Mechanical Characteristics Investigation of Ultra High Performance Concrete Using Design of Experiment and Response Surface Methodology

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ABSTRACT

Attention to the mechanical properties of concrete for higher strength and ductility and also the increase in its durability has resulted in the innovation for several types of concrete. Ultra high performance concrete (UHPC) is one of the latest concrete with the unique properties such as high compressive strength, exhibiting tensile and flexural strength with increase in energy absorption (toughness), high durability, improved resistance against freezing- thawing and various chemical attacks. UHPC represents the highest development of high performance concrete in different curing conditions.

One of the main disadvantages of UHPC is huge amounts of binder content used for producing UHPC. The purpose of this study was to improve the mechanical properties of UHPC relative to using local materials in two different phases:

The purpose of phase one was to find the models of 7, 14 and 28-day compressive strength, 28-day splitting tensile strength, modulus of rupture, and flexural toughness of Ultra High Performance Concrete, as well as, study on the interaction and correlation of five variables including silica fume (SF), cement, steel fibers, superplasticizer (SP), and w/c ratio. The models are valid for mixes made with 1.0 part sand, 0.15-0.30 part silica fume amount, 0.70-1.30 part cement amount, 0.10- 0.20 part steel fiber, 0.04- 0.08 part superplasticizer (all values by sand weight) and 0.18-0.32 water cementitious material ratio.

In phase two, the effect of quartz powder (Qp), quartz sand (Qs), and different water curing temperatures on UHPC performance was investigated, the correlation between these variables and mechanical properties were found. The offered models are valid for the variables between: quartz powder 0 to 20% of cement substitution, quartz sand 0 to 50% of aggregate substitution, and water curing temperature 25 to 95 ºC.

The experiments were designed by central composition with α=1 (face centered). The response surface methodology was analyzed between the variables and responses. The correlation of variables and mathematical models in terms of coded variables were established by ANOVA.

Keywords: Ultra high performance concrete, strength, durability, silica fume, steel fiber, quality sand, modelling.

ÖZ

Betonda hedeflenen yüksek mukavemet ve esneklik nedeni ile betonun dayanıklılığı ve beton çeşitliliğinde gelişmeler olmaktadır. Ultra yüksek performanslı beton ise gelişmiş beton türleri içeriside yüksek basınç mukavemeti, çekme dayanımı, basmada çekme dayanımı, artırılmış enerji emme kapasitesi, yüksek dayanıklılık, donma çözünme dayanımı, ve kimyasallara karşı direnci ile öne çıkan bir beton türüdür. Bu özelliklerinden dolayı çeşitli kür şartlarında geliştirilmiş özellikleri olan bir betondur.

Ultra yüksek perfoamanslı betonun en olumsuz tarafı ise yüksek miktarlarda bağlayıcı malzeme kullanımı ihtiyacıdır. Bundan dolayı bu araştırmanın amacı kullanılan bağlayıcı miktarını azaltmak için çalışma yapmaktır. Yapılacak olan çalışmada iki farklı faz kullanılacaktır:

Birinci faz 7, 14 ve 28 günlük basınç dayanımı, 28 günlük yarmada çekme dayanımı, kopma modülü ve basma tokluğunun ultra performanslı beton için modellenmesidir. Bu çalışmada ayrıca beş farklı değişken olan silis dumanı miktarı, çimento miktarı, çelik elyaf hacmi, super akışkanlaştırıcı miktarı, ve su çimento oranı arasındaki ilişkiye de bakılacaktır.

Elde edilen modelerin geçerliliği ise 1 oranında kum, 0,15-0,30 oranı arasında değişen silis dumanı, 0,70-1,30 oranı arasında değişen çimento, 0,10-0,20 oranı arasında değişen çelik elyaf, 0,04-0,08 oranı arasında değişen super akışkanlaştırıcı ve 0,18-0,32 oranı arasında değişen su çimento oranı için geçerlidir. Buradaki oranlamalarda ise kumun ağırlığı esas alımnıştır.

İkinci fazda ise kuvarz tozunun, kuvarz kumunun ve farklı kür sıcklıklarının ultra yüksek performanslı betonun mekanik özelliklerine olan etkisi ve ayrıca bu malzemelerin kendi aralarındaki ilişkilere bakılmıştır. Elede edilen modeller ise sadece kuvarz kumunun çimento ile yüzde 0-20 arasındaki ikamesi, kuvarz kumunun yüzde 0-50 arasında agrega ile ikamesi, ve su kür sıcaklığının 25-95ºC olan şartlar için geçerlidir.

Deney tasarımları ise merkezi kompoze metodunun α=1 olduğu durum için yapılmış olup korelasyon ANOVA kullanılarak gerçekleştirilmiştir.

Anahtar Kelimeler: ultra yüksek performanslı beton, mukavemet, dayanım, silis dumanı, çelik elyaf, kuvarz kumu, modelleme.

تقدیم

به روح پرفتوح پدرم، برکت وجود مادرم و همراهی همیشگی همسرم

Dedication

To the spirit of my father, kind mother,

and infinity supports of my wife

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LIST OF ABBREVIATIONS

α	Axial points range
ANOVA	Analysis of Variance
DOE	Design of Experiment
HPC	High Performance Concrete
HRUHPC	Heavy Reinforcment Ultra High Performance Concrete
HSC	High Strength Concrete
ITZ	Interfacial Tranzition Zone
LCA	Life Cycle Assesment
NSC	Normal Strength Concrete
OC	Ordinary Concrete
PC	Portland Cement
QP	Quartz Powder
QS	Quartz Sand
RPC	Reactive Powder Concrete
RSM	Response Surface Methodology
SHSC	Super High Strength Concrete
SF	Silica Fume
SP	Superplasticizer
SRPC	Self Reactive Powder Concrete
UHPC	Ultra High Performance Concrete
UHSC	Ultra High Strength Concrete

Chapter 1

INTRODUCTION

1.1 Background

Concrete is a common construction material used for different construction purposes. Uses of cementitious material could be dated back to hundreds of centuries in countries like Italy, Egypt, Greece, and the Middle East especially in ancient Iran. Portland cement is known as an important component in concrete which was first invented and used in 19th centuries by Aspdin in England (Gooding & Halstead, 1954). Since ancient times, human have been looking for construction materials which have better and higher performance in building unique structures which could be taller, bigger, and more stable having more aesthetics (Allen & Iano, 2011). As the cost of construction materials escalates, the demand for more resolute and improved building materials have also increased globally.

In the mid 60's, concrete with strength range from 35 MPa to 85 MPa, called high performance concrete (HPC) was developed (Karmout, 2009). It was first used in meaningful quantities in major structures in cities like Chicago, USA. As the development of cement and concrete industry continued (Sutherland et al., 2001), the definition of high-strength concrete was changed. In the 1950s, concrete with a compressive strength of 34 MPa was known as high strength concrete. In the 1960s (Eldagal, 2008), concrete with compressive strength between 41 MPa and 52 MPa were used commercially and in the early 1970s, 62 MPa concrete was produced and introduced in many applications such as high-rise buildings and long-span pre-stressed concrete bridges (Kumar, 2015).

More recently, concrete compressive strengths over 100 MPa by different definition have been known as Ultra High Performance Concrete (UHPC) (Wille et al., 2011). Therefore, nowadays it is possible to produce lighter members with thinner cross sectional area and open new possibilities for high-rise buildings, bridges and suggest economic advantages through savings in reinforcing steel and cross section dimension which leads to lower dead weight (Gogou, 2012), therefore, permits larger spans. When it is compared with HPC (High Performance Concrete), UHPC exhibits superior properties like durability, advanced compressive and tensile strength, and long term stability, resulting in reducing the maintenance expenses (Mohammed, 2015). Ultra high performance concrete (UHPC) are made by using fine, rarely coarse aggregates, very low amounts of water and high amounts of cement (Kang et al., 2010). These materials are characterized by a dense microstructure. The sufficient workability is obtained by using superplasticizers in combination with low-water demand of the fresh concrete (Rashid & Mansur, 2009).

The mechanical performance, durability and ductility behavior of UHPC differs scientifically from normal and high strength concretes due to the high-packing density of these materials (Wille et al., 2012).

1.2 Statement of the Problem

In recent years many researchers worked to improve the properties of ultra-high performance concrete. This study tries to solve the problems that engineers facing with problems listed below:

1) Production of UHPC needs huge amount of cement which increases the cost of production.
2) There is no any valid mix design process on mechanical properties of UHPC with high accuracy for the local materials.
3) The interaction of the ingredients in UHPC have not been studied yet. The study about interactions of ingredients can make a good interpretation about the treatment of UHPC.

1.3 Goals

The aim of this study is to produce the optimum Ultra High Performance Concrete UHPC by using available materials and local methods and also to study and model the effect(s) of controllable mix design parameters individually and collectively in order not to limit only to mechanical properties of UHPC but also decreasing environmental hazards into different phases.

1.4 Objectives

The objectives of this study are given below:

1. To model a practical and feasible mix design to produce UHPC.
2. To study the effect of different variables on mechanical properties of UHPC.
3. To obtain the minimum amount of cement consumption with the best efficiency and performance of UHPC.
4. To model the relationship between density and compressive strength.
5. To obtain the relationship between modulus of rupture and compressive strength.
6. To study the influence of quartz sand and powder as well as thermal water curing regimes on mechanical properties of UHPC.

1.5 Methodology

1. Conduct a comprehensive literature review related to UHPC.
2. Selection of suitable materials required for producing UHPC in two different phases.
3. Determine the relative quantities of these materials in order to produce UHPC mixes.
4. Design of experiments by using response surface methodology in two levels: one with five variables and another one with three variables.
5. Perform physical and mechanical tests on UHPC samples and compare the results with available standards.
6. Analyze the model by using ANOVA and interpret by monitoring 3D modeling.

Chapter 2

LITERATURE REVIEW

2.1 Definition of Ultra High Performance Concrete

Ultra-high performance concrete (UHPC) is one of the latest batch of concrete production that produced in these years (Shah & Ribakov, 2011). When it is compared with a previous class of concrete such as HPC, ultra high performance concrete expresses higher properties like advanced compressive and tensile strength, workability, durability, and long term stability (Mohammed, 2015).

UHPC is a very dense structured material with a low water /cement ratio smaller than 0.30, having high cement content and different mineral admixtures which increase the bond between cement paste and aggregates (Van Tuan, 2011). The optimized UHPC leads to minimize the defects such as pore spaces and micro cracks that allow a higher percentage of the strength potential capacity which is defined by its ingredients and providing better durability properties. Because of having high compressive strength, this class of concrete is also named as Ultra High Strength Concrete (Mohammed, 2015).

Concrete is classified from strength point of view in five classes (Sivakumar & Santhanam, 2007):

1. Normal Strength Concrete (NSC) up to B41/60 MPa
2. High Strength Concrete (HSC) from B41/60 to B70/90 MPa

3. Very High Strength Concrete (VHSC) from B70/90 to B120/150 MPa
4. Ultra-High Strength Concrete (UHSC) from B120/150 to B200/250 MPa
5. Super High Strength Concrete (SHSC) above B200/250 MPa

Ultra-High Performance Concrete is categorized by one of the three categories of Ultra High Strength Concrete (UHSC) such as (Kumar, 2015):

1. Compacted Reactive Powder Concrete (RPC).
2. Self-Compacted Reactive Powder Concrete (SRPC).
3. Compacted Ultra High Performance Concrete (UHPC).

2.2 Advantages of UHPC

It can be mentioned that the minimum advantage of UHPC is its high level of strength. Other advantages include improved microstructure, low porosity, homogeneity, and high flexibility and ductility with addition of fibers (Karmout, 2009). As a result, UHPC has found its application in many purposes like, bridges, piers, nuclear roof storages seismic-resistant structures and designed structures to resist dynamic loads (Mohammed, 2015). Due to its improved properties, structural precast members could be fabricated in slender form to enhance aesthetics. Durability problems in normal concrete have been approved for many decades and very significant expenses have been required to maintain aged infrastructure (Li, 2011).

UHPC contains low porosity, good durability properties and low capillaries which account for its endurance. UHPC construction requires lower maintenance costs in its service life than conventional concrete (Karmout, 2009). UHPC might incorporate larger quantities of synthetic fibers or steel and has enhanced ductility, high temperature performance and improved impact resistance. This enables structural members to be built entirely from fiber reinforced Ultra High Performance Concrete without using of conventional transverse reinforcement (Hensher, 2013).

2.3 Definition of Sustainable Construction

"Sustainability" is one of the word which is used but least understood. Its meaning is sometimes included by differing explanations and interpretations and by a tendency for the topic to be treated superficially. For most countries, companies, and individuals who follow the subject seriously, the meaning of sustainability embraces the protection of the environment plus critical development, related problems such as the efficient use of resources, stable economic growth, continual social progress, and the eradication of poverty (Ding, 2008).

In the construction world, structures have the capacity to make a big contribution to a more stable future for our planet. The Organization for Economic Cooperation and Development (OECD), for example, estimates the buildings in developed countries account for more than 40% of energy consumption over their lifetime (including raw material production, construction, operation, maintenance and demolishing) (Kibert, 2008). As well as, at first time in human's history over half of the world's population now lives in urban environments and it is clear that sustainable buildings have become vital cornerstones for securing long term economic, environmental, and social viability.

2.4 Aim of Sustainable Construction

Sustainable construction aims to see present day needs for constructing, working environments and infrastructure without compromising the potential of future generations to visit their own requirement in times to come. It incorporates the economic efficiency elements, social responsibility, environmental performance and contributes to the biggest extent when architectural technical innovation, quality, and transferability are included (Ding, 2008).

Sustainable construction involves many matters such as design, construction, and construction management; construction technology, materials performance and processes; resource and energy efficiency in structures, maintenance and operation, long-term monitoring; socially-stable environments; stakeholder participation occupational health and safety and working conditions; innovative financing models; improvement to existing contextual conditions; interdependencies of landscape, infrastructure, urban fabric and architecture; flexibility in building use, function and change; and the dissemination of knowledge in related academic, technical and social contexts (Augenbroe, 1998).

2.5 Sustainable Development and UHPC

The focus is on high performance green concrete composites engineered to reduce operational energy, the embodied and greenhouse gas emission of concrete buildings produced worldwide (Damtoft, 2008). After aluminum and steel, the Portland cement manufacturing is the most energy intensive processes. It needs about 5 GJ of energy per ton and during the production 1 ton of carbon dioxide for each ton of cement is produced. High performance green concrete composites would have the potential to reduce this high energy consumption (Hasanbeigi, 2012).

UHPC and HPC generally contain a higher mass of cement than normal concrete, which causes to increase their embodied energy and carbon dioxide emissions. The same is true for steel fibers. However, they allow to construct slender and lightweight structures, which might result in less concrete usage in the foundation and lower emissions productions due to transportation of material (Yazıcı et al., 2010). Use of HPC could also become more sustainable when increased concrete durability allows for a reduction in the time periods of repair. Whether HPC/UHPC or others, more conventional solutions are more sustainable, however, they should be decided case by case by performing a specific Life Cycle Assessment (LCA) (Resplendino & Toulemonde, 2013).

2.6 Concrete Constituents

The ultra high performance concrete used in this thesis is patterned product of a major worldwide concrete producer (Graybeal, 2005). This product has a number of different material compositions depending on the particular application.

While considered the relatively new material, UHPC consists mostly of the same constituents as normal strength concrete such as Portland cement, silica fume, water, and quartz sand (Habel, 2006). However, it also includes finely ground quartz, steel fibers, and superplasticizer. Most UHPC mixes consist of these basic elements. The combination of these components creates a dense packing matrix which improves rheological and mechanical properties, and also reduces permeability (Schmidt & Fehling 2005).

Portland cement is primary binder that is used in UHPC, but at higher proportion rates than in ordinary concrete or high performance concrete. Low water/ cementitious materials ratio prevents all cement from hydrating. After thermal reaction, unhydrated cement grains exist in matrix and acting as particle packing materials. Cement with high proportions C_3A and C_3S are desirable for UHPC, as C_3S and C_3A contribute high early strength, and lower Blaine fineness decreases water demand (Šerelis et al., 2015).

Despite the large amount of particles left unhydrated, an Reactive Powder Concrete (RPC) with a water-to-cementitious material ratio of 0.20 would reach discontinuous capillary porosity when 26% hydration of cement has occurred (Bonneau et al. 2000). The additional silica fume fulfills many roles consisting particle packing, raising flowability due to spherical nature, and pozzalonic activity leading to production of additional calcium-silicate hydrate (Richard & Cheyrezy, 1995).

Quartz sand with a maximum diameter of 5.0 mm is the largest constituent aside from the steel fibers. Both quartz sand and quartz ground participate to optimized packing. Furthermore, the most permeable portion of a concrete will be the interfacial transition zone (ITZ) between cement matrix and coarse aggregates (Mehta & Monteiro 2006), so then, the exclusion of coarse aggregates helps to improve durability of ultra high performance concrete. This zone is the area around any inclusion in cementitious matrix, and is where the cement grains have difficulty growing because of presence of large surface which impedes crystal growth. Silica fume (the smallest component in ultra high performance concrete with a diameter of 0.2 μm) helps fill this zone, and because it is highly pozzolanic, aids in improved strength and reduced permeability. Reduction of the interfacial transition zone between coarse aggregates increases tensile strength and decreases the porosity of cementitious matrix (Fujikake et al., 2003). By decreasing amount of water necessary to make fluid mix, and so then permeability, the polycarboxylate superplasticizer also improves the durability and workability. Finally, addition of steel fibers aids in preventing the propagation of microcracks and thus limits the width of cracks, thus, permeability. For this particular application of UHPC, straight high carbon steel fibers with a diameter of 0.2 mm and length of 13 mm in UHPC (Graybeal, 2005). This was the largest particle in the mix and was added at 2 percent by volume to the mix. Because of its size relative to other constituents, it reinforced the concrete on the micro level and eliminated the need for secondary reinforcement in prestressed bridge girders. The choice and quantity of this fiber was chosen because of its availability, use in previous research, and likelihood that it will be used in the structures industry; specifically bridges (Graybeal, 2005).

2.7 Performance Criteria for Structures

For modern structures, researchers look for materials with four unique properties which are: workability, durability, strength, and affordability. For the first three properties basically consist all mechanical performance requirements listed above. Affordability is cost. When it is said high performance, it will be asked to the improvement in some or all of these properties like (Ter Maten, 2011):

- Compressive strengths up to 200 MPa,
- Flexural strengths up to 50 MPa,
- Modulus of elasticity 45 to 50 GPa,
- Tensile strength up to 30 MPa,
- Ductility,
- Durability,
- High flexural strength,
- Low capillary porosity (high endurance),
- High resistance to deicing salt,
- Greatly reduced permeability to moisture, chlorides and chemical attack,
- Increased resistance to abrasion, erosion and corrosion,
- Speedy construction.

Some of these properties will be discussed one by one below:

2.7.1 Strength

Higher strength causes savings materials. Weight, in the other words, the dead load which is major load in structure designs. Therefore, higher strength generally gives two benefits which contains: less weight and material (Tang, 2004). Weight reduction decreases demand on material because it decreases loads that structure has to bear. With increasing the strength up to 200 MPa, the UHPC is nearly acting like steel except its tensile capacity is still relatively low so it could not be used like steel (Ter Maten, 2011).

2.7.2 Workability

A structure is not only designing, but also it should be constructed. Workability influences the time and cost required to construct the structure. Clearly cost and time are often two essential determinants on whether a bridge or a certain type of structure will be built (Tang, 2004). Despite, this concrete is mostly used in pre-casting formwork, therefore, the workability effect was not considered in this study.

2.7.3 Durability

When it is looked at some of the ancient structures of Roman and Byzantine eras that are still standing, it is wonderful how long our structures will last. Ancient fellow engineers just built major structures based on their best knowledge and usually expected the structures to last forever. Today, it is known that nothing will last forever and we become more humble and design buildings and bridges to a defined design period. With design life of major bridges usually between 100 to 150 years, durable materials is needed that will last a long time and be easy to maintain (Tang, 2004). UHPC offers high potential in this respect. But, in the engineering world that values performance records, a certain amount of time will be needed to assure people that the long term performance of the material is what the laboratory tests have shown.

2.7.4 Affordability

Cost is often a determination factor, if a structure will be built. There are possibly other good construction materials that could be used for construction except that their high cost may have prevented them from using for the purpose of construction (Bonasia, 1975).

2.8 History of Development of UHPC

In the 1960s, concrete with a compressive strength of 800 MPa has been produced under specific laboratory conditions. They were compacted under thermal treatment and high pressure. In the early 1980s, the idea was shaped to develop fine particle concretes with high density and homogeneous cement matrix to prevent the micro cracks development within the structure when being loaded. Because of the restricted grain size of less than five mm and the high packing density owing to the use of different reactive inert or mineral additions they were named Reactive Powder Concretes (RPC) (Li, 2011). Meanwhile, there existed a bigger formulations range and the term 'Ultra-High-Performance Concrete (UHPC) was established worldwide for concretes with a minimum compressive strength of 200 MPa (Kumar, 2015). The first practical applications started in 1980. It was mainly used for specific purposes in the security industry like strong rooms and protective defense constructions and military purposes. First developments and research goal at an application of UHPC in constructions started in about 1985. Since then, different technical solutions were developed parallel or one after the other and marked as below:

- Heavy reinforced Ultra High Performance Concrete (HRUHPC) precast member for decks of bridges; in situ applications for the restoration and rehabilitation of deteriorated concrete industrial floors and bridges (Kumar, 2015).
- Different types of Ductal concrete, consisting of Reactive Powder Concrete (RPC) resulting from joint research by Bouygues, Rhodia, and Bouygues marketed by Lafarge in France (Resplendino, 2004).
- D.S.P (Densified with Small Particles Concrete) produced in Denmark with or without additional reinforcement It is used for precast members and other purposes like offshore bucked foundations (Karmout, 2009).
- BSI "Béton Spécial Industriel" (Special Industrial Concrