, waste PET undergoes different processes depending on the purpose of use (Andrady, 2015). Generally, waste PET is prewashed, sorted, granulated and pelletized. Sorting is typically done manually, but there are automatic sorters available in the market. For shredding, the common rate is at 3 tons/hour, there are available shredders in the market depending on the required particle size. Additional equipment can be used for pulverizing particles as fine as 300 μm. This equipment substantially increases the costs. The plastic is then, flaked in water, floated to separate remaining impurities, and pelletized (Andrady, 2015). Figure 2.1 shows in the upper section the standard production of PET bottles. In the lower section, the current treatments for waste PET bottles are depicted.

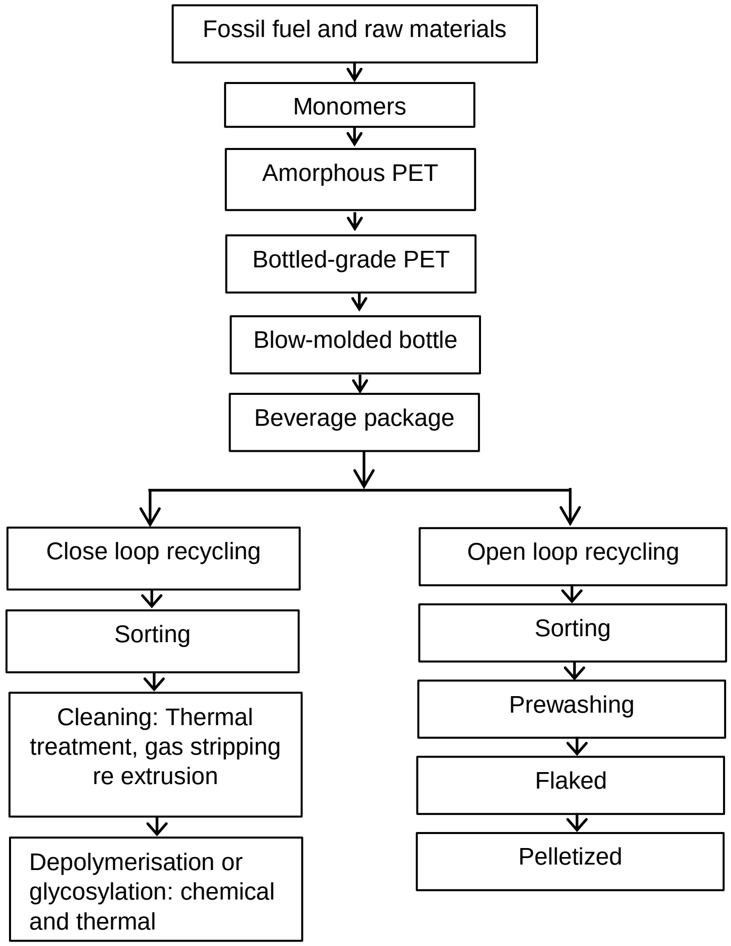


Figure 2.1: PET bottles production and recycling. Adapted from Andrady (2015).

In the present study, mainly bottles used for drinking water and soft drinks will be used. The open loop option will be used considering that a major goal of the present study is to keep the costs as low as possible. Some stages such as prewashing, washing, and pelletizing that increase costs would not be used. However, the lids and labels of the bottles will be manually removed.

2.2. Shredding methods for incorporation of waste PET into concrete.

The majority of previous studies used unwashed waste PET, but a few studies such as Saikia and de Brito (2007) and Ferreira et al. (2012), washed the waste PET with chemical substances to remove contamination or impurities. However, washing leads to extra time and expenses.

In the majority of previous studies, waste PET was processed using only mechanical means. The material was shredded and sieved. However, some investigators (e.g. Choi et al. (2005), Khoury et al. (2008), Choi et al. (2009), and Ferreira et al. (2012)) used an additional thermal process after grinding to obtain waste PET pellets. Ferreira et al. (2012) washed the waste PET, heated it into a reactor power vacuum, and extruded it through an extruder spindle. Choi et al. (2005) and Choi et al. (2009) mixed the waste PET with sand at a temperature of 250 °C, then air-cooled it and sieved it. Khoury et al. (2008) mixed the grinded waste PET with soil and melted it until the waste PET reached a uniform consistency.

From the above studies, no information on the type of shredders used were mentioned.

2.3. Influence of mixture components on workability, compressive strength, and splitting tensile strength

The mixture of the components and the ratios among some components are influential to some concrete properties (Newman and Choo, 2003). In the present study, these influential aspects are reviewed. Additionally, workability, compressive strength, and splitting tensile were identified as key properties and are discussed in the present study.

Workability refers to the consistency or the ease of handling and working with fresh concrete. Influential factors that affect workability are the water content of the mixture, the ratio between aggregate/cement, grading of aggregates, maximum size of aggregates, fineness modulus (which is an indication of how fine is the aggregate).

The shape and texture of the aggregates are the most important factors that influence workability in the mixture (Mehta and Monteiro, 2014). Strength of the concrete is one of the most important and appreciated characteristics of this material. Porosity also plays an important role in the properties of concrete because high porosity decreases strength. The porosity of aggregates and the interfacial zone between the cement paste and coarse aggregates significantly influence strength. Factors such as water/cement ratio, curing time, aggregates, and admixtures highly impact the porosity of the mixture and subsequently the strength. Additionally, deleterious materials such as organic impurities affect the bonding between the cement past and the coarse aggregates resulting in a low strength of the mixture (Mehta and Monteiro, 2014). The resistance of the concrete to be tensioned is tested through the splitting tensile strength test (Mehta and Monteiro, 2014). Factors such as aggregates, water/cement ratio, cement type, and admixtures can influence the splitting tensile strength (Mehta and Monteiro, 2014).

2.4 Influence of waste PET as an aggregate on workability, compressive strength and splitting tensile strength

2.4.1 Aggregate replacement

The effect of the size and type of waste PET aggregate was reviewed based on the work of Ferreira et al. (2012) and Silva et al. (2013). Silva et al. (2013) substituted and tested both coarse and fine waste PET aggregates. The authors concluded that the incorporation of waste PET as a coarse aggregate decreased the mechanical properties more than concrete incorporated with waste PET as a fine aggregate. Furthermore, Ferreira et al. (2012) compared the incorporation of angular shape coarse aggregates, angular shape fine aggregates, and rounded and homogeneous waste PET pellets. The authors observed that in general when waste PET was incorporated, the compressive strength and splitting tensile strength decreased. However, the mixes that contained waste PET pellets showed the least reduction, followed by mixes containing waste PET as fine aggregate. Ferreira et al. (2012) and Silva et al. (2013) also agreed that as waste PET size increases, the porosity of the sample also increases. Thus, as larger particles of waste PET are used, the mechanical properties of the mixture will diminish.

Albano et al. (2009) investigated the effect of particle size of waste PET when it is added to concrete. The authors used two different particle sizes, 2.6 mm and 11.4 mm, as well as a mix of 50% of each size. The authors asserted that with the 50% mix, the slump values were higher, indicating a better distribution of particles compared with only one size. The authors also pointed out that in the 50% mix, the mixture's fluidity increased while the porosity decreased. Thus, slump and mechanical properties increased compared with mixes with only one size of waste PET particles.

Frigione (2010) used a blade mill grinder to obtain very fine waste PET particles. However, this type of equipment requires more energy consumption and additional steps in the process. Additionally, Frigione (2010) separated the particles according to their size and reorganized them with specific percentages of each size in the mix. Thus, the author obtained an excellent gradation of fine particles ranging from 0.15 to 2.36 mm. Frigione (2010) argued that concrete with a replacement of fine waste PET particles of 4% volume, cement content ranging from 300 to 400 kg/m^3 and water/cement of 0.45, only reduced the compressive strength and splitting tensile by 0.4% and 1.9% respectively, compared with reference concrete.

2.4.2 Waste PET percentage incorporation

Batayneh (2007), Ismail and Al-Hashmi (2008), and Albano et al. (2009) used replacement of sand by waste PET percentages up to 20%. All the studies included waste PET shredded and replaced fine aggregates. In all the investigations the blends showed a decrease in slump, compressive strength, and splitting tensile when compared to reference mixes. Albano et al. (2009) observed that the mix with 10% of replacement of waste PET, two particles sizes of waste PET, and w/c of 0.50 showed the best mechanical property values, reaching compressive strengths between 21 to 30 MPa. The authors also observed that the mixtures with higher particle size (11.4 mm) and 20% waste PET replacement provided the lowest compressive strength and splitting tensile values.

Waste PET replacement percentages up to 15% were used in Ferreira et al (2012) and Silva et al. (2013). Ferreira et al (2012) reported that the best values of mechanical properties were obtained with the lowest WPET replacement. However when using 10% and 25% of replacement with pellets, the values of compressive strength reached over 30 MPa when using 10% and 15% replacement. Additionally, Silva et al. (2013) reported that when increasing the WPET substitution, the water absorption increased proportionally.

Choi et al. (2005) and Choi et al. (2009) incorporated waste PET to concrete with percentages of 25%, 50%, 75%, and 100%. Both studies used thermal processes to heat, melt, and mix waste PET with sand. Contradictory to other studies, these authors argued that the slump increased proportionally with the increase of WPET replacement regardless the w/c ratio. Additionally, the authors noted that the compressive strength obtained for mixes with 25%, 50%, and 75% only decreased 6%, 16%, and 30%.

Batayneh et al. (2007) declared that with up to 20% volume of waste PET replacement, the workability decreased less than 15%. However, the mechanical properties suffered a decreased of 72% compared to reference mixes. Batayneh et al. (2007) also claimed that for replacements of 5% V (volume) of waste PET, the reduction in mechanical properties was under 25%**.**

2.4.3 Water/cement ratio in mixtures containing waste PET

Choi et al. (2005) and Choi et al. (2009) utilized w/c ratios of 0.45, 0.49, and, 0.53 as mentioned previously, the authors observed that with any w/c ratio value, in both studies the slump increased when increasing waste PET substitution. The authors also stated that the best mechanical properties were obtained when using w/c ratio of 0.49 and 25% substitution. In Choi et al. (2005) blast furnace was added to the mix. The authors found that with the addition of blast furnace, the compressive strength reduced. However, the workability improved as the replacement ratio, blast furnace percentage, and w/c ratio increased.

Frigione (2010) employed w/c ratios of 0.45 and 0.55. The author states that at a low w/c ratio of 0.45, the difference in compressive strength between 28 days and 1 year is insignificant. However, when using a high w/c ratio, such as 0.55, the bleeding effect produces a significant reduction in compressive strength.

Albano et al (2009) argued that as particle size and WPET percentage have a large impact in the decrease of the mechanical properties, high values of w/c ratio also have a detrimental impact in the mixture properties. The authors explained that this is because of the low absorptivity of waste PET. Thus, the water in the mixture that did not react with the concrete will create voids and empty spaces that increase the porosity of the mix and affect the mechanical properties of the concrete.

Ferreira et al. (2012) studied the influence of curing conditions on the mechanical and durability properties of concrete that contained WPET as coarse, fine and spherical aggregates. The authors concluded that despite the type of curing or the curing conditions, the mechanical and durability properties of all the mixes decreased. The authors also observed that all the mixes have better performance with humid curing conditions. Drier curing regimes benefit mechanical properties in the short term, while humid regimes benefit mechanical properties in longer terms.

Ismail (2008) studied the behavior of the mixes containing waste PET under varying curing conditions compared with reference mixes. According to the author, the mixes containing waste PET under curing conditions experienced a slight increase in the mechanical properties, while the reference mixes increase their mechanical properties significantly when exposed to curing conditions**.**

2.5 Ranges of the components in mixtures containing waste PET

Appropriate ranges of variation for each component of the mixture should be set as part of the mixture design process. The available information on the incorporation of waste PET into concrete has been reviewed and the proportions of components are analyzed to provide a guide for the present study. In total 10 studies out of the available information offered complete information on the experimental details. Additionally, the properties obtained in the studies were also reviewed in order to apply the available knowledge for the present study.

2.5.1 Cement content

The minimum cement content utilized was 295 kg/m^3 by Juki et al (2013) and the maximum value was 446 kg/m^3 by Batayneh et al. (2007) and Siddique et al., (2007). Figure 2.2, shows that out of 10 studies, the median of the researchers used 350 kg/m^3 and 50% of researchers used the range of 327 kg/m^3 to 428 kg/m^3.

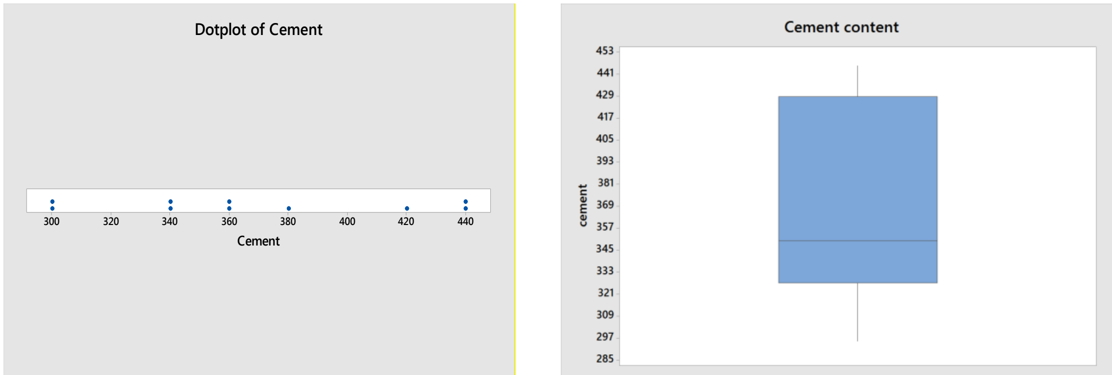


Figure 2.2: Cement fraction used in past research.

2.5.2 Water/ cement ratio

The minimum w/c ratio was 0.45 used in several studies such as Frigione (2010), Açkazönoglu (2010), and Juki et al (2013). The maximum w/c ratio was 0.65, employed by Juki et al (2013). Figure 2.3, shows that with a sample size of 10 studies, the median of the researchers used a w/c ratio of 0.53, and 50% of the studies used the range from 0.45 to 0.56.

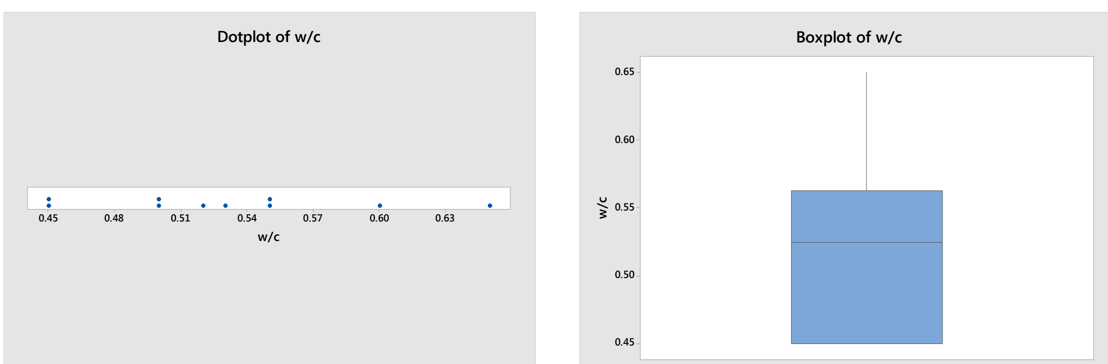


Figure 2.3: w/c ratio employed in past research.

2.5.3. Coarse aggregate content

The minimum value of coarse aggregate used in past research was 961 kg/m^3 by Batayneh et al. (2007), the maximum value used was 1600 kg/m^3 by Frigione (2010). Figure 2.4 depicts that with a sample size of 10 studies, the median of the researchers used 1050 kg/m^3 of coarse aggregate. While 50% of the studies utilized coarse aggregate ranging from 960 to 1510 kg/m^3.

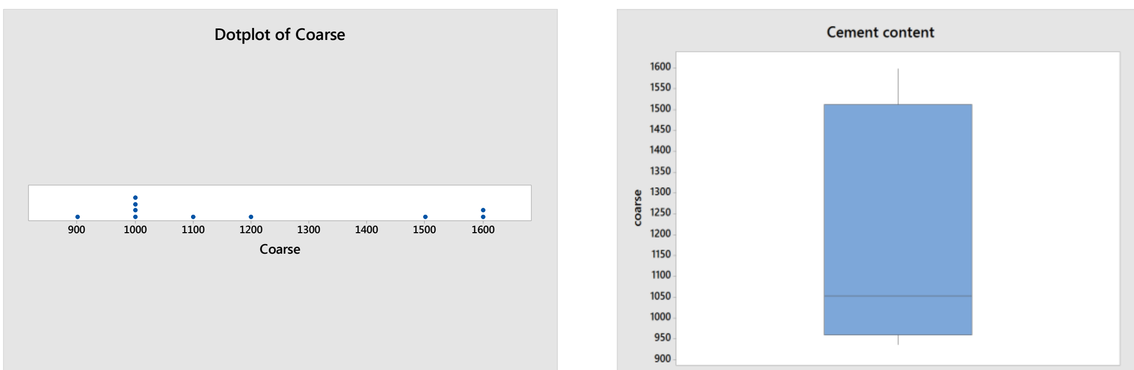


Figure 2.4: Coarse aggregate proportion used in past research.

2.5.4. Fine aggregates

The minimum value of fine aggregate employed in the studies was 420 kg/m^3 used by Frigione (2010). The maximum value was 901 kg/m^3 used by Silva et al. (2013). Figure 2.5 shows that with a sample size of 10 studies, the median of the researchers used 566 kg/m^3 of fine aggregate, while 50% of the studies used fine aggregate ranging from 495 kg/m^3 to 800 kg/m^3.

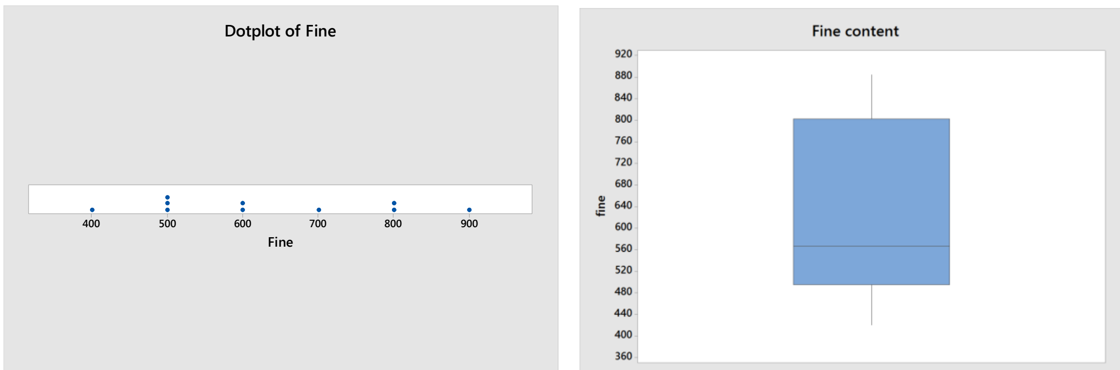


Figure 2.5: Fine proportion used in past research.

2.6 Properties of mixtures containing waste PET

Past research has evaluated mostly slump and mechanical properties of the concrete mix. Few authors also included durability properties such as water absorption, shrinkage, and chloride resistance. However as mentioned before, the present research focuses only on slump, compressive strength, and splitting tensile.

2.6.1 Workability

In Choi et al., 2005; Choi et al., 2009 ; Batayneh et al., 2007; Albano et al., 2009; Ferreira et al., 2012 workability was measured with the slump test according to the standard ASTM C-143/C143M-15, with a cone of 30 cm height and 10 cm diameter.

The majority of the studies such as (Ismail and Al-Hashmi, 2007; Batayneh et al., 2007; Albano et al., 2009; Frigione, 2010; Akcaozoglu, 2010; Saikia and de Brito, 2012; Juki, 2013; Ge et al., 2014) found that the slump of the concrete containing waste PET aggregate decreases with the increase in the substitution. These authors concluded that the reduction was caused by the angular sizes and the sharp edges of the waste PET aggregates. On the contrary, Choi et al. (2005) and Choi et al. (2009) reported that the slump increased when increasing the incorporation of waste PET particles. In these studies the waste PET particles were melted and mixed with sand that generated spherical and smooth particles. However, Ferreira et al (2012) that also included smooth waste PET particles (pellets) as part of the experimentation declared that the slump reduced when increasing waste PET substitution.

Batayneh (2007) declared that with a waste PET substitution up to 20% the slump reached 58 mm which the authors considered an acceptable value for workability. Ferreira et al (2012) used a constant w/c ratio, waste PET replacements of 7.5% and 15% with fine, coarse, and fine pellets and obtained a desired slump ranging in 130 mm +/- 10. Silva et al. (2013) claimed that they obtained slump values of 130 mm +/- 15 in all the mixes using replacements of 7.5% and 15% waste PET, with both coarse and fine aggregates, and w/c values ranging between 0.52 and 0.61.

2.6.2 Compressive strength

The majority of authors established the compressive strength according to the standard ASTM C-39/C39M-18 at 28 days. Some authors also reported values of compressive strength at 1, 3 or 7 days.

Choi et al., 2005; Choi et al.,2009; Ismail and Al-Hashmi, 2007; Albano et al., 2009; Frigione, 2010; Akcaozoglu, 2010; Saikia and de Brito, 2012; Juki, 2013; Saikia and de Brito, 2012; Ferreira et al., 2012 stated that the inclusion of plastic aggregates into the concrete mix decreases the compressive strength. Authors such as (Choi et al., 2005; Choi et al.,2009; Albano et al., 2009; Saikia and de Brito, 2012; Ferreira et al., 2012) observed that a low bonding between waste PET and the cement paste as well as, the poor capacity of waste PET to allow water movement into the mix were important causes that decrease the compressive strength. Furthermore, these authors observed that the decrease in compressive strength was proportional to the increase of waste PET substitution. Albano et al (2009), Ferreira et al. (2012), and Silva et al (2013) pointed out that angular shape, big size and poor gradation of waste PET are other important reasons that explain why compressive strength can decrease. Authors such as Batayneh et al. (2007), Frigione (2010), and Saikia (2012) also identified the influence of a high w/c ratio in the decrease of the compressive strength. For instance, Frigione (2010) stated that when substituting fine aggregates with 4%V of PET particles with fine particles (5mm - 150μm) at a low w/c ratio (0.45) the compressive strength at 28 days only reduces approximately 2%. However, when increasing the w/c ratio the compressive strength can reduce significantly. Juki et al. (2013) stated that with substitutions of 25%, 50% and 75% of fine particles of waste PET ranging from 0.1 to 5 mm and a low w/c ratio of 0.45 the highest values of compressive strength were found for all substitutions.

Despite the reductions in compressive strength of concrete incorporating waste PET the authors found values that can be useful. For instance, Silva et. al (2013) obtained a compressive strength of 29.6 MPa when using a mixture of w/c ratio of 0.56 and 7.5% replacement of fine waste PET. Albano et al. (2009) obtained a compressive strength of 22.5 MPa with a w/c of 0.5 and a substitution of 20% of fine aggregate.

2.6.3 Splitting tensile strength

Choi et al., 2005; Choi et al.,2009; Ismail and Al-Hashmi, 2007; Albano et al., 2009; Frigione, 2010; Akcaozoglu, 2010; Saikia and de Brito, 2012; Juki, 2013; Saikia and de Brito, 2012; Ferreira et al., 2012 reviewed and identified that the replacement of natural aggregates for waste PET decreases the splitting tensile strength. Frigione (2010) and Ferreira (2012) emphasized that similar to compressive strength, there is a detrimental influence on the splitting tensile strength when increasing waste PET substitution and w/c ratio. However, Batayneh et al. (2007) argued that, despite that splitting tensile reduces when incorporating waste PET, splitting tensile reduces less than compressive strength. Albano et al. (2009) obtained values ranging between 2.3 and 2.5 MPa with w/c of 0.50 and 10% fine replacement by waste PET.

2.7 Traditional methods of concrete mix design and preparation

Beall (2001) asserts that concrete is produced through a controlled mix of cement, water, coarse aggregate, fine aggregate, and in some cases, additives. Additionally, the proportions of these components as well as the mixing method can affect the resulting properties of concrete (Beall, 2001). The conventional concrete mix design methods (Figure 2.6) include the determination of influential aspects such as w/c ratio, cement content, coarse and, fine aggregates according to desired properties such as slump and compressive strength (Newman and Choo, 2003). However, conventional methods do not consider interactions among components, do not optimize the mixture, and they require a large number of trial mixes to obtain the desired outcome (Kharazi, 2013; Simon et al., 1997). The outcome of traditional methods is a trial mix that provides guidance to start the experimentation process (Kharazi, 2013). Additionally, the information and charts included in the conventional methods do not include the influence of other materials such as waste PET.

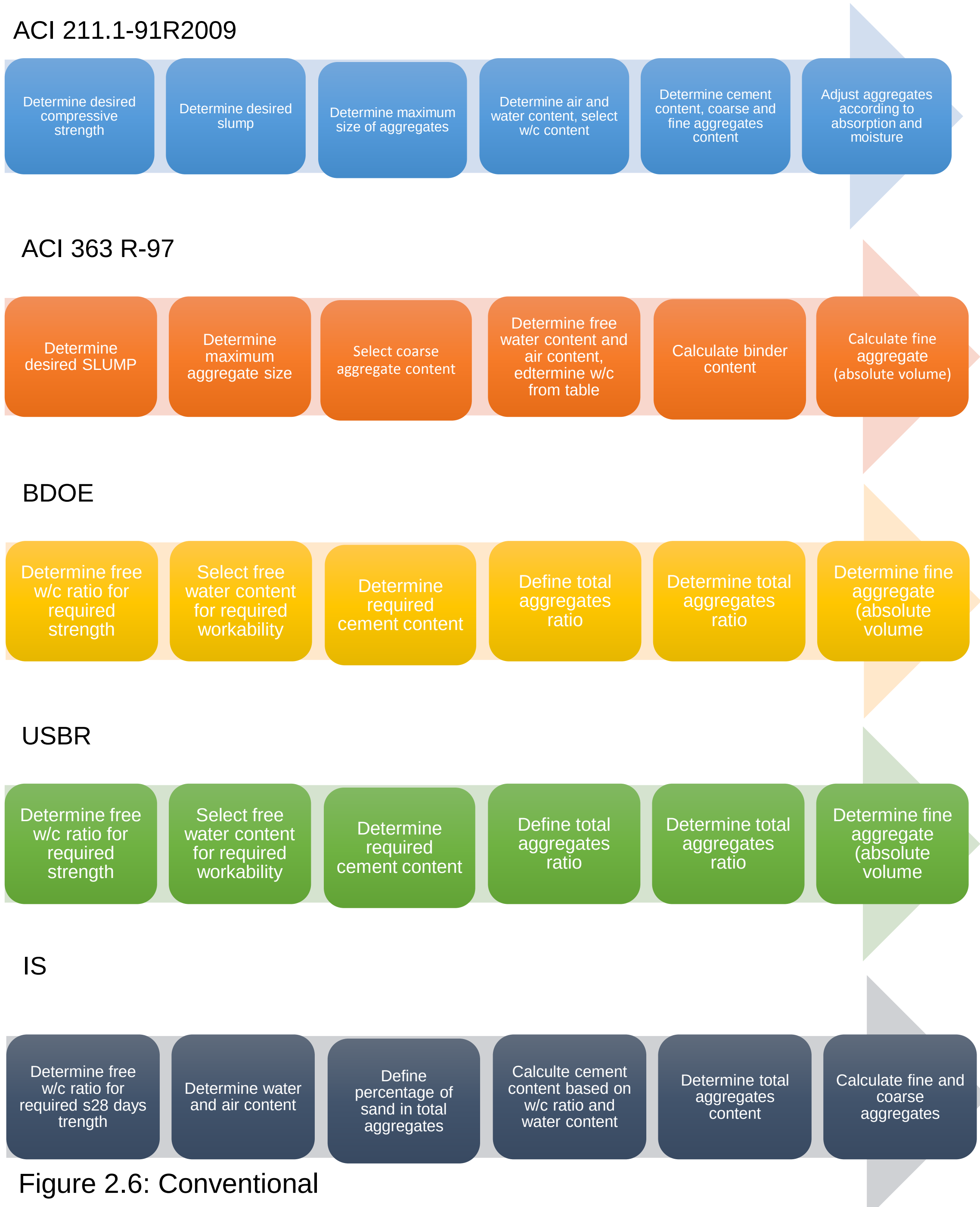


Figure 2.6: Conventional concrete mix design methods. Adopted from Kharazi (2013).

In contrast, when designing concrete mixtures by the statistical mixture design approach, the components are modified and tested together instead of one at time (Cornell, 2002). The components and their interactions are carefully analyzed using statistical tools (Myers and Montgomery, 2009). In mixture design, mathematical methods are employed to select specifically the experiments that will offer significant information. Thus, fewer experiments that offer valuable information should be conducted (Lye, 2002). After the experimental execution, the data are analyzed and a regression model fitted to give a predictive model (Cornell, 2002). Once, the model is selected, numerical or graphical optimization takes place and the desired values or ranges of each component are established and possible sets of combinations of components that meet all the constraints are obtained (Cornell, 2002; Myers and Montgomery, 2009).

2.8. Previous studies using statistical mixture design approach

There is no currently available information on the incorporation of waste PET into concrete using mixture design approach. However, there are a variety of studies that use the mixture design approach for concrete mixtures. Kharazi (2013) completed a comprehensive study on the design and optimization of concrete using mixture design. The author tested a five components mixture (cement, water, coarse aggregate, fine aggregate, and HRWRA) with only 20 experiments.

The author provided a comprehensive database, which provides information on concrete performance and optimization. It includes slump, 3-7-28-56 day compressive strength, and 3-7-28-56 modulus of rupture. The author found the predictive models that provided the optimal mixes are within the desired values.

Simon, Lagergren, and Snyder (1997) formulated high performance concrete using statistical mixture design. The authors optimized a six component mixture (cement, water, microsilica, HRWRA, coarse aggregate, and fine aggregate) that had several limits and performance restrictions. In total 36 mixes were prepared and analyzed. The authors tested the mixes for workability (through slump test), 1 and 28 days compressive strength, and rapid chloride test. The authors found that the slump and rapid chloride tests results fitted linear models, while the 28 days compressive strength fitted in a quadratic model. Then, using the optimization tool and with the identified predictive models the authors established several combinations of components that met all the high performance requirements.

Santos et al., (2017) used the mixture design approach to determine the appropriate proportion of metakaolin and limestone filler in the cement paste. The authors held a constant w/c ratio and superplasticizer percentage. In order to test the viscosity of the paste, the flow time was measured through the Marsh funnel.

Yield stress was measured through an oscillatory rheometer to measure the thixotropy. Compressive strength was measured only after 7 days of curing as the goal of the research was mainly based on the rheological behaviour of the mixes. The authors found that the thixotrophy recovery of the mixture fitted a linear model, the viscosity followed a quadratic model, and the elastic modulus fitted a cubic model. Finally, using the optimization function, the authors set the desired properties and obtained the combination of cement, metakaolin, and limestone filler that fulfilled all the requirements.

Kunhanandan and Ramamurthy (2006) studied the influence of the components in a foam concrete using response surface methodology. The authors tested the incorporation of fly ash as replacement of sand as fine aggregate. The fly ash incorporation percentages of 50, 65 and 70% of were tested as well as the filler/cement ratio and the foam volume. Compressive strength at 7-28 and 90 days was tested as well as the dry density. The dry density was found to be linear, while the compressive strength at all ages was found to be quadratic. The response surface and contour plot were used to find the optimal level of the components and the appropriate percentage of fly ash was found at all ages.

2.9. The statistical mixture design approach

In mixture design, the variables to be modified are considered ingredients or components and the measured response depends on the proportion of each component (Myers and Montgomery, 2009; Cornell, 2009). When a proportion of a component increases, the proportion of some of the other components should decrease (Myers and Montgomery, 2009). Cornell (2002) points out that simplex design is the most basic mixture design. It is used to examine the influences caused by the components when all possible combinations of each component can be used without restrictions and the total sum of the mixture is equal to the unity (Cornell ,2002).

On the other hand, Cornell (2002) states that a mixture design is considered constrained when the components of a mixture have additional restrictions, such as maximum or minimum limits. The constrained mixture design is appropriate for concrete mix preparations. This is due to the restrictions on the components, which depends on the desirability of determined properties (Myers and Montgomery, 2009). The constrained design can be solved as a simplex design by using pseudo components (x’). Through the pseudo components the original proportions are changed in the range of 0 to 1 and the design can be assumed as a simplex design as shown in Equation (2.1) and Figure 2.7 (Myers and Montgomery, 2009; Cornell 2002).

$$0 \leq L_i \leq x_i \leq U_i \leq 1 \; with \; i = 1,2,3..q \qquad (2.1)$$

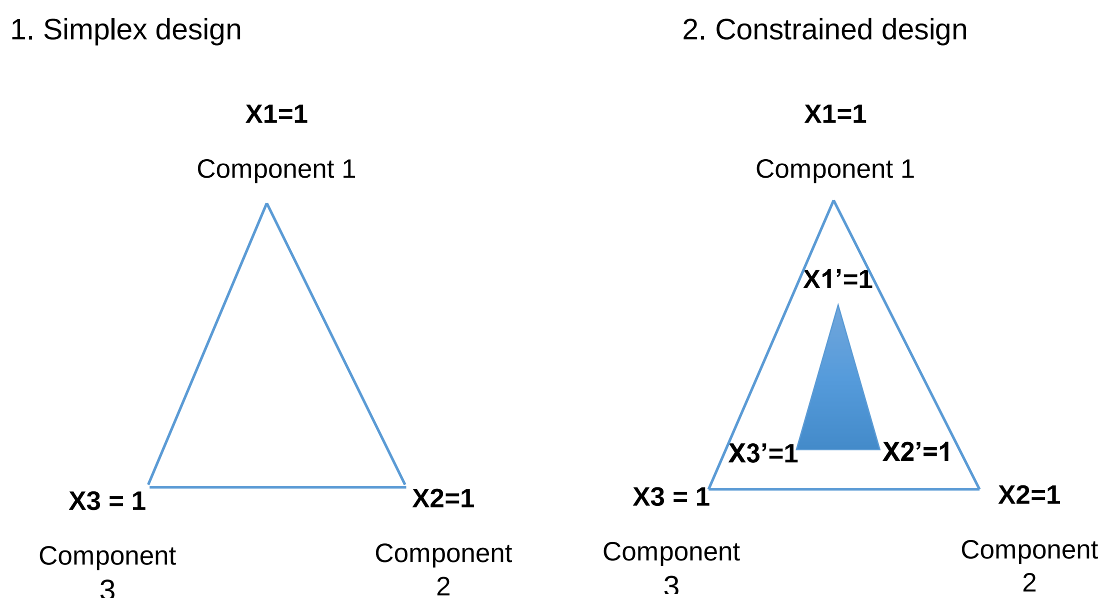


Figure 2.7. 1. Simplex design, 2. Constrained design. Adapted from Cornell (2009).

The design space implies the possible combination of the components restricted by the limits or constraints (Myers and Montgomery, 2009). The figure shows the representation of the design space of two mixes: 1) simplex design, the triangle describes the space among three components. The components are shown as X1, X2, and X3. 2). The inner triangle describes the actual constrained design area among three components, while the outside triangle describes a simplex design area. The pseudo components used to transform a constrain design in a simplex design are shown as X1’, X2’, and X3’.

2.10.1 Scheffé equations for mixture designs

Considering the restriction of the mixture design that the total sum of the components is unity and that the measured response depends on the component’s proportions, Scheffé developed the basic equation for a simplex design from the original polynomial equation as follows

Considering a first order polynomial model as equation (2.2),

$$E(y) = \beta_0 + \sum_{i=1}^{q} \beta_i x_i \qquad (2.2)$$

Taking into account the restriction of the unity as shown in equation (2.3) and multiplying some terms on the original model by the identity in equation (2.4), Scheffé obtained the basic equation for simplex designs (2.5).

$$x1 + x2 + xq + \cdots = 1 \qquad (2.3)$$

$$E(y) = \beta_0\left(x1 + x2 + \cdots x_q\right) + \sum_{i=1}^{q} \beta_i x_i \quad (2.4)$$

$$E(y) = \sum_{i=1}^{q} \beta_i^{*} x_i \qquad (2.5)$$

where $\beta_i^{*} = \beta_0 + \beta_i$ in equation (2.5)

This is the canonical or Scheffé form based on the original linear regression applied to a simplex mixture design. The model was applied to linear, quadratic and cubic models for the basic simplex design obtaining equations: (2.6) for the linear model, (2.7) for the quadratic model, (2.8) for the full cubic model, and (2.9) for the special cubic model (Myers and Montgomery, 2009).

Linear

$$E(y) = \sum_{i=1}^{q} \beta_i^{*} x_i \qquad (2.6)$$

Quadratic

$$E(y) = \sum_{i=1}^{q} \beta_i^{*} x_i + \sum_{i<j=2} \sum^{q} \beta_{ij}^{*} x_i x_j \quad (2.7)$$

Full cubic

$$E(y) = \sum_{i=1}^{q} \beta_i^{*} x_i + \sum_{i<j=2} \sum^{q} \beta_{ij}^{*} x_i x_j + \sum_{I<J=2} \sum^{q} \delta_{ij} x_i x_j \left(x_i - x_j\right)$$

$$+ \sum \sum_{i<j<k=3} \sum^{q} \beta_{ijk} x_i x_j x_k \qquad (2.8)$$

Special cubic

$$E(y) = \sum_{i=1}^{q} \beta_i \quad x_i + \sum_{i<j=2} \sum^{q} \beta_{ij} \quad x_i x_j + \sum \sum_{i<j<k=2} \sum^{q} \beta_{ijk} x_i x_j x_k \quad (2.9)$$

Where β_i in all the equations represents the expected response for the pure mixture.

2.9.2. Analysis of the data in constrained form

The mixture of concrete represents a complex statistical space with lower and upper bounds as well as extra constraints such as ratios among components (Myers and Montgomery, 2009). The experimenter should assure an appropriate number of experiments that will offer a complete understanding of the behavior of the components and their proportions (Cornell, 2002).

The optimal designs employ algorithms for the selection of specific points (combination of components) in a constrained design, such as a concrete mixture. (Anderson and Whitcomb, 2009). This algorithm selects adequate points to reduce the variance on the predictor model (Myers and Montgomery, 2009).

According to Myers and Montgomery (2009) for a linear prediction model, the model selects vertices, edge centers, overall centroid, and axial points. For a quadratic model fit it is necessary to include vertices, edge centers, constrained plan centroids, axial, and overall centroid. Finally, for the cubic model it is necessary to include the vertices, thirds of edges, the constraint plane centroids, overall centroid and axial points (Myers and Montgomery, 2009). The feasible points are shown in the Figure 2.8. Vertices are points 1,2, and 3. Edge centers, points 4,5, and 6. Axial check points, 7,8,9, 10, and 11. And overall centroid point 12.

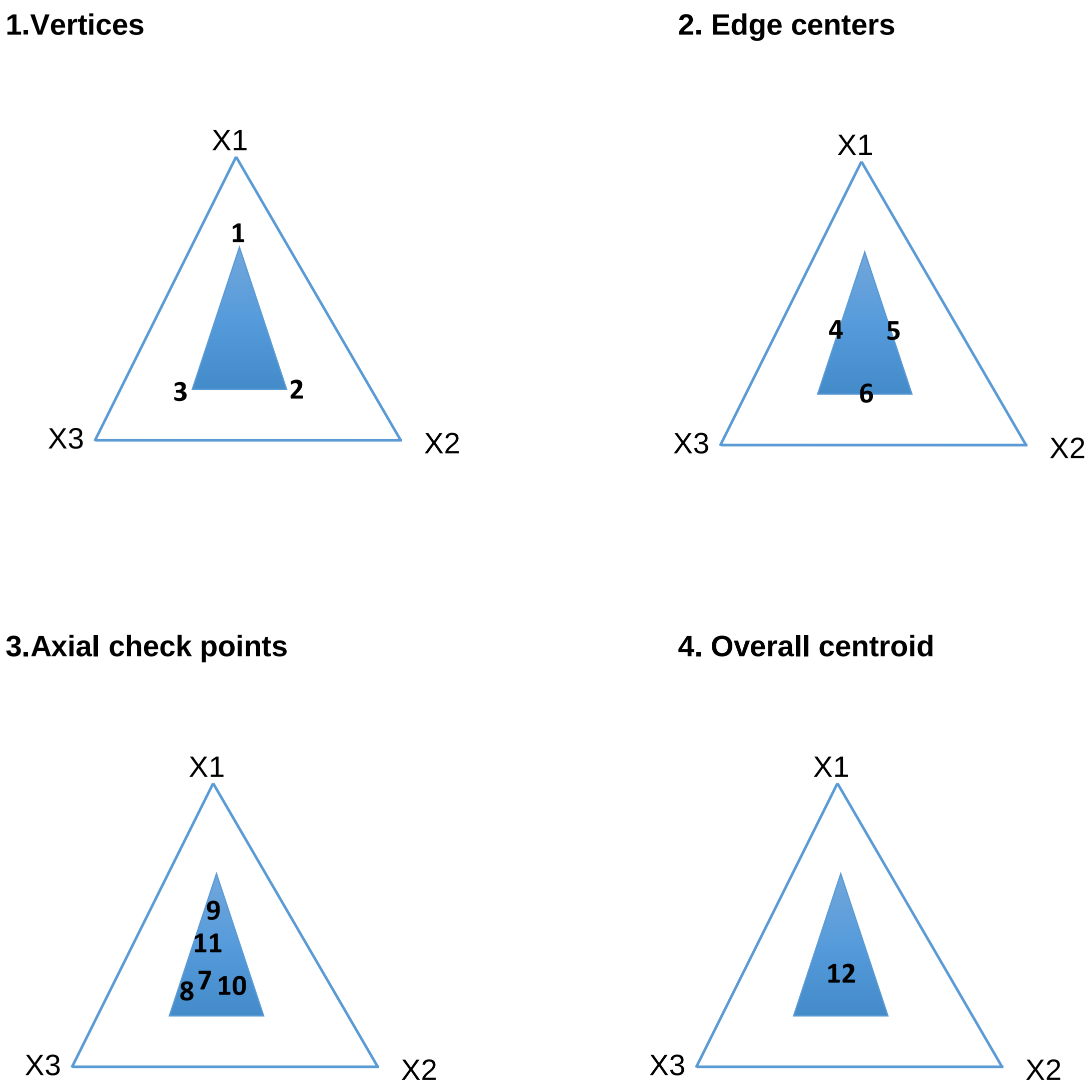


Figure 2.8: Points in a constrained mixture design of three components. Vertices are points 1,2, and 3. Edge centers, points 4,5, and 6. Axial check points, 7,8,9, 10, and 11. And overall centroid point 12.

2.9.3. Estimation of the model and optimization

The experiments should be conducted in a randomized and independent manner (Cornell, 2002). Once the experiments are conducted and the response is measured, analysis of variance (ANOVA) and regression analysis are employed to obtain a functional relationship between the responses of interest and the significant components. A linear or polynomial model may be fitted (Cornell 2002; Montgomery, 2009). Each model, component, and interactions among components are statistically evaluated and the null hypothesis is rejected when the components in the mixture influence the measured response (Myers and Montgomery, 2009). The significance level (α) is usually set at 0.05.

The adjusted and predicted coefficients of determination (adjusted R-squared, predicted R-squared) are analyzed to check if the model can be properly explain the variation of the data. The residuals are divided into residuals error and pure error from replicates. The lack of fit is considered significant when the residuals error exceeds the pure error (Myers and Montgomery, 2009). Finally, Myers and Montgomery (2009) point out that the adequacy of the model should be verified by an analysis of residuals. The residuals' behavior can show possible problems with the selected model (Myers and Montgomery, 2009). Thus, a graphical verification of the residuals is executed to ensure that all the assumptions are met when fitting the selected model and the power of the model is enough to predict the data behavior (Anderson and Whitcomb, 2009).

Once the predictive models are statistically selected for each response, the optimization can be executed. First, trace and contour plots are performed to analyze the influence of the components on the responses, and then the numerical optimization is carried out. The goals of the optimization for the responses and components must be first selected. The goal can be none, maximize, minimize, or approach a target. Then the limits of the components must be selected (lower and upper) (Anderson and Whitcomb, 2009). Finally, the desirability function approach will be used to obtain the set of solutions which will meet the requirements (Anderson and Whitcomb, 2009). These combinations are tested and verified in the experimental process. If there is agreement between the actual and the predicted values, the experimental design was successful. On the contrary, if there is not agreement, the design can be augmented or the ranges of variation can be changed (Anderson and Whitcomb, 2009).

2.10. Applications of concrete based on workability and compressive strengt

The focus of the present research is to incorporate waste PET into concrete mixture to produce a useful new product. The criteria of performance for the present study will be linked to the minimum standards of workability and strength as a first stage to indicate the utility of this concrete. However, workability and compression strength are only indicators that the concrete could be useful, other important properties should be later tested such as freeze/thaw properties for cold climate use etc.

2.10.1 Workability standards

As workability relates to the ease to work with the concrete, it is important to determine what acceptable ranges for this property are. Table 2.1 shows acceptable ranges for different applications of concrete.

Table 2.1: Typical workability values. Modified from Mehta and Monteiro (2014) and Beall (2001).

Examples	Minimum slump (mm)	Maximum slump (mm)
Foundation basements, walls, slabs. Not exposed to weather	25	127
Foundation basements, walls. Exterior vertical concrete. Exposed to weather. Not exposed to water accumulation	76	127
Driveways, garage slabs, walks, patios.	76	127

2.10.2 Compressive strength standards

Regarding compressive strength, the intention of this study is to produce a mixture of concrete with low to moderate strength. Low strength category includes concretes with less than 20 MPa of compressive strength. While moderate category includes concrete with compressive strength between 20 to 40 MPa.

Table 2.2: Typical compressive strength requirements concrete. Modified from Beall (2001).

Exposure class	Examples	Minimum requirement (MPa)
F0	Basement and foundation walls and slabs, not exposed to freezing temperatures. Structures or members inside buildings such as garages, sidewalks and steps. Members buried in the soil bellow frost line.	17.5
F1	Members not exposed to ice or snow accumulation such as exterior walls, beams, and slabs.	21
F2	Members exposed to snow accumulation. Foundation or basement walls that will support snow accumulation.	24.5

CHAPTER 3

METHODOLOGY

In this chapter the methodology of statistical mixture design was applied for the mixing of the concrete incorporating waste PET. The components and their ranges were determined and set. The number of mixes was defined according to the mixture design approach. The experiments were setup and carried out in the concrete laboratory.

3.1 Summary of mixture design approach

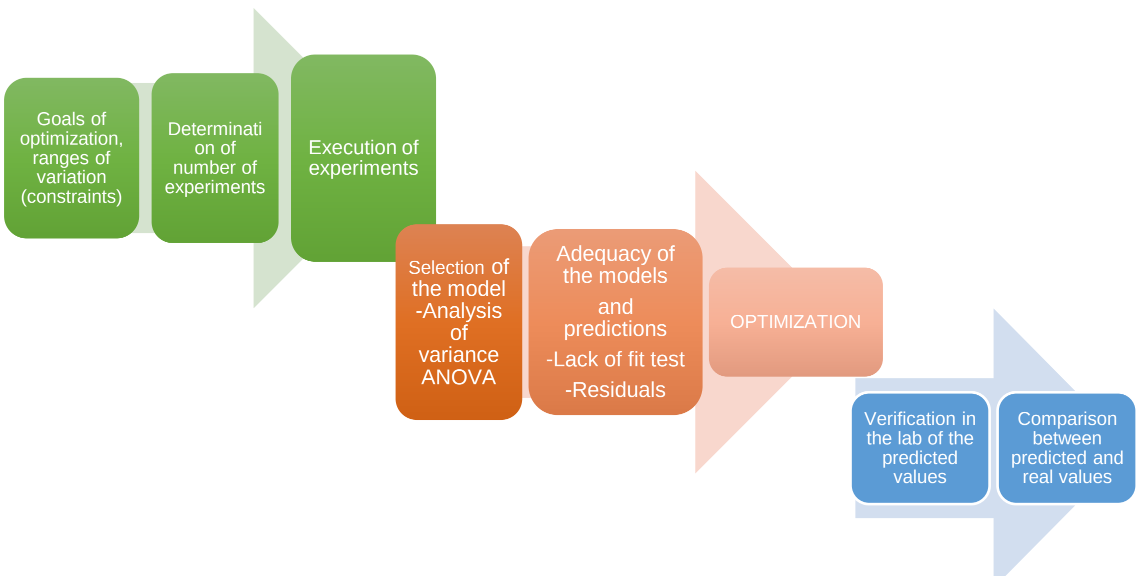


Figure 3.1: Mixture design process, adapted from Anderson and Whitcomb (2005) and Kharazi (2013).

Figure 3.1 outlines the mixture design process according to the stages described by Anderson and Whitcomb (2005) and Kharazi (2013). The first stage involves:

1) determination of optimization goals, selection of the components and their ranges of variation and additional constraints; 2) identification of the responses and the number of experiments; and 3) execution of the experiments and measurement of the responses.

The second stage involves 1) selection of the model through the analysis of variance (ANOVA); 2) analysis of adequacy, through lack of fit test, R-squared adjusted and predicted, and graphical analysis of residuals; and 3) optimization through graphical and numerical optimization using the desirable function approach.

In the third stage, the proposed optimal combination of components should be tested and confirmed (Myers and Montgomery, 2009). The actual and predicted values are compared. The experimentation is either completed or the design is augmented through the addition of few more experiments to obtain better results (Anderson and Whitcomb, 2005).

In this study, the constrained mixture design was employed, and the commercial available software Design Expert 10 (Statease Inc.) was used for the design, statistical analysis, definition of the points (experiments), and optimization of the mixture.

3.2. Optimization goals

One of the goals of this research was to generate a practical and useful mixture of concrete containing waste PET. Thus, the performance criterion was oriented to reach workability and compressive strength parameters for different applications. According to Beall (2001), the minimal requirements for compressive strength range between 17.5 and 24.5 MPa. Thus, the optimization would be oriented to maximize the compressive strength as much as possible. Additionally, workability would be set to range between 25 and 127 mm.

Table 3.1: Optimization goals for the present study.

Property	Minimum	Maximum
Compressive strength (MPa)	17	-
Slump (mm)	25	127

3.3. Selection of materials

The materials selected were based on achieving the proposed criteria as well as the availability of materials in the university lab. The selected materials were Portland cement, tap water, coarse aggregate (crushed stone), fine aggregate (sand), and waste PET aggregate. Furthermore, superplasticizer was added to the mixture to address concerns about w/c ratio and workability. Batch size is selected as 0.03 m^3 of concrete. Because of the small amount of concrete mixed in each batch, air volume was not considered as a component.

3.3.1. Cement

The cement selected was an ordinary Type 1 Portland cement. It is available at the university lab and meets the requirements of the ASTM C150/C150M-17.

3.3.2. Water

The water used for the experiments was tap water at room temperature from the university lab.

3.3.3. Aggregates

Fine and coarse natural aggregates were provided from local suppliers. The fine aggregate was sand (crushed granite). The coarse aggregate was crushed stone with a maximum size of 20 mm. Sieving of coarse aggregate was executed according to the standard ASTM C136M-14 as shown in Table 3.2 and Figure 3.2. The sieving analysis of fine aggregate was also performed according to the standard ASTM C136 as shown in Table 3.3 and Figure 3.3. The specific gravity of the coarse aggregate was tested according to the standard ASTM C127-15 and the specific density of the fine aggregate was measured according to the standard ASTM C 128-15. Table 3.4 shows the results of specific gravity, bulk density, and absorption for coarse and fine aggregate.

Table 3.2: Grading of the coarse aggregate used in the experiments.

Sieve size (mm)	% Passing by mass		
	Coarse 1	Coarse 2	Coarse 3
28	100	100	100
20	96	99.4	95.3
14	68.7	78.4	66.2
10	39.8	21.3	25.8
5	8	6.9	10.1
2.5	1.4	0.5	0.5

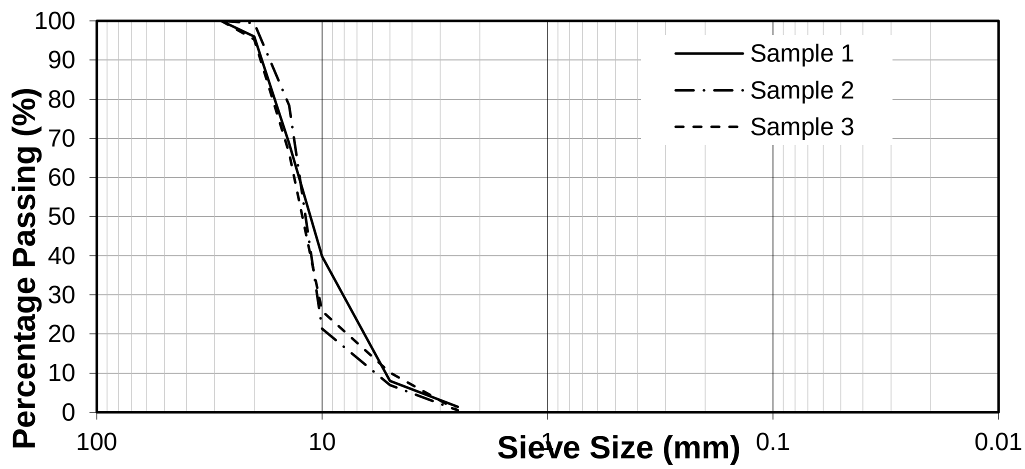


Figure 3.2: Grading curves of the coarse aggregate employed in the experiments.

Table 3.3: Grading of the fine aggregate used in the experiments.

Sieve size (mm)	% Passing by mass		
	Sand 1	Sand 2	Sand 3
10	100	100	100
5	97.3	96.1	97.7
2.5	80.8	75.8	88.4
1.25	60.8	58.9	68.4
0.63	48.9	45.6	46.5
0.32	27.3	21	26.5
0.16	15.4	13.4	10.3
0.1	3.4	6.5	3.3

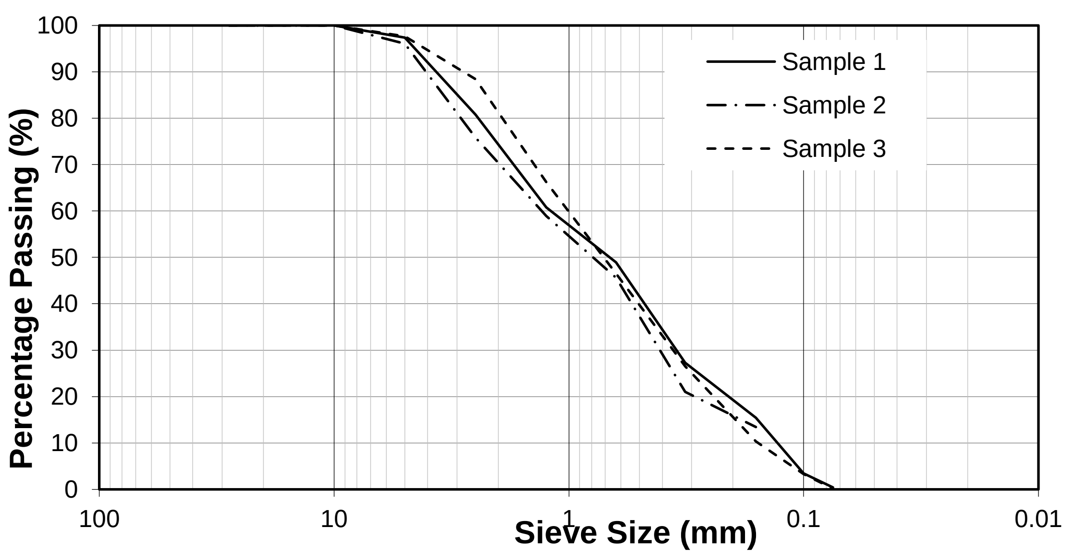


Figure 3.3: Grading curves of fine aggregate employed in the experiments.

Table 3.4: Properties of coarse and fine aggregate used in the experiments.

Property	Coarse	Fine
Specific gravity (kg/m^3)	2621	2617
Bulk density (kg/m^3)	1452	1635
Water absorption (%)	0.75	1.16

3.3.4. Superplasticizer

Choi et al., 2005; Choi et al.,2009; Ismail and Al-Hashmi, 2007; Albano et al., 2009; Frigione, 2010; Akcaozoglu, 2010; Saikia and de Brito, 2012; Juki, 2013; Saikia and de Brito, 2012; Ferreira et al., 2012 have concluded that when increasing the waste PET percentage in the concrete mixture, the slump decreases. Thus, superplasticizer ADVA 190 ASTM C494 Type A and F and ASTM C1017 Type I was incorporated into the mixture in order to obtain enhanced workability of the mix and incorporate higher proportions of waste PET into the concrete.

3.3.5. Waste PET

The waste PET was collected from the waste stream at the St. John's recycling centre. The collected waste PET was mostly water and soft drink bottles. Three main types of waste PET were identified based on the volume and width of the bottles as shown in Table 3.5. The bottles were unwashed and not separated by colors. The labels and the lids were manually removed before shredding.

Table 3.5: Main types of bottles received from the recycling centre.

Type	Volume (ml)	Thickness (mm)	Typical use
1	500	0.10	Spring water
2	2000	0.25	Soft drink
3	591	0.40	Water, soft drink

(a) (b) (c)

Figure 3.4: Types of bottles used in the experiments. (a) Type 1, (b) Type 2, (c) Type 3.

Based on past research, the incorporation of waste PET as a fine aggregate produced more advantages than the incorporation of waste PET as coarse aggregate. Thus, the screen of the shredder was selected to generate as fine a particle as possible. The shredded particles were then reprocessed. It is important to note that, shredding waste PET to produce fine particles consumes higher energy and time than shredding waste PET into coarse particles.

3.3.5.1. Reduction in volume by shredding

There are no available plastic bottle shredder in St. John's, Newfoundland. There are however several commercially made shredders available from China. Unfortunately none of the suppliers is willing to ship to St. John's. In addition, cost of the available shredders were way above the budget allocated for this research.

In the present study, the shredder used was built by Memorial University of Newfoundland, Technical Services using plans from an international plastic recycling online organization called Precious Plastic (https://preciousplastic.com). This website organization is devoted to increasing knowledge about plastic recycling worldwide. A complete guide to building a pilot plastic shredder machine is available on the website. The electrical and structural plans from this guide were used to fabricate the shredding machine, Figure 3.5 shows the final result of the shredder machine built at Memorial University.

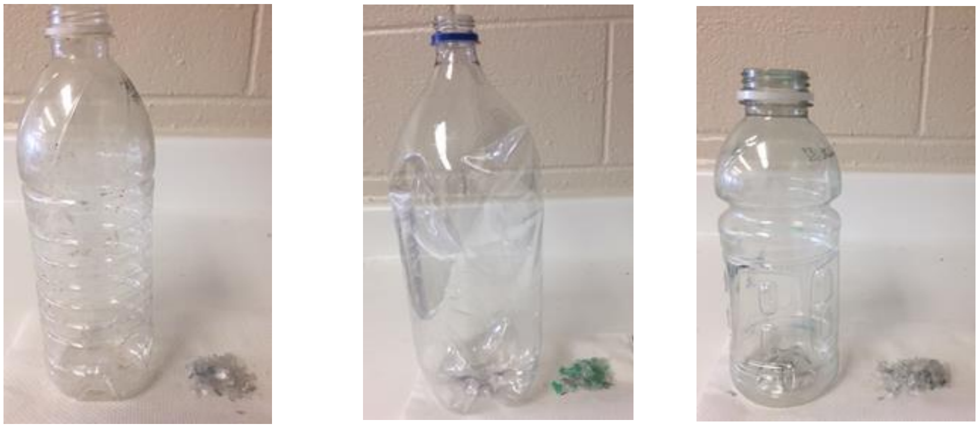
The volume obtained from shredding the waste particles using the shredder was measured and compared with the original volume. The bulk density was measured for all types of waste PET according to standards ASTM C29. Table

3.6 shows the results for the reduction of volume after shredding the different types of bottles. As shown in Table 3.6 the reduction of volume of the waste PET bottles was 29 fold or 96.6% for Type 1, 23 fold or 95.6% for Type 2, and 10 fold or 88.7% for Type 3. This shows that shredding the bottles provide tremendous savings in landfill or storage space.

Figure 3.5: Shredder built at Memorial University of Newfoundland.

Table 3.6: Reduction in volume of waste PET

Type of bottle	Thickness (mm)	Weight of 1 bottle (g)	Initial volume (ml)	Bulk density (g/ml)	Final volume of 1 bottle (ml)	Reduction in volume
1	0.1	6	500	0.34	17	29 fold (96.6%)
2	0.25	30	2000	0.34	88	23 fold (95.6%)
3	0.40	20	591	0.33	60	10 fold (90%)



(a) (b) (c)

Figure 3.6: Initial and final volume of the bottles used in the experiments. (a) Type 1, bottle of 500 ml and 0.1 mm of thickness; (b) Type 2, bottle of 2000 ml and 0.25 mm of thickness and (c) Type 3, bottle 591 ml and 0.4 mm of thickness.

3.3.5.2. Grading of waste PET

After shredding, sieve analysis was performed to determine the size and grading of the waste PET particles. In order to verify any difference in the size distribution of the particles due to differences in the thickness of waste PET, sieve analysis of each type of waste PET were tested. These tests were performed according to the ASTM C136-14. The results of the tests are presented in Table 3.7.

Table 3.7: Size distributions of different types of waste PET.

Waste PET	Sieve size						
	9.5mm	4.75 mm	2.36 mm	1.18 mm	0.6 mm	0.3 mm	0.15 mm
Type 1	100	97.4	18.43	6.1	0.27	-	-
Type 2	100	93.88	12.36	4.67	0.23	-	-
Type 3	100	93.62	16.74	3.66	0.43	-	-
Mix	100	96.09	17.19	4.89	0.26	-	-

As highlighted in Table 3.7, the waste PET had different size distributions due to the difference in the thicknesses. Thus, it was decided that the waste PET used for the experiments were selected. The proportions of each type of waste PET received from the recycling center were considered. The amount of bottles Type 1 was 50%, Type 2 was 30%, and Type 3 was 20%. Additionally, as the particle distribution of bottles Type 1 was better than the other two types, its percentage was increased. The waste PET was mixed for all the experiments using the following proportions: 70% Type 1, 20% Type 2, and 10% Type 3.

Figure 3.7 shows the final grading of the aggregates used in all the experiments. The waste PET gradation was deficient in particle sizes lower than 1 mm and had a high percentage of particles ranging between 5 and 9.5 mm.

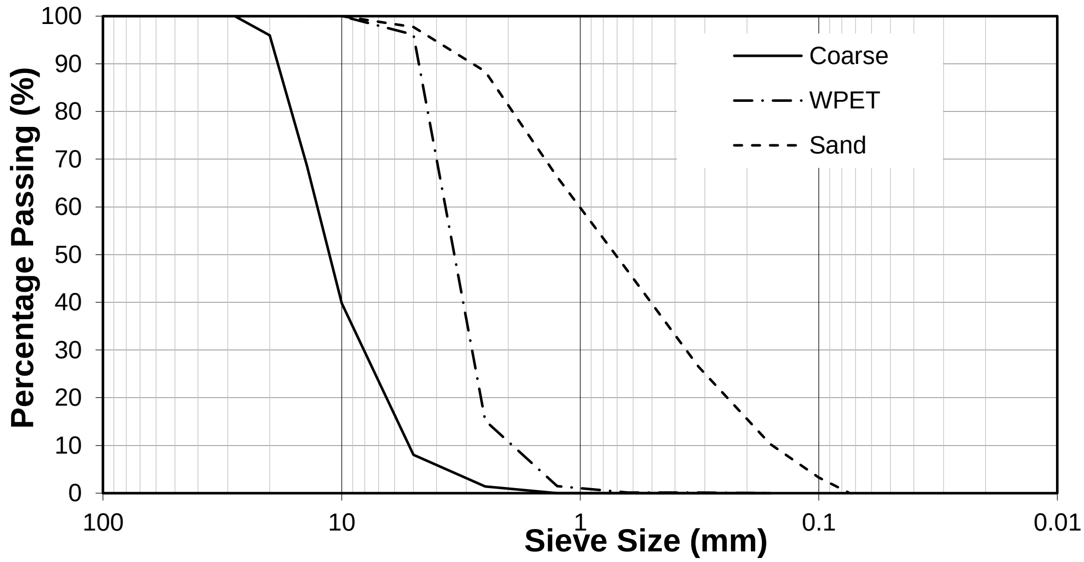


Figure 3.7: Comparative sieving analysis for aggregates.

3.3.5.3. Properties of waste PET

Table 3.8 shows the physical properties of the shredded waste PET. The apparent specific gravity was measured according to standard ASTM C127-15, bulk density according to standard ASTM C29M-12 and water absorption according to standard ASTM C128-15. The obtained density of waste PET was 1330 kg/m^3 which is larger than the density of the water. This means that is likely that the shredded waste PET will float on the water .However, as tested in the laboratory when mixing all the materials, the waste PET did not float over the water.

Table 3.8. Properties of the waste PET used in the experimental design.

Property	Value
Specific gravity (kg/m^3)	1330
Bulk density (kg/m^3)	337
Water absorption (%)	0.16

3.4. Definition of proportions

According to Myers and Montgomery (2009) the components and their ranges should be given in proportions by volume, weight or by mole fraction. In the present study, the ranges of variation of the components were selected in volume fractions. Thus, all the mixes had the restriction that the sum of the components would be 1 m^3 of concrete. For practical mixing purposes, the specific gravity of each component was used to convert volume to mass. The selection of the ranges was based on past research reviewed.

3.4.1. Cement proportion

Based on Choi et al., 2005; Choi et al.,2009; Ismail and Al-Hashmi, 2007; Albano et al., 2009; Frigione, 2010; Akcaozoglu, 2010; Saikia and de Brito, 2012; Juki, 2013; Saikia and de Brito, 2012; Ferreira et al., 2012, 50 % of the studies used a cement fraction ranging between 327 kg/m^3 and 428 kg/m$^{3.}$ However, considering the high environmental impacts caused by cement production such as the large generation of CO_2 (Mehta and Monteiro, 2014) the cement fraction was set as low as possible but still in a reasonable range according to the literature review. Thus, the minimum and maximum values were selected as 305 kg/m^3 and 365 kg/m^3, respectively.

3.4.2. w/c ratio and water proportion

Based on past research, 50% of the studies used w/c ratios ranging between 0.46 and 0.56. The selected minimum and maximum w/c ratio bounds of 0.43 and 0.55, respectively were selected for the present study. Because of the incorporation of superplasticizer, this research uses a lower bound than the past studies. The water proportion was calculated according to the water/cement ratio and the cement lower and higher bounds. The water lower and maximum values were set as 144 kg/m^3 and 177 kg/m^3, respectively.

3.4.3. Coarse aggregate proportion

Based on past research, 50% of the studies utilized coarse aggregate ranging between 960 kg/m^3 and 1510 kg/m^3. In the present study, the coarse aggregate content ranged between 1153 kg/m^3 and 1257 kg/m^3.

3.4.4. Fine aggregate proportion

Based on past studies, 50% of the studies used fine aggregate ranging from 495 kg/m to 800 kg/m3. The present study selected the range of variation ranging between 502 kg/m^3 and 653 kg/m^3, respectively.

3.4.5. Waste PET proportion

The goal of this research was to incorporate as much waste PET as possible into the concrete mixture, while maintaining the desired workability, compressive strength and splitting tensile strength. According to the results obtained in past research, the present study took into account multiple considerations: first, higher waste PET substitution significantly decreases the mechanical properties of the mixture. However, as authors such as Albano et al. (2009) suggest, up to 20% V waste incorporation moderately reduces strength and workability in the mixture. Frigione (2010) argued that a substitution of 4 % V of waste PET only decreased compressive strength and splitting tensile strength by 0.4% and 1.9%, respectively. The present study selected ranges of incorporation of waste PET from 8.4% to 17%. Another consideration was that, past research highlighted that flaky waste PET significantly decreased workability, while waste PET in pellets increased workability. In the present study, intending on keeping costs and energy consumption as low as possible, the waste PET was only shredded, and thermal processes were not employed. However, the present study incorporated a superplasticizer in order to increase the workability of the mixtures. A final consideration was that, when increasing the w/c ratio, reductions on the mechanical properties are produced. In the present study, the incorporation of superplasticizer in the mixture, allowed the use of lower w/c to achieve high strength.

3.4.6. Superplasticizer proportion

The superplasticizer dosage was selected according to the technical datasheet recommendations. However, the use of superplasticizer along with waste PET was not found in past research.

3.4.7. Summary of the proportions

Table 3.9 shows the summary of components and their proportions used in the present study. The table shows the proportions in mass for practical calculations and the proportions in volume according to the design requirements. The conversion from volume to mass fraction were performed using the specific gravity of each component.

Table 3.9: Summary of the proportions of the components in mass and volume.

Component	Specific gravity (kg/m^3)	Mass fraction (kg/m^3) Low	Mass fraction (kg/m^3) High	Volumetric fraction (m^3) Low	Volumetric fraction (m^3) High
Cement	3150	306	365	0.097	0.116
Water	1000	144	177	0.144	0.177
Coarse	2621	1153	1257	0.440	0.480
Fine	2617	506	653	0.193	0.249
Waste PET	1330	24	53	0.018	0.040
Superplasticizer	1000	3	14	0.003	0.014

3.5. Number of mixtures

The Scheffé model for mixture designs presented in Chapter 2 was used in the present study (Equation 3.1). The mixture of the present study included 6 components. The execution and development of this model comprises at least 21 experiments (Equation 3.2). The requirement for this model was a minimum of 21 experiments in order to determine 21 coefficients that generate the combination of the 6 components of the mixture. This project employed additional experiments in order to have enough data to test the adequacy of the model. Hence, three additional points (experiments) were selected to provide information about the error and lack of fit. Three replicated points were included to better understand the behavior of the data due to repeatability. Finally, four additional center points were considered to increase the power of the predictive model. In total 31 experiments (mixtures) were executed and analyzed.

$$E(y) = \sum_{i=1}^{q} \beta_i^{*} x_i \qquad (3.1)$$

$$E(y) = b_1x_1 + b_2x_2 + b_3x_3 + b_4x_4 + b_5x_5 + b_6x_6 + b_{12}x_1x_2x_5 + b_{13}x_1x_3 + b_{14}x_1x_4 + b_{15}x_1x_5 + b_{16}x_1x_6 + b_{23}x_2x_3 + b_{24}x_2x_4 + b_{25}x_2x_5 + b_{26}x_2x_6 + b_{34}x_3x_4 + b_{35}x_3x_5 + b_{36}x_3x_6 + b_{45}x_4x_5 + b_{46}x_4x_6 + b_{56}x_5x_6 \qquad (3.2)$$

3.6. Mixing procedure

The mixes were prepared in the concrete lab at Memorial University. In order to standardize and control the same mixing procedure for all the experiments, a drum with mixer capacity of 0.12 m^3 was used. A constant amount of 0.03 m^3 was prepared for each mixture. The same procedure was used for the preparation of all the mixes. The absorptivity and moisture of coarse and fine aggregates were measured before mixing, and the necessary water was added to the mixture in order to maintain the w/c ratio for each mixture. The aggregates, including waste PET, were mixed first for 30 seconds. The cement was added and mixed for another 30 seconds. The water was added and mixed for 30 seconds and finally the superplasticizer was added then and mixed for 2 minutes. The mixing was stopped for 2 minutes for absorption to take place, and then the mixing resumed for an additional minute.

Table 3.10 shows the volumetric proportions for each component as well as the additional constraints of w/c ratio, cement/aggregate ratio and, volume percentage of waste PET (WPET) used. As previously mentioned, the total sum of the components in each experiment is equal to a 1 m^3.

Table 3.10: Summary of experiments in volumetric proportions.

Run	Cement (m^3)	Water (m^3)	Coarse (m^3)	Fine (m^3)	WPET (m^3)	Superplasticizer (m^3)	w/c	agg/cem	% V WPET
1	0.116	0.157	0.478	0.207	0.028	0.014	0.43	5.0	11.9
2	0.102	0.176	0.440	0.235	0.038	0.010	0.55	5.7	13.9
3	0.097	0.153	0.470	0.247	0.028	0.004	0.50	6.3	10.2
4	0.116	0.157	0.463	0.24	0.022	0.003	0.43	5.1	8.4
5	0.107	0.165	0.465	0.222	0.033	0.008	0.49	5.4	12.9
6	0.116	0.177	0.457	0.219	0.020	0.012	0.48	4.9	8.4
7	0.116	0.177	0.480	0.202	0.022	0.003	0.48	5.0	9.8
8	0.097	0.166	0.460	0.242	0.032	0.003	0.54	6.1	11.7
9	0.107	0.165	0.465	0.222	0.033	0.008	0.49	5.4	12.9
10	0.106	0.144	0.457	0.247	0.036	0.010	0.43	5.7	12.7
11	0.106	0.175	0.480	0.196	0.029	0.014	0.52	5.4	12.9
12	0.106	0.160	0.458	0.238	0.025	0.014	0.48	5.6	9.5
13	0.097	0.168	0.464	0.229	0.028	0.014	0.55	6.1	10.9
14	0.116	0.177	0.442	0.226	0.036	0.003	0.48	4.9	13.7
15	0.107	0.165	0.465	0.222	0.033	0.008	0.49	5.4	12.9
16	0.097	0.165	0.447	0.242	0.036	0.014	0.54	6.0	12.9
17	0.103	0.177	0.456	0.239	0.022	0.003	0.55	5.7	8.4
18	0.104	0.150	0.474	0.237	0.021	0.014	0.46	5.8	8.4
19	0.106	0.175	0.480	0.196	0.029	0.014	0.52	5.4	12.9
20	0.104	0.158	0.458	0.227	0.040	0.014	0.48	5.7	15.0
21	0.114	0.164	0.440	0.236	0.032	0.014	0.46	5.1	11.9
22	0.116	0.164	0.476	0.198	0.040	0.007	0.45	5.0	16.8
23	0.106	0.144	0.48	0.229	0.038	0.003	0.43	5.7	14.2
24	0.116	0.164	0.476	0.198	0.040	0.007	0.45	5.0	16.8
25	0.116	0.177	0.452	0.202	0.040	0.014	0.48	4.8	16.5
26	0.100	0.173	0.48	0.22	0.020	0.008	0.55	5.9	8.4
27	0.107	0.165	0.465	0.222	0.033	0.008	0.49	5.4	12.9
28	0.116	0.177	0.452	0.202	0.040	0.014	0.48	4.8	16.5
29	0.097	0.157	0.48	0.213	0.040	0.013	0.51	6.1	15.8
30	0.102	0.177	0.472	0.206	0.040	0.003	0.55	5.7	16.3
31	0.104	0.155	0.452	0.246	0.040	0.003	0.47	5.7	14.0

3.7. Curing

As mentioned by Mehta and Monteiro (2014), curing highly benefits strength in the concrete. Additionally, Silva et al. (2012) observed that humid curing regimes highly benefits mixes including waste PET as an aggregate. In the present study curing of the samples was considered according to the standard ASTM C192/ C 192 M-16. The cylinders were left in the casting room for three days and then taken to the curing room at a humidity of 100% and temperature of 23 +/- 2°C until being tested.

3.8. Test procedures

3.8.1. Workability

Immediately after the mixing, slump was measured to test workability. This property was tested according to the standard ASTM C143/C 143M-15. Three measurements of slump were taken and the average of the tests was reported.

3.8.2. Compressive strength

According to the ASTM C31M-15 standard, three cylinders of 100 mm x 200 mm were prepared from every mix. Subsequently, the compressive strength test was performed according to the standard ASTM C39/C39M-18. After 28 days, the samples were taken from the curing room to be tested. The average of the strength was reported.

3.8.3. Splitting tensile strength

According to standard ASTM C31M-15, three cylinders of 100 mm x 200 mm were prepared and roded from every mix. After 28 days the samples were taken from the curing room to be tested for splitting tensile strength. The splitting tensile strength test was performed according to standard ASTM C496/C496M-17 by applying a continue load over a steel bar placed on the body of the cylinder to test the tensile strength.

CHAPTER 4

ANALYSIS OF RESULTS

In this chapter the results are presented, the responses are evaluated through ANOVA and regression analysis, and a model is fitted for each property. Additionally, the adequacy of all the models is tested before the predictive equations for each property is determined.

4.1. Results

Table 4.1 shows the average of three measurements of each property and the mass proportions of the components in each mixture. The complete information of all the samples will be included in Appendix C. In the next stage, the results of the experiments were statistically analyzed, fitted to a linear or polynomial model, checked for adequacy, and optimized.

4.1.1. Summary of the experiments

Table 4.1 shows the results of workability (slump), compressive strength, and splitting tensile for each experiment.

Table 4.1: Summary of experiments in mass fraction

Run	Cement (kg/m^3)	Water (kg/m^3)	Coarse (kg/m^3)	Fine (kg/m^3)	WPET (kg/m^3)	Superplas (kg/m^3)	w/c	a/c	% V WPET	Slump (mm)	Comp. Str (MPa)	Split. Tens (MPa)
1	365	157	1251	544	37	14	0.43	5.0	11.9	128	17.5	3.0
2	320	176	1151	615	51	10	0.55	5.7	13.9	146	15.1	2.2
3	306	153	1231	649	37	4	0.50	6.3	10.2	102	12	1.5
4	364	157	1211	630	29	3	0.43	5.1	8.4	121	27.8	3.2
5	338	165	1217	581	44	8	0.49	5.4	12.9	87	12.9	2.3
6	365	177	1195	573	26	12	0.48	4.9	8.4	154	23.6	3.1
7	365	177	1256	528	29	3	0.48	5.0	9.8	136	21.89	3.2
8	306	166	1203	635	42	3	0.54	6.1	11.7	128	8.8	1.4
9	338	165	1217	581	44	8	0.49	5.4	12.9	116	11.6	1.7
10	334	144	1196	646	48	10	0.43	5.7	12.7	67	19.3	2.7
11	335	175	1256	513	39	14	0.52	5.4	12.9	142	13.5	2.4
12	332	160	1198	624	34	14	0.48	5.6	9.5	140	12.39	2.7
13	306	168	1214	601	38	14	0.55	6.1	10.9	129	7.46	2.1
14	365	177	1155	592	48	3	0.48	4.9	13.7	140	19.05	2.7
15	338	165	1217	581	44	8	0.49	5.4	12.9	115	16.05	2.4
16	306	165	1169	633	48	14	0.54	6.0	12.9	121	10.1	1.5
17	324	177	1193	625	30	3	0.55	5.7	8.4	155	18.8	2.7
18	328	150	1241	622	28	14	0.46	5.8	8.1	164	15.5	2.8
19	335	175	1256	513	39	14	0.52	5.4	12.9	149	11.5	2.2
20	326	158	1198	594	53	14	0.48	5.7	15.0	99	14.4	2.1
21	359	164	1151	619	43	14	0.46	5.1	11.9	121	19.1	2.9
22	365	164	1244	519	53	7	0.45	5.0	16.8	51	16.6	2.9
23	334	144	1256	600	50	3	0.43	5.7	14.2	84	13.5	1.9
24	365	164	1244	519	53	7	0.45	5.0	16.8	94	17.3	2.7
25	365	177	1183	528	53	14	0.48	4.8	16.5	116	16.3	2.6
26	314	173	1256	575	26	8	0.55	5.9	8.4	154	13.5	1.6
27	338	165	1217	581	44	8	0.49	5.4	12.9	117	15.6	2.5
28	365	177	1183	528	53	14	0.48	4.8	16.5	97	15.6	2.6
29	306	157	1256	558	53	13	0.51	6.1	15.8	127	10.6	1.1
30	322	177	1234	540	53	3	0.55	5.7	16.3	103	8.0	1.5
31	328	155	1182	645	53	3	0.47	5.7	14.0	84	16.7	2.1

4.1.2. Appearance of the samples

Figure 4.1 shows the examples of the samples prepared and analyzed. The samples (a) contained a low cement content (306 kg/m^3), medium waste PET replacement (13%), and high w/c ratio (0.54). The sample (b) contained a medium cement content (332 kg/m^3) a w/c ratio of 0.48 and a low waste PET content of (9.5%). The sample (c) contained high cement content (365 kg/m^3), low waste PET replacement (8.4%), and low w/c ratio of 0.43. In figure 4.1. (a) The creation of honeycombs can be realized. Honeycombs are empty spaces left inside the concrete mass. The concrete did not reach all the space creating cavities and empty spaces. Honeycombs reduce the strength of the concrete and the structural properties.

(a) (b) (c)

Figure 4.1: Samples of different mixes.

4.1.3. Failure modes in compressive strength test

Figure 4.2 shows the common types of failures exhibited by the samples when testing for compressive strength. The most common failure observed among the samples was longitudinal.

(a) (b) (c)

Figure 4.2: Types of failures presented in the compressive strength testing: (a) longitudinal, (b) diagonal, and (c) side fractures.

4.1.4. Failure modes in splitting tensile strength test

Figure 4.3 represents the typical failure of the samples when tested for slitting tensile. The majority of the samples showed the same transversal failure in the middle of the sample.

Figure 4.3: Splitting tensile strength test failures.

4.2. Analysis of the results

4.2.1. Compressive strength

Hypothesis

$$Ho: \beta_1 = \beta_2 = \beta_3 \ldots . + \beta_k = 0 \qquad (4.1)$$

$$alternative, H_a: At\ least\ one\ is\ not\ zero.$$

Assumptions:

- The residuals follow a normal distribution
- The residuals have constant variance
- The residuals are independent and randomized

Table 4.2: Analysis of variance of compressive strength

Source	Sum of squares	Degree of freedom	Mean square	F-value	p-value prob> F
Linear vs. Mean	473.11	5	94.62	17.52	< 0.0001
Qudratic Vs. linear	112.91	15	7.53	3.40	0.0280
Sp. Cubic vs. Quadratic	5.89	4	1.47	0.55	0.7100
Residual	16.22	6	2.70		

The statistical analysis was performed on the data for compressive strength as shown in the Table 4.2. The level of significance selected for this design was 0.05. Thus, when p-value probability > F was less than 0.05 the model was significant and could explain the behavior of the data. Table 4.2 shows that he linear model presented a F-value of 17.52 and a p-value probability > F equal to < 0.0001. Thus, the null hypothesis (H_0) was rejected and the data of the experiments on compressive strength fitted a linear model. The components A (cement), B (water), C (coarse aggregate), D (fine aggregate), E (waste PET), and F (superplasticizer) significantly influenced the compressive strength. On the other hand, the quadratic model presented p-value probability > F equal to 0.028 that is lower than the significance level (0.05), but the linear model presented a lower value (<0.0001) which fitted better the data of the experiments.

Once the data was fitted in the linear model, the lack of fit test was performed. Table 4.3 shows the lack of fit test. The test obtained F- value equal to 2.31 and a p-value probability >F equal to 0.1517, which meant that the residual error did not exceed the pure error. Therefore, the model was adequate to fit the data from the experiments.

Table 4.3: Lack of fit test of compressive strength.

Source	Sum of squares	Degree of freedom	Mean square	F-value	p-value prob> F
Linear	118.80	19	6.25	2.31	0.152
Quadratic	5.89	4	1.47	0.55	0.710
Special cubic	0	0			
Pure error	16.22	6	2.70		

The adequacy of the model was evaluated through adjusted R-squared and predicted R-squared. R-squared was not considered because it indicates how well the model fit the data removing the variability proportion form the model. This indicator increases if data increases, so that it is less reliable than r-squared adjusted and predicted (Myers and Montgomery, 2009).

The adjusted R-squared represents the variation of the rest of experiments compared with the mean, while the predicted r-squared explains the accuracy on the predictions of the model (Myers and Montgomery, 2009). The evaluation of these parameters ranges between 0 and 1. While 0 indicates a poor fitting, 1 indicates an excellent fit (Myers and Montgomery, 2009).

Table 4.4: Adjusted R-squared, Predicted R-squared, and PRESS of compressive strength.

Source	Std. Dev	R-squared	Adjusted R-squared	Predicted R-squared	PRESS
Linear	2.32	0.778	0.7336	0.6530	211.01
Quadratic	1.49	0.9636	0.8909	-0.6567	995.33

Table 4.4 shows the values R-squared adjusted and predicted for both linear and quadratic models. The predicted R-squared value was 0.65 for the linear model whereas it was negative for the quadratic model. A negative predicted R-squared means that the model is no better than using the mean value. Hence the choice of the linear model is the correct choice.

Residual charts were examined in order to corroborate the linear model. Figure 4.4 shows that the linear model meets the normality, independence and randomized distribution assumptions for the experiments. In the normal probability vs. internally studentized residuals chart, the data from the experiments followed a normal distribution. In the internally studentized residuals vs. predicted values chart (Figure 4.4) the data showed that the residuals did not follow any pattern, and there is a constant variance. In the internally studentized residuals vs. run, there is no obvious patterns indicating that the experiments were properly randomized. In the predicted vs. actual chart, the points were randomly distributed along the 45 degree line, indicating areas above or under prediction of the linear model. Finally, in the Box-Cox chart the model showed there is no need for transformations.

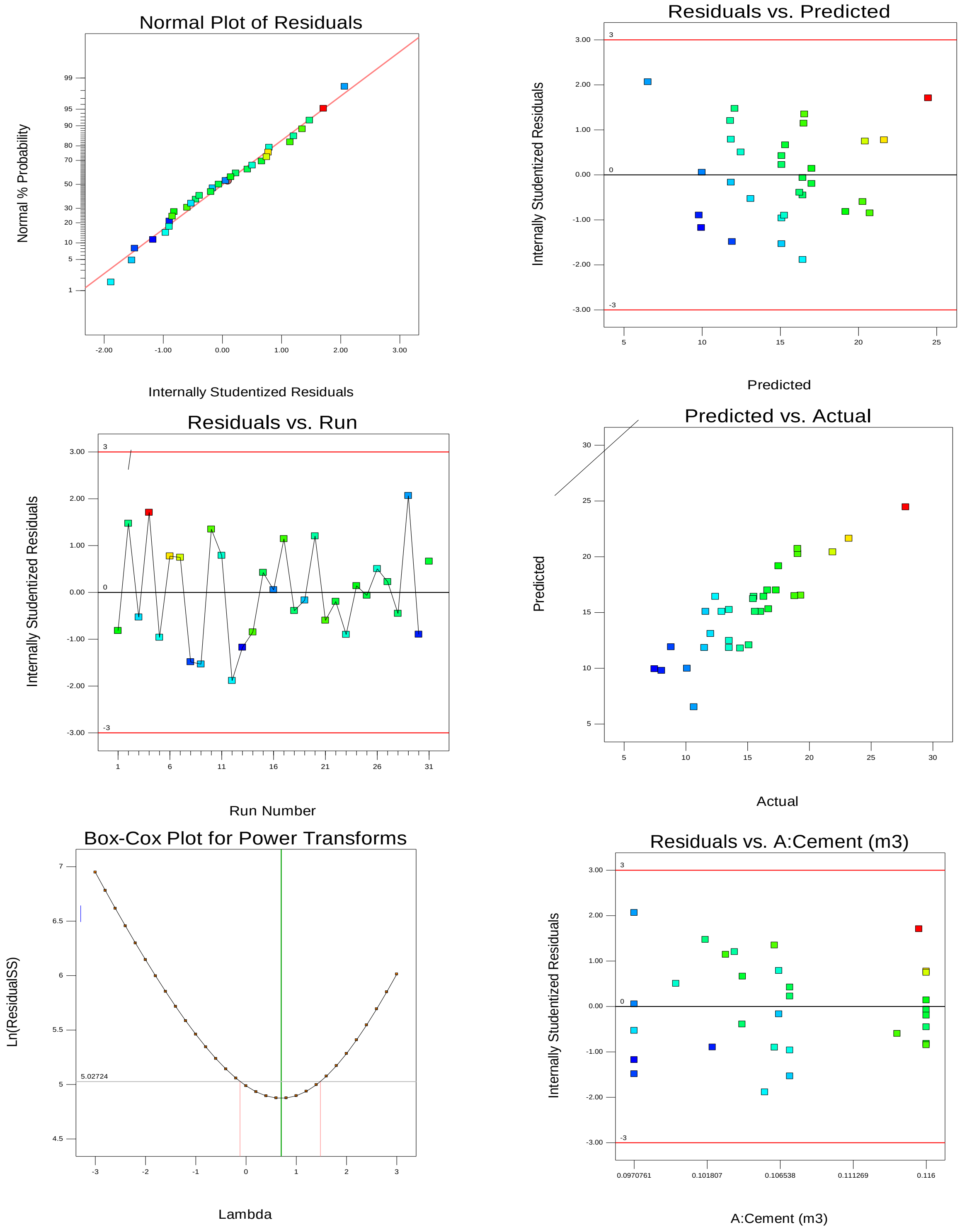


Figure 4.4: Residuals charts of the compressive strength experiments.

The linear model was established and the coefficients of the components for the linear predictive equation were defined. These coefficients represent the influence of each component on the compressive strength. Each coefficient was established as a pseudo component, and then it was transformed into an actual value. Equation 4.2 shows the linear model fitted for compressive strength considering pseudo components.

$$Compressive\ strength\ (MPa) = -23.87(A) + 22.02\ (B) + 21.45(C) + 13.54\ (D) + 33.53(E) + 29.85\ (F) \quad (4.2)$$

The components in m^3: (A) Cement, (B) Water, (C) Coarse, (D) Fine, (E) PET, and (F) Superplasticizer.

The predictive equation of the linear model in real values is shown in equation (4.3) as:

$$Compressive\ strength\ (MPa) = +535.96(A) - 72.61\ (B) - 65.16(C) + 39.76\ (D) - 225.38(E) - 176.51\ (F) \quad (4.3)$$

Analyzing the predictive equation with real values of compressive strength, the components that influence the most the compressive strength can be identified. The cement, waste PET, and superplasticizer highly influence the compressive strength. A moderate variation in one of these components will significantly impact the compressive strength outcome. The positive sign in cement content (A) indicates that increasing the cement content will increase compressive strength, whereas the negative sign in the remaining components indicates that increasing these components decrease compressive strength.

4.2.2. Slump

Hypothesis

$$H_0: \beta_1 = \beta_2 = \beta_3 \dots . + \beta_k = 0 \qquad (4.4)$$

$$alternative, H_a: At\ least\ one\ is\ not\ zero.$$

Assumptions:

- The residuals follow a normal distribution
- The residuals have a constant variance
- The residuals are independent and randomized

Table 4.5: ANOVA tests for slump.

Source	Sum of squares	Degree of freedom	Mean square	F-value	p-value prob> F
Linear vs. Mean	15659.65	5	3131.93	11.73	< 0.0001
Qudratic Vs. linear	4588.32	15	305.89	1.47	0.2734
Sp. Cubic vs. Quadratic	321.6	4	80.41	0.27	0.8848
Residual	1762.25	6	293.71		

Similar to compressive strength the model that best described slump was linear. With a F-value of 11.73 and a prob > F value equal to < 0.0001 the linear model was identified as significant for slump. The null hypothesis was rejected and the components A (cement), B (water), C (Coarse aggregate), D (Fine aggregate), E (waste PET), and F (superplasticizer) influenced the slump of the concrete. The quadratic and cubic models showed prob> F values equal to 0.2734 and 0.8848, respectively, which meant that none of them could explain and predict the slump in the concrete.

Table 4.6 shows the lack of fit test performed to slump as measured response. The obtained values in the tests were F- value of 0.88 and a prob > F of 0.62. Which meant that the lack of fit of the slump experiments was not significant.

Table 4.6: Lack of fit test performed to slump.

Source	Sum of squares	Degree of freedom	Mean square	F-value	p-value prob> F
Linear	4909.98	19	258.42	0.88	0.6206
Quadratic	321.66	4	80.41	0.27	0.8848
Special cubic	0	0			
Pure error	1762.25	6	293.71		

Table 4.7 shows the values of adjusted R-squared and predicted R-squared for slump as a linear model. The difference between adjusted and predicted R-squared was 0.12, which was a good agreement. The predicted R-squared was 0.5205, which estimates that the model is moderately strong to predict new observations.

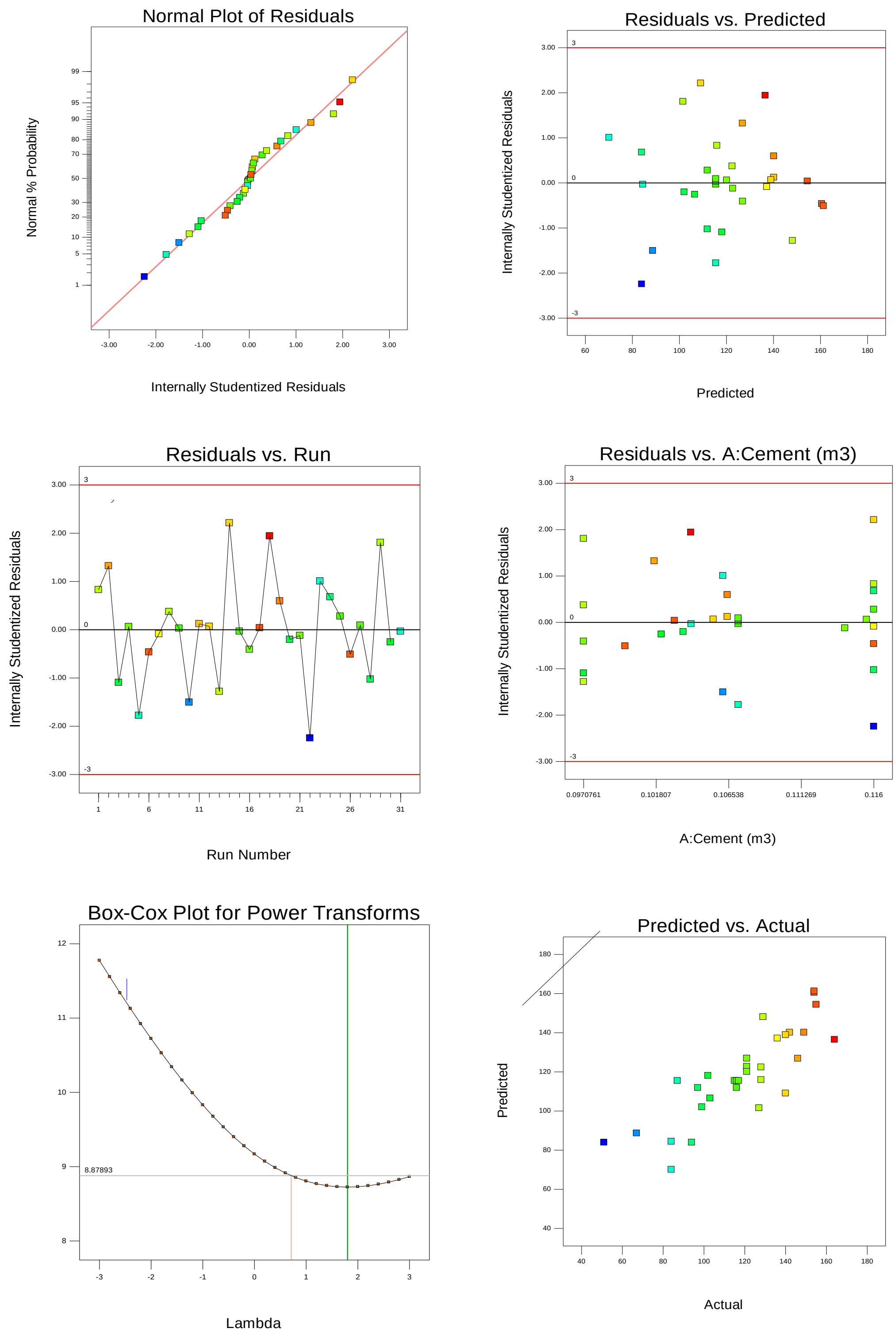


Figure 4.5: Residuals charts of slump.

Figure 4.5 shows the residuals of the experiment. The residuals were considered to be approximately normally distributed. Additionally, the residuals have constant variance, are random and independent among the experiments.

Table 4.7: R-Squared, Adjusted R-Squared, Predicted R-Squared, and PRESS tests for slump.

Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS
Linear	16.34	0.7012	0.6415	0.5205	10708.98
Quadratic	14.44	0.9067	0.7201	-2.1348	70005.97
Special Cubic	17.14	0.9211	0.6054		

The linear predictive model equation for slump in pseudo components was stated according to equation (4.5) as follows:

$$Slump(mm) = +178.59\,(A) + 22.84\,(B) + 122.01(C) + 105.96\,(D) + 299.65(E) - 1.75\,(F) \qquad (4.5)$$

Considering the components: (A) Cement, (B) Water, (C) Coarse, (D) Fine, (E) PET, and (F) Superplasticizer.

The predictive linear equation of the slump in actual values was:

$Slump\ (mm) =$

$$-755.04(A) + 1310.60\ (B) - \ 4.61(C) + \ 208.15(D) - 2360.66(E) + 1636.75\ (F)$$

(4.6)

According to the predictive model for slump in real values (Equation 4.6). The most significant components were waste PET, superplasticizer, and water. The addition of waste PET considerably decreased the slump, while the addition of superplasticizer and water increased the slump. The positive sign in water (B) and superplasticizer (E) indicates the compressive strength will increase if increasing the content of these components, whereas the negative sign in the remaining components indicates that increasing these components decrease compressive strength.

4.2.3. Splitting tensile strength

Hypothesis

$$H_0: \beta_1 = \ \beta_2 = \beta_3 \ldots . + \beta_k = 0 \qquad (4.7)$$

$$alternative, H_a: At\ least\ one\ is\ not\ zero.$$

Assumptions:

The residuals follow a normal distribution

The residuals have a constant variance

The residuals are independent and randomized

As shown in Table 4.8 the model that fitted the splitting tensile strength was again a linear model. With a F-value of 23.28 and a prob> F value equal to < 0.0001 the linear model was appropriate for splitting tensile strength. The null hypothesis (H_0) was rejected. The components A (cement), B (water), C (Coarse aggregate), D (Fine aggregate), E (waste PET), and F (superplasticizer) were included in the linear model.

Table 4.8: ANOVA of splitting tensile experiments.

Source	Sum of squares	Degree of freedom	Mean square	F-value	p-value prob> F
Linear vs. Mean	8397.11	5	1679.42	23.28	< 0.0001
Qudratic Vs. linear	1055.74	15	70.38	0.94	0.5563
Sp. Cubic vs. Quadratic	364.04	4	91.01	1.42	0.3323
Residual	383.56	6	63.93		

The quadratic and cubic models showed prob> F values equal to 0.5563 and 0.3323, respectively. Which indicated that they did not describe properly the splitting tensile of the specimens.

Table 4.9: Lack of fit test for splitting tensile.

Source	Sum of squares	Degree of freedom	Mean square	F-value	p-value prob> F
Linear	1419.78	19	74.73	1.17	0.45
Quadratic	364.04	4	91.01	1.42	0.33
Special cubic	0	0			
Pure error	383.56	6	63.93		

The analysis of the lack of fit test (Table 4.9) showed a F- value of 1.17 and a p-value >F equals to 0.45. These values demonstrated that the linear model for splitting tensile strength did not present a lack of fit. The values of adjusted R-squared and predicted R-squared for splitting tensile are shown in Table 4.10. The adjusted r-squared and predicted r-squared obtained for splitting tensile were higher compared to slump and compressive strength. The difference between adjusted and predicted R-squared was 0.06, which is a very good agreement. The predicted R-squared was 0.7285, which means that the predictive model is adequate for predicting new observations.

Table 4.10: R-Squared, Adjusted R-Squared, Predicted R-Squared, and PRESS for splitting tensile.

Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS
Linear	8.49	0.8232	0.7879	0.7284	2770.66
Quadratic	8.65	0.9267	0.7801	-3.1611	42445.45
Special Cubic	8.00	0.9624	0.8120		

Figure 4.6 demonstrated a normal distribution of the residuals of the splitting tensile experiments, constant variance, random and independent among the residuals of the model.

Finally, the predictive linear model for splitting tensile strength expressed by pseudo components is:

$$Splitting\ tensile\ (MPa) = -2.86\ (A) + \ 3.19\ (B) + \ 3.21(C) + \ 2.52\ (D) + 4.96(E) + 1.70\ (F) \qquad (4.8)$$

Considering the components: (A) Cement, (B) Water, (C) Coarse, (D) Fine, (E) PET, and (F) Superplasticizer.

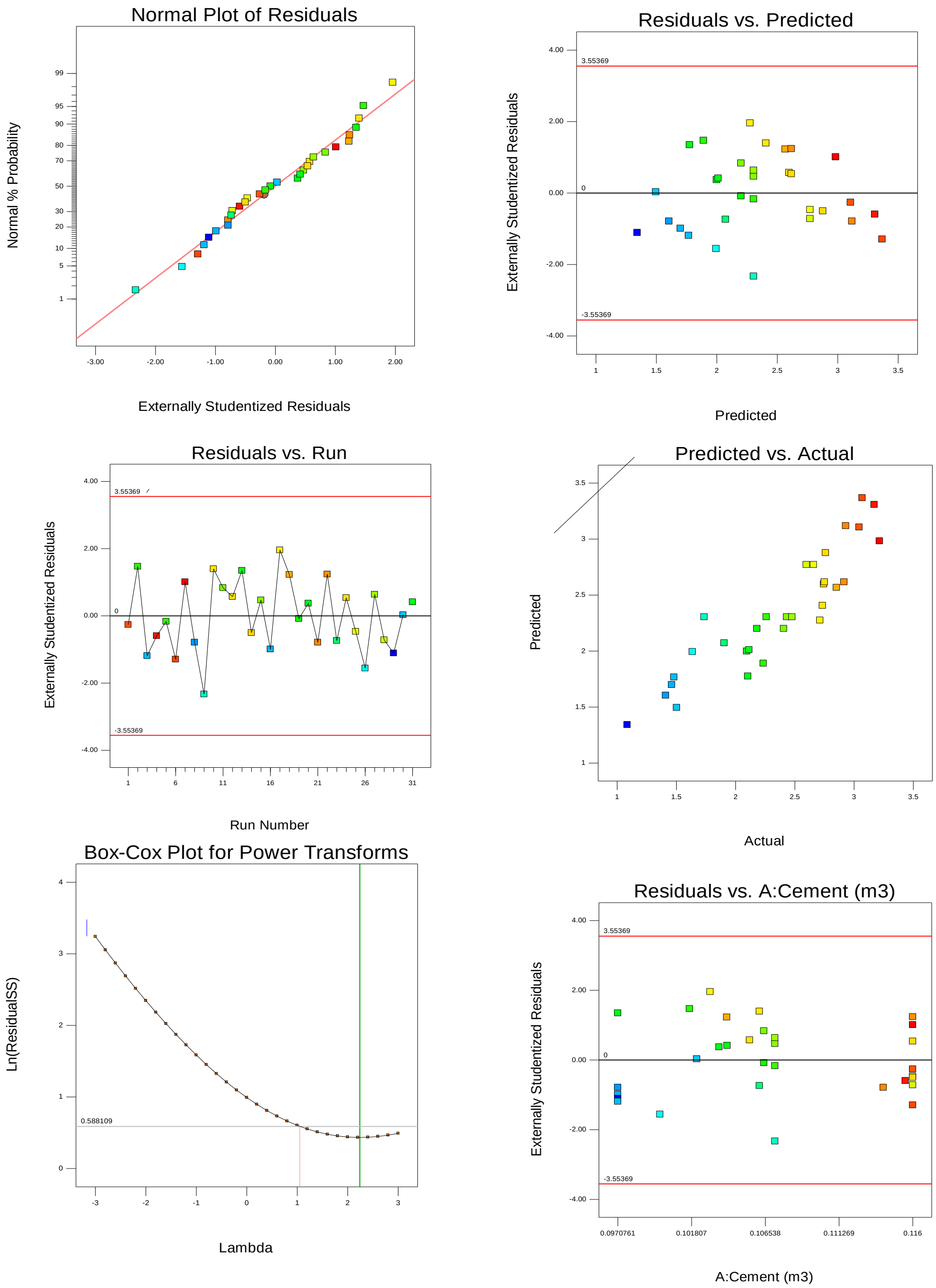


Figure 4.6: Residuals charts of the splitting tensile experiments.

The splitting tensile strength predictive equation in real values is:

$$Splitting\ tensile\ (MPa) = +72.59\ (A) - 7.52\ (B) - 7.78(C) +\ 1.37(D) - 31.06(E) + 12.2\ (F) \qquad (4.9)$$

The most influential components in the model are cement, waste PET, and superplasticizer. The cement content significantly increases splitting tensile, the waste PET reduces slump, and superplasticizer increases the splitting tensile strength.

Table 4.11: Summary of the predictive models.

Property	Predictive model (real values)	R-squared adjusted	R-squared predicted
Compressive strength	$\text{Comp. strength (MPa)} = +535.96(\text{A}) - 72.61\ (\text{B}) - 65.16(\text{C}) + 39.76\ (\text{D}) - 225.38(\text{E}) - 176.51\ (\text{F})$	0.73	0.65
Slump	$\text{Slump (mm)} = -755.04(\text{A}) + 1310.60\ (\text{B}) - 4.61(\text{C}) + 208.15(\text{D}) - 2360.66(\text{E}) + 1636.75\ (\text{F})$	0.64	0.52
Splitting tensile strength	$\text{Splitting tensile (MPa)} = +72.59\ (\text{A}) - 7.52\ (\text{B}) - 7.78(\text{C}) + 1.37(\text{D}) - 31.06(\text{E}) + 12.2\ (\text{F})$	0.79	0.73

CHAPTER 5

OPTIMIZATION

This chapter covers the graphical analysis and the numerical optimization of the previously reported experimental results. It also discusses the amount of natural aggregate saved due to its replacement with waste PET into concrete and the quantity of used bottles in the process.

The graphical optimization method focused on trace and contour plots. Trace plots highlighted the influence of each components in the measured response, while contour plots only show the influence of three components in the responses (Smith, 2005). Hence, for mixtures containing more than four components, the numerical optimization is more suitable than the graphical one (Anderson and Whitcomb, 2005). In this study the graphical method was used to interpret and analyze the main influences of each component on the different responses. Subsequently, the numerical optimization was used to obtain the optimal proportions for all the components using the desirability function approach (Anderson and Whitcomb, 2005). Via this tool, a set of optimized combinations of all the components was obtained. The optimized combinations were tested in the lab and compared with the predictions from the models. These values should be within the prediction intervals.

5.1 Graphical analysis

Usually, trace plots are first employed to screen the components with the highest influence in the response (Smith, 2005). Then, contour plots are utilized to show the situations with minimal or maximal responses using the most influential components (Myers and Montgomery, 2009).

5.1.1. Trace plots

Trace plots consist of lines that represent each component of the mixture. The effect of each component as shown is the slope of each line (Simon et al., 1999). Trace plots use pseudo components and pseudo-coding, which employ inverted values. Thus, in trace plots, a positive slope represents a negative effect (Smith, 2005). Components represented by horizontal lines are assumed to have no influence on the response of the experiment, while components represented by vertical lines have a strong influence over the response (Simon et al., 1999).

5.1.1.1. Compressive strength

Figure 5.1 shows the trace (Cox) plot for the compressive strength. As expected, cement content (A) has the most significant influence in the compressive strength. Increasing the cement content produces an increase in the compressive strength. Waste PET (E) also has a large influence on the compressive strength. Contrary to cement, increasing waste PET (E) decreases the compressive strength. Additionally, superplasticizer (F) and water (B) also negatively influence the compressive strength. Increasing the amount of superplasticizer or water, decreases the compressive strength. Components such as coarse aggregate (C) and fine aggregate (D) had moderated influence on the compressive strength.

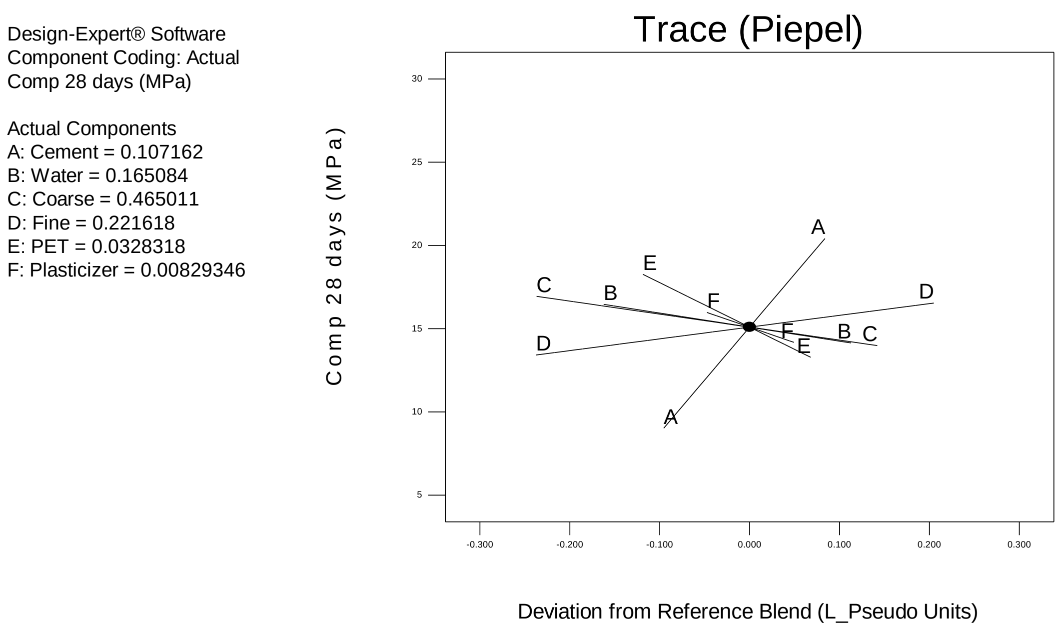


Figure 5.1. Trace plot of compressive strength.

5.1.1.2. Slump

Figure 5.2 shows the trace (Cox) plot for slump. As can be noted, the waste PET (E) has the largest influence in the slump. Increasing waste PET (E), decreases the slump. Additionally, increasing the water (A) and superplasticizer (F) increase slump. Cement content (A), coarse aggregate (C), and fine aggregate (D) had moderate influence on the slump of the experiments.

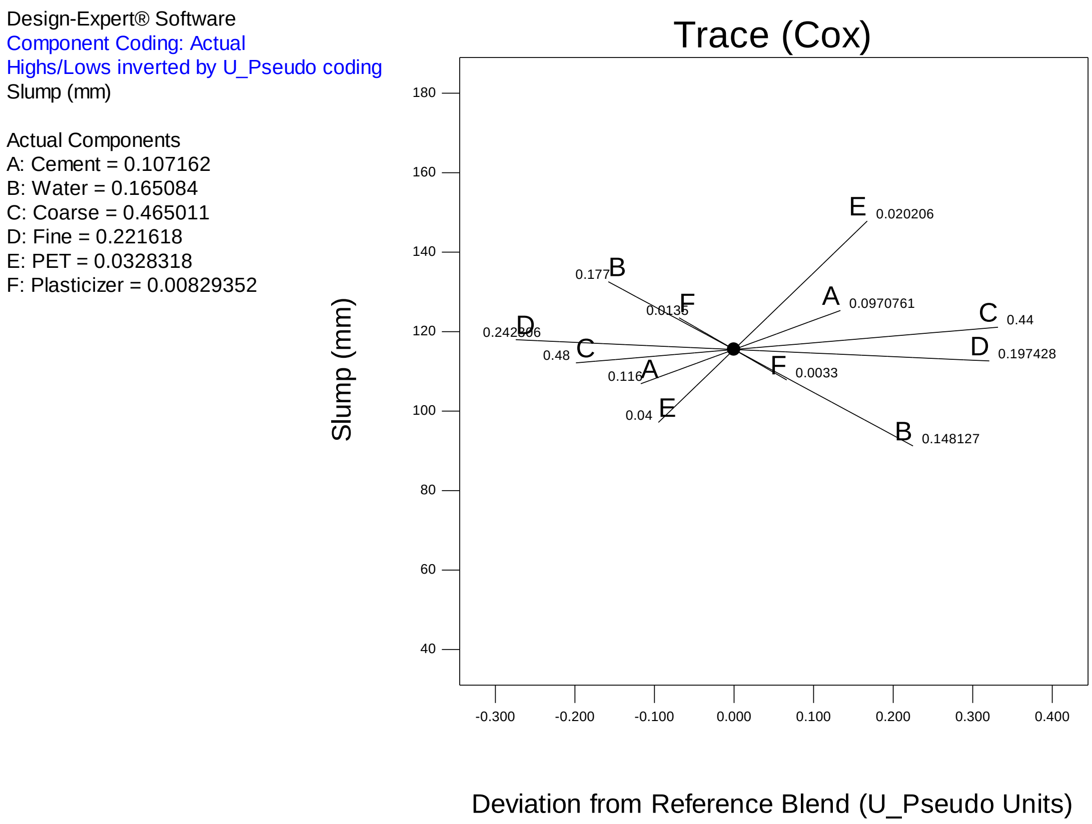


Figure 5.2. Trace plot of slump.

5.1.1.3. Splitting tensile strength

Figure 5.3 shows the splitting tensile strength trace (Cox) plot. As expected, the cement content (A) exerts a strong influence on the splitting tensile. Increasing the cement content, results in a splitting tensile increase. The incorporation of waste PET also influences the splitting tensile strength. Increasing waste PET content, results in a reduction on the splitting tensile strength. Finally, increasing the water content (B) and coarse aggregate (C) decreases the splitting tensile strength.

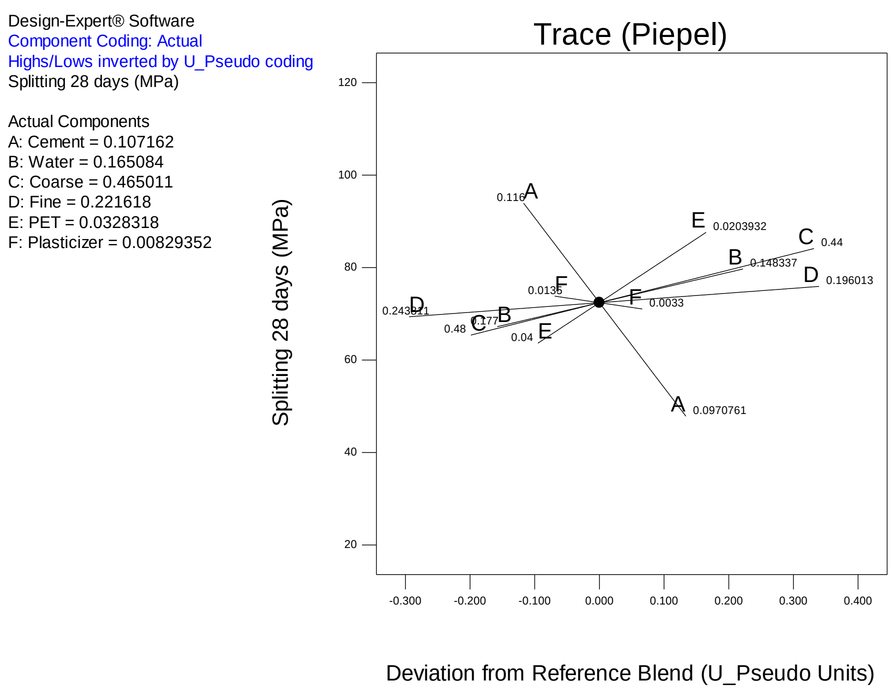


Figure 5.3. Trace plot of splitting tensile strength.

5.1.2.Overlay plots

Contour plots, particularly overlay plots, use only three components at a time. These plots are used to examine the combinations of components that allow the maximum or minimum values in the response. The remaining components that do not cause a strong influence in the response remain fixed (Simon et al., 1999). However, when the mixture has more than four components, many trial mixtures are needed to achieve the optimization. Therefore, this method is not recommended for mixtures with more than four components (Smith, 2005).

5.1.2.1. Overlay option 1

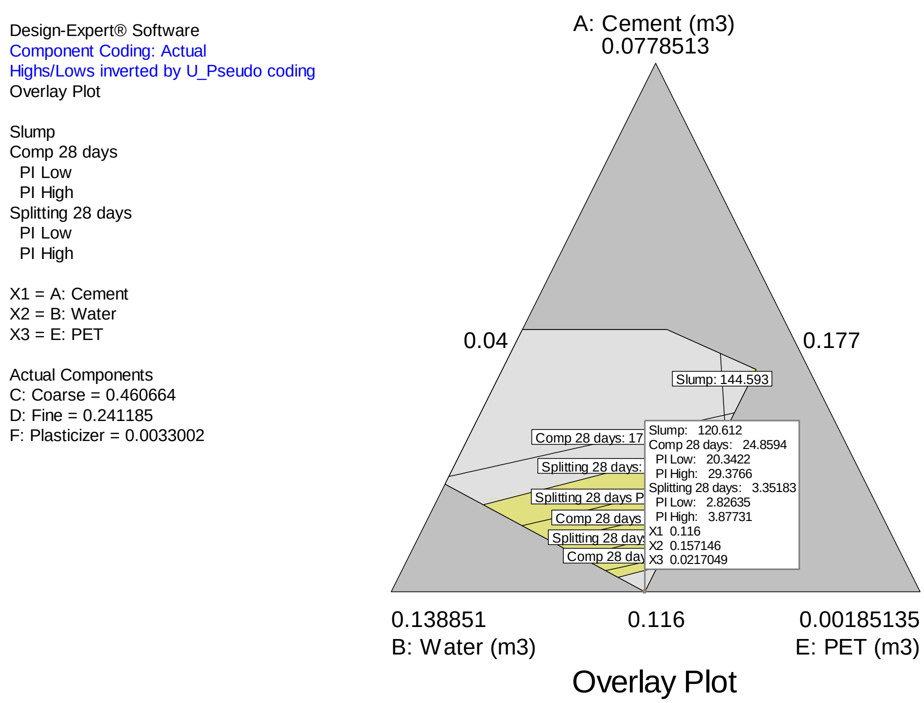


Figure 5.4. Overlay plot 1.

Figure 5.4 shows a combination of cement, water, and waste PET that obtained the desired properties as follows: compressive strength of 24.86 MPa, a splitting tensile of 3.35 MPa and a slump of 120.6 mm. The components should be set at: (1) Cement: 0.116 m^3, 365 kg; (2) water: 0.1557 m^3, 155.7 kg; and (3) waste PET: 0.021 m^3, 27.9 kg/m^3. The remaining components were kept fixed in: fine aggregate: 0.24 m^3, 628 kg/m^3 (D), coarse aggregate (C): 0.46 m^3, 1206 kg/m^3 and superplasticizer (F): 0.003 m^3, 3 kg/m^3.

5.1.2.2. Overlay option 2

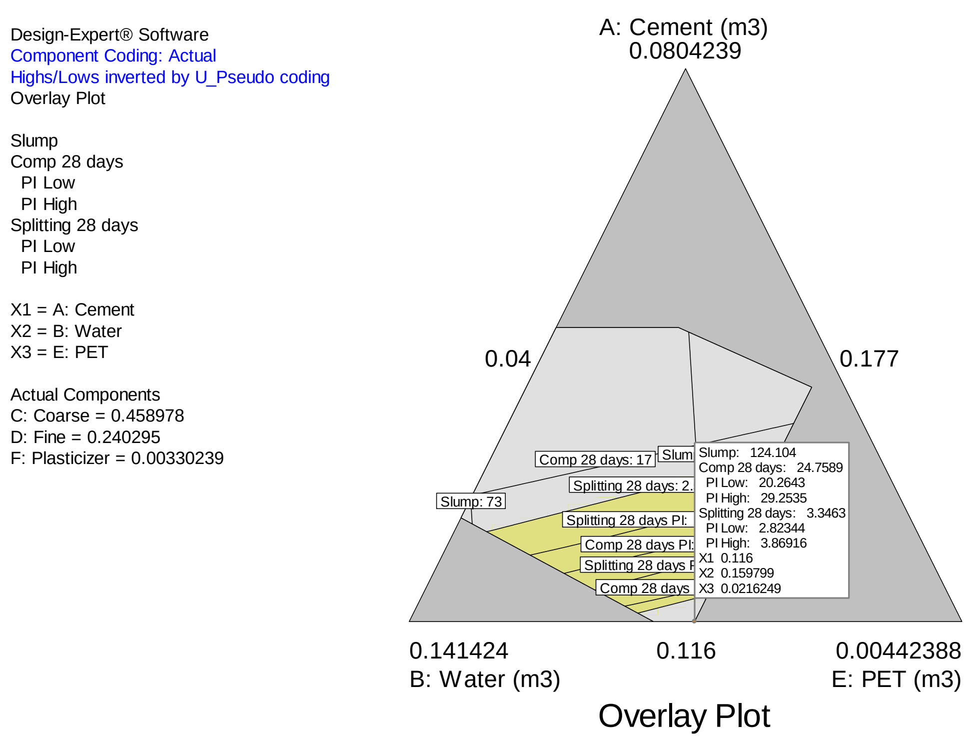


Figure 5.5 Overlay plot 2.

Figure 5.5 shows a second combination of the components cement (A), water (B), and waste PET (E) that obtains a compressive strength 24.76 MPa, slump 124 mm, and splitting tensile 3.34 MPa. The modified components are: (1) cement 0.116 m^3 (365 kg/ m^3), (2) water 0.157 m^3 (157 kg/ m^3), and (3) waste PET 0.021 m^3 (27.93 kg/ m^3). The remaining components were kept fixed in: fine aggregate: 0.24 m^3, 628 kg/ m^3 (D), coarse aggregate (C): 0.46 m^3, 1206 kg/ m^3 and superplasticizer (F): 0.003 m^3, 3 kg/ m^3.

5.2. Numerical optimization

In the numerical optimization (Figure 5.6), using the desirability function approach, the components or properties can be optimized. As mentioned in Chapter 2, the desirability function allows the experimenter to maximize, minimize or keep within target of the goals (Anderson and Whitcomb, 2005). The importance of each goal can also be established. By setting a higher importance, the goal is prioritized over the other goals (Anderson and Whitcomb, 2005). Once the goals and the importance are selected, the desirability function analyses all the possible sets of components that achieve the goals, and ranks them from 0 to 1(Simon et al., 1999). This is used to arrange the options according to their desirability (Anderson and Whitcomb, 2009). Finally, the experimenter tests the predicted combination and verifies the results against the prediction intervals (Anderson and Whitcomb, 2009).

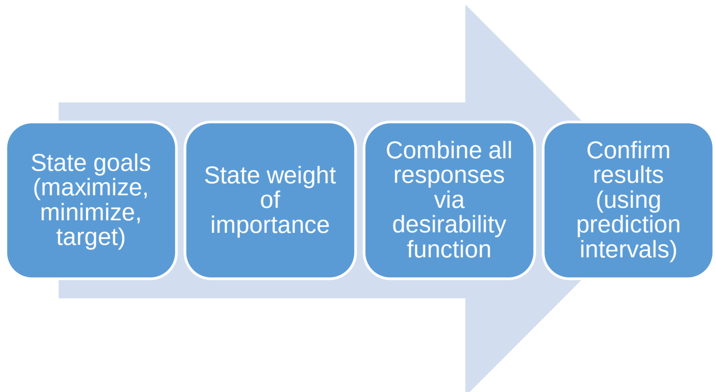


Figure 5.6. Numerical optimization process, adapted from Anderson and Whitcomb, 2009.

The desirability function (D) transforms each response into an individual desirability function d_i. If the response falls into the desired ranges the function converts the response into 1 (Anderson and Whitcomb, 2009). On the contrary, if the response does not fall into the desired ranges, it will be converted to 0. The overall desirability combines the goals for all the responses, as shown in equation (5.1) (Anderson and Whitcomb, 2009).

$$D = (d_1 d_2 d_3 d_z)^{\frac{1}{n}} \qquad (5.1)$$

If different ratings of importance are designated for the goals, the desirability function will follow equation (5.2) (Anderson and Whitcomb, 2009).

$$\boldsymbol{D} = (\boldsymbol{d_1^{r_1}} \times \boldsymbol{d_2^{r_2}} \times \boldsymbol{d_3^{r_3}} \times \boldsymbol{d_n^{r_n}})^{\frac{1}{\sum r}} = \left[\prod_{i=1}^{n} \boldsymbol{d_i^{r_i}}\right]^{\frac{1}{\sum r}} \qquad (5.2)$$

5.2.1. Optimization goals

As stated in Chapter 2, the main goal for this thesis was to achieve the minimum required workability and compressive strength for its applicability in various settings. The ranges of slump for different usages are shown in Table 5.1. The minimum compressive strength requirements are shown in Table 5.2.

It is important to note that the numerical optimization was attempted to maximize the incorporation of waste PET. However, the maximum compressive strength values reached in that case would be 20 MPa, which merely reach one of the possible applicability options. Thus, the waste PET was maintained in range instead of maximized.

Table 5.1. Typical workability values. Modified from Mehta (2014) and Beall (2001).

Examples	Minimum slump (mm)	Maximum slump (mm)
Foundation basements, walls, slabs. Not exposed to weather	25	127
Foundation basements, walls. Exterior vertical concrete. Exposed to weather. Not exposed to water accumulation	76	127
Driveways, garage slabs, walks, patios.	76	127

Table 5.2. Typical compressive strength requirements concrete. Modified from Beall (2001).

Exposure class	Examples	Minimum requirement (MPa)
F0	Basement and foundation walls and slabs, not exposed to freezing temperatures. Structures or members inside buildings such as garages, sidewalks and steps. Members buried in the soil bellow frost line.	17
F1	Members not exposed to ice or snow accumulation such as exterior walls, beams, and slabs.	21
F2	Members exposed to snow accumulation. Foundation or basement walls that will support snow accumulation.	24.5

5.2.2. Importance of goals

As shown in Table 5.3, the compressive strength was set with a higher importance than that of the remaining properties. The latter were maintained at the default importance of (3). The components and the properties were set as shown in Table 5.3.

Table 5.3. Importance of components and properties.

Component/ Response	Importance	Goal	Lower limit	Upper limit
(A) Cement (m^3)	3	in range	0.097	0.12
(B) Water (m^3)	3	in range	0.14	0.18
(C) Coarse (m^3)	3	in range	0.44	0.48
(D) Fine (m^3)	3	in range	0.19	0.25
(E) PET (m^3)	3	in range	0.018	0.04
(F) Superplasticizer (m^3)	3	in range	0.0033	0.014
Compressive strength (MPa)	4	Maximize	7.46	27.8
Slump (mm)	3	in range	73	127
Splitting tensile (MPa)	3	Maximize	1.08	3.21

5.2.3. Proposed solutions

On the basis of the previously mentioned goals, the desirability function proposed a set of optimized options. Four options with higher desirability were selected from the list and further tested. Some components such as cement, waste PET, and superplasticizer content produced a strong influence over the compressive strength. Thus, their proportions were very similar in all the optimization options. Components such as water, coarse aggregate, and fine aggregate had slight variations in the optimized options. Table 5.4 shows the combination of components proposed by the desirability function to be the most convenient to reach the desired properties.

Table 5.4. Optimization options ranked based on the desirability function.

Number	Cement (m^3)	Water (m^3)	Coarse (m^3)	Fine (m^3)	PET (m^3)	Plasticizer (m^3)	Slump (mm)	Comp. Str (MPa)	Splitting (MPa)	Desirability
1	0.116	0.157	0.461	0.241	0.022	0.003	120.6	24.8	3.3	0.915
2	0.116	0.160	0.459	0.240	0.022	0.003	124.1	24.7	3.3	0.912
3	0.116	0.163	0.456	0.239	0.022	0.003	126.9	24.5	3.3	0.905
4	0.116	0.157	0.458	0.241	0.024	0.003	115.2	24.5	3.3	0.903

5.2.4. Verification

According to the combinations proposed by the numerical optimization, the mixes were tested in the lab and the results were compared with the prediction intervals for all the selected options. The tests were performed in the same fashion as the 31 previous mixes.

5.2.4.1. Verification option 1

Table 5.5. Optimization option N. 1. Components.

Component/ Response	Value m3	Value mass kg/m3	Lower limit	Upper limit
(A) Cement (m3)	0.116	365	0.120	0.0970
(B) Water (m3)	0.157	157	0.180	0.1400
(C) Coarse (m3)	0.461	1209	0.480	0.4400
(D) Fine (m3)	0.241	631	0.250	0.1900
(E) PET (m3)	0.022	29	0.040	0.0180
(F) Superplasticizer (m3)	0.003	3	0.014	0.0033

Table 5.6. Optimization option N. 1. Predicted results Vs. Experimental results.

Response	Predicted value	Experimental value	Standard deviation	95% Prediction interval	
				Lower limit	Upper limit
Slump (mm)	120.614	123.0	16.3367	82.33	158.9
Compressive strength (MPa)	24.859	23.8	2.32397	19.41	30.31
Splitting tensile (MPa)	3.352	3.3	0.270345	2.72	3.99

The prediction in option 1 had good agreement with the laboratory tests. The experimental values of the properties fell into the 95% prediction interval. The values of slump (123 mm) and compressive strength (23.8 MPa) are suitable values for the concrete utilization.

5.2.4.2. Verification option 2

Table 5.7. Optimization option N. 2. Components.

Component/ Response	Value m3	Value mass kg/m3	Lower limit	Upper limit
(A) Cement (m3)	0.116	365	0.120	0.0970
(B) Water (m3)	0.160	160	0.180	0.1400
(C) Coarse (m3)	0.459	1203	0.480	0.4400
(D) Fine (m3)	0.240	628	0.250	0.1900
(E) PET (m3)	0.022	29	0.040	0.0180
(F) Superplasticizer (m3)	0.003	3	0.014	0.0033

Table 5.8. Optimization option N. 2. Predicted results Vs. Experimental results.

Response	Predicted value	Experimental value	Standard deviation	95% Prediction interval	
				Lower limit	Upper limit
Slump (mm)	124.10	132.0	16.3367	86.01	162.2
Compressive strength (MPa)	24.75	20.10	2.3239	19.34	30.18
Splitting tensile (MPa)	3.35	3.28	0.2703	2.72	3.98

In option 2, the prediction was also a good match with the experimental values of all the responses. All the experimental values fell within the 95% prediction interval. The compressive strength (20.1 MPa) and slump (132 mm) obtained values indicate that this mix can be used in the exposure class 0.

5.2.4.3. Verification option 3

Table 5.9. Optimization option N. 3. Components.

Component/ Response	Value m3	Value mass kg/m3	Lower limit	Upper limit
(A) Cement (m3)	0.116	365	0.120	0.0970
(B) Water (m3)	0.163	163	0.180	0.1400
(C) Coarse (m3)	0.456	1195	0.480	0.4400
(D) Fine (m3)	0.239	625	0.250	0.1900
(E) PET (m3)	0.022	29	0.040	0.0180
(F) Superplasticizer (m3)	0.003	3	0.014	0.0033

Table 5.10. Optimization option N. 3. Predicted results vs. experimental results.

Response	Predicted value	Experimental value	Standard deviation	95% Prediction interval	
				Lower limit	Upper limit
Slump (mm)	126.99	154.0	16.3367	89.14	164.86
Compressive strength (MPa)	24.52	18.9	2.3239	19.14	29.91
Splitting tensile (MPa)	3.32	2.82	0.2703	2.70	3.95

The experimental values of slump and splitting tensile strength in option 3 fell within the prediction interval. However, the value of compressive strength was lower than the lower limit of the prediction interval. Because the predictive model has a predictive r-square of 0.65 this result was not surprising.

5.2.4.4. Verification option 4

Table 5.11. Optimization option N. 4. Components.

Component/ Response	Value m3	Value mass kg/m3	Lower limit	Upper limit
(A) Cement (m3)	0.116	365	0.120	0.0970
(B) Water (m3)	0.157	157	0.180	0.1400
(C) Coarse (m3)	0.464	1216	0.480	0.4400
(D) Fine (m3)	0.238	623	0.250	0.1900
(E) PET (m3)	0.021	28	0.040	0.0180
(F) Superplasticizer (m3)	0.003	3	0.014	0.0033

Table 5.12. Optimization option N. 4. Predicted results vs. experimental results.

Response	Predicted value	Experimental value	Standard deviation	95% Prediction interval	
				Lower limit	Upper limit
Slump (mm)	115.16	131.0	16.3367	103.24	155.62
Compressive strength (MPa)	24.48	25.6	2.3239	20.88	28.33
Splitting tensile (MPa)	3.29	3.31	0.2703	2.90	3.77

Option 4 had good agreement between the predicted values and the experimental values. All the values fell within the 95% prediction interval.

Based on the optimization goals, the option that fulfils both, the workability and compressive strength requirements is option 1. It is important to note that option 4 obtained a higher compressive strength but did not satisfied the workability requirements. Thus, option 1 was selected as the most suitable combination.

5.3. Other experimental results

In order to check if the manual mixing method produces a better results, mixes 1, 2, and 4 were tested for compressive strength with the manual method. The results of this experimentation (Table 5.13) showed a substantial difference between the outcomes when using the manual method and the drum mixer. This means that potentially higher substitution of waste PET and lower cement contents can be tested along with the mixing method and mixing parameters.

Table 5.13: Comparison between compressive strength obtained manual and drum method of mixing.

Method	Option 1	Option 2	Option 4
Drum	23.8 MPa	20.1 MPa	25.6 MPa
Manual	37.3 MPa	34.7 MPa	38.5 MPa

5.4. Savings in natural aggregate utilization and bottles recycled

As an additional benefit achieved by this thesis was the preservation of natural aggregates and the recycling of waste PET. The preservation of natural aggregates was accomplished by the substitution of 58 kg/m^3 of sand by 29 kg of waste PET. Additionally, when substituting this natural aggregate 3755 bottles of waste PET can be recycled (3414 Type 1 bottles, 195 Type 2 bottles, and 146 Type 3 bottles).

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

6.1. Conclusions

- The statistical mixture design approach was shown to be a useful and practical tool for the examination of the influences among components in mixtures. For this study, 31 experiments were conducted to combine six different components and the responses of slump, compressive strength, and splitting tensile strength were statistically analyzed. Linear models fitted all the studied properties (measured responses). The adequacy of the model and the residuals were analysed. The adjusted R-squared and predicted R-squared for all the models were: (1) compressive strength: 0.73 and 0.65 (2) slump: 0.64 and 0.52 (3) splitting tensile strength: 0.79 and 0.73, respectively. These values suggested a high chance of appropriate prediction of new observations. Finally, through numerical optimization, the project goals were set and the optimized combinations of components were found. Four combinations were subsequently tested in the laboratory and compared to the predicted values.
- The incorporation of waste PET into concrete was tested in this study as follows: (1) the waste PET selected was a mixture of waste PET bottles of different thicknesses; (2) waste PET was shredded into flaky particles

ranging from 0.6 mm to 4.75 mm and waste PET was partially substituted for natural fine aggregate ranging from 8.4% to 16.8%. After optimization, the proportion of waste PET that reached the required properties was established to be 8.4% V (29 kg/m^3). This amount of waste PET allows the recycling of 3755 bottles of waste PET can be recycled (3414 bottles type 1, 195 bottles type 2, and 146 bottles type 3).

- The optimized combination with highest compressive strength was: (1) cement= 0.116 m^3, 365 kg/m^3 (2) water=0.157 m^3, 157 kg/ m^3 (3) coarse aggregate= 0.461 m^3 1209 kg/m^3 (4) fine aggregate= 0.241 m3, 631 kg/m^3 and (5) waste PET= 0.022 m3, 29 kg/m^3 The response values reached by this combination was: (1) Compressive strength 23.8 MPa; (2) slump 123 mm; and (3) splitting tensile of 3.33 MPa. This mix can be used in basements foundation walls or slabs, inside buildings not exposed to freezing temperatures.
- The optimized incorporation of waste PET into concrete also allowed the preservation of the fine natural aggregate. The amount of fine natural aggregate (sand) replaced by 29 kg of waste PET was 8.4%, 58 kg/m^3.
- Although the incorporation of waste PET into concrete reached the standards for the properties proposed in this study, additional tests, such as chloride penetration and column leach test, should be tested to determine the feasibility of the use of waste PET as a fine aggregate substitute.

- The shredding machine used in this study was built at Memorial University of Newfoundland based on a design by the Precious Plastic organization. The operation of the machine was simple and high amounts of waste PET were successfully processed.
- The volume of the waste PET bottles reduced drastically after shredding. This reduction was (1) 29 fold for bottles of 500ml and 0.1 mm of thickness (2) 23 fold for bottles of 2000 ml and 0.25 mm of thickness, and 10 fold for bottles of 591 ml and 0.4 mm of thickness.
- The components with the largest influence on the responses were cement, waste PET, and superplasticizer. Based on the predictive equations, trace, and contour plots, high cement contents had a positive influence on compressive strength and splitting tensile strength, as expected and high waste PET contents decreased the measured properties. Finally, high superplasticizer contents had a large positive influence on slump of the mixtures.
- The superplasticizer showed positive performance in the experiments. The slump values ranged from 51 to 164. Additionally, the incorporation of superplasticizer allowed the use of lower w/c ratios, which increased compressive strength and splitting tensile strength in the mixtures.
- Suitable values of compressive strength, slump, and splitting tensile strength were reached with the use of the highest cement content for this

study. Unfortunately, low cement contents combined with waste PET led to a substantial decrease in the desired properties.

- Results that showed samples with high w/c ratios, high waste PET contents, and low cement content, largely increased the porosity of hardened concrete. After these sample dried, the remaining water that did not react with the cement was not absorbed by the waste PET particles producing cavities. Once the samples were stripped off and the water was released, the cavities became empty, increasing the porosity and largely decreasing compressive strength and splitting tensile strength.
- Segregation was observed in some samples. These samples were characterized as having high w/c ratio, low cement content, and high waste PET incorporation. However, the segregation can also be associated with the moderate gradation of waste PET. Size distribution of waste PET presented deficiency in particles ranging from 0.075 mm to 1 mm and excess in particles ranging from 1 mm to 4.75 mm.
- The numerical method for optimization showed to be the most suitable approach for mixtures containing six different components. The graphical method showed similar values as the numerical method, however the numerical method was more straightforward and involved all the studied components.

6.2. Recommendations

- This study assumed that the responses might follow quadratic models. Thus, 21 experiments would be necessary to determine all the coefficients for a mixture with six components. Additionally, three extra points are necessary to provide information about the error and lack of fit, and three replicated points was included to better understand the behaviour of the data due to repeatability. Finally four additional center points were added for extra design power. However, the present study found that all the properties followed a linear model. If this is known ahead of time, the number of experiments required would be about 15 saving resources and time to investigate other factors.
- Based on the satisfactory results obtained with low w/c ratios and superplasticizer, even lower w/c values could be tested. This may result in higher compressive strength and splitting tensile strength.
- Improving the grading of the waste PET by producing finer particle sizes, ranging from 1mm to 0.075 mm, could allow the use of a higher content of waste PET in mixtures. Additionally, some problems such as segregation and formation of voids may be avoided with finer particles. However, this would require significant modifications to the blades of the current shredder.
- The mixing method was not examined in this study. Manual mixing can be tried in future studies, as long as it is controlled and standardized.

Additionally, time of mixing and volume of mixing can be examined in future research. Manual mixing may not work when larger volumes need to be mixed. Manual mixing using the same mix design seems to produce much higher compressive strength for a few samples tested.

- This study used a standardized mix of waste PET of different thicknesses. The influence of the waste PET thickness remains unknown. Therefore, different types of waste PET, with different thicknesses, could be tested to develop an understanding of the impact that waste PET thickness has on the measured properties.
- Some admixtures, such as metakaolin or furnace slag, can be incorporated in the mixture in order to enhance the properties of the concrete. However, it is important to note that the addition of any admixture would increase the cost of the project.

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Appendix A

Past research on waste PET incorporation into concrete

Reference	Recycling method	Gradation / particle shape	Replacement	Admixtures	Workability	Compressive strength 28 days (MPa)	Splitting tensile (MPa)	Other properties
Choi et al. (2005)	Mechanical Thermal	5 - 15 mm / rounded	25%, 50%, 75% V	Granulated blast furnace	Slump 100 - 205 mm	21.8 - 37.2	1.94 - 3.32 Mpa	Modulus of elasticity (15.6 - 25.5 GPa)
Juki (2013)	Mechanical	5 mm / flaky	25%, 50%, 75% V	-	-	15.6 - 31.27	Reductions from 15 to 60%	Modulus of elasticity (10.4 - 25.9 GPa)
Choi et al. (2009)	Mechanical Thermal	5 - 15 mm / rounded	25%, 50%, 75% V	Water reducer	Slump 100 - 222 mm	21 - 35	1.9 - 3.2	Modulus of elasticity (18 - 30 GPa)
Albano et al. (2009)	Mechanical	Fine 0.26 cm / flaky Coarse 1.14 cm / flaky	10%, 20% V	-	Slump 20 - 90 mm	12 - 27	1.4 - 2.8	Modulus of elasticity (12 - 29 GPa)
Frigione (2010)	Mechanical	<2 mm / flaky	5% W	-	VeBe 37 - 62	40 - 69.7	4.1 - 6.3	Shrinkage 1 year (650 - 987 x 10^{-6})
Ackaozoglu et al. (2010)	Mechanical-washing	0-4mm / flaky	50% V	Granulated blast furnace	-	22.4 - 27	-	Water absorption of concrete with WPET (11.9 - 22%)
Silva et al. (2013)	Mechanical/ Thermal	Fine 4mm / flaky Coarse 2 - 11.2mm / flaky	7.5%, 15% V	-	Slump 133 - 141 mm	19.7 - 36.7	-	Carbonation depth (14 -28.8 mm)
Ferreira et al. (2012)	Mechanical/ Thermal	Fine 4mm, flaky Coarse 2 - 11.2mm, flaky	7.5%, 15% V	-	Slump 120 - 140 mm	22 - 38	1.5 - 3.4	Modulus of elasticity (17- 38 GPa)
Ismail and Al-Hashmi (2008)	Mechanical	0.15 - 4.75 mm / flaky	10%, 15%, 20% V	-	Slump 20 - 80 mm	22 - 43	-	Flexural strength (3 - 6 Mpa)
Batayneh et al. (2007)	Thermal	0.15 -4.75 mm / flaky	5%, 10%, 20% V	-	Slump 57 - 78 mm	10 - 34	0.6 - 4	Flexural strength (0.6 - 5 Mpa)

Appendix B

Results of slump, compressive strength, and splitting tensile strength.

Run	Slump			Average	Compressive strength			Average	Splitting tensile			Average
1	122	134	127	**128**	3	17.8	17.3	**17.5**	17.5	3	3	**3.1**
2	144	142	151	**146**	2.2	14.6	15.8	**15.1**	14.8	1.8	2.1	**2.9**
3	99	105	103	**102**	1.5	10.5	15.2	**12**	10.4	1.3	1.5	**1.6**
4	132	114	120	**121**	3.2	29.3	25.5	**27.8**	28.6	3.1	3.4	**3.1**
5	76	89	95	**87**	2.3	14.7	10.2	**12.9**	13.9	1.7	2.3	**2.8**
6	162	148	153	**154**	3.1	26.5	19.7	**23.6**	24.8	3.1	3.3	**2.8**
7	129	132	148	**136**	3.2	25.9	19.4	**21.9**	20.3	3.4	3.3	**2.9**
8	118	112	153	**128**	1.4	9.7	7.7	**8.8**	8.9	1.1	1.5	**1.6**
9	110	115	122	**116**	1.7	14.2	10.9	**11.6**	9.7	1.7	1.5	**2**
10	61	79	72	**67**	2.7	16.9	19.8	**19.3**	21.4	2.7	2.6	**2.8**
11	135	154	140	**142**	2.4	11	13.2	**13.5**	16.4	2.1	2.5	**2.7**
12	131	148	140	**140**	2.7	11.2	10.6	**12.4**	15.4	3	2.5	**2.7**
13	128	137	121	**129**	2.1	6.3	8.7	**7.5**	7.4	1.9	2.2	**2.5**
14	135	143	135	**140**	2.7	16.3	19.3	**19.1**	21.5	2.5	2.8	**2.9**
15	110	115	121	**115**	2.4	11.5	18.4	**16**	18.2	2.5	2.5	**2.3**
16	120	121	118	**121**	1.5	8.8	9.7	**10.1**	11.8	1.7	1.2	**1.5**
17	151	165	149	**155**	2.7	18.4	16.7	**18.8**	21.2	2.7	2.6	**2.9**
18	169	166	156	**164**	2.8	16.1	12	**15.4**	18.4	2.9	2.7	**2.9**
19	133	147	140	**149**	2.2	12.4	10.3	**11.5**	11.8	2	2.2	**2.4**
20	82	96	118	**99**	2.1	10.1	14.7	**14.4**	18.4	2	2.3	**1.9**
21	133	120	110	**121**	2.9	22.9	15.7	**19.1**	18.6	3	3.1	**2.7**
22	50	55	51	**51**	2.9	14.8	13.4	**16.6**	21.6	2.8	3	**3**
23	86	79	80	**84**	1.9	12.4	14.5	**13.5**	13.6	2.1	1.6	**1.9**
24	95	88	99	**94**	2.7	14.3	19.1	**17.3**	18.6	2.7	2.7	**2.8**
25	125	120	104	**116**	2.6	19.2	17.4	**16.3**	12.3	2.8	2.5	**2.7**
26	150	165	148	**154**	1.6	10.5	17.6	**13.5**	12.4	1.1	1.2	**2.6**
27	110	115	117	**117**	2.5	18.3	14.7	**15.6**	13.9	2.9	2.1	**2.5**
28	89	106	96	**97**	2.6	11.1	16.8	**15.6**	18.8	2.4	2.7	**2.6**
29	119	137	126	**127**	1.1	11.1	9.6	**10.6**	11.3	1.1	0.9	**1.2**
30	112	100	97	**103**	1.5	7.2	8.1	**8**	8.9	1.6	1.4	**1.5**
31	72	97	83	**84**	2.1	14.7	19.8	**16.7**	15.6	2.3	2	**2.1**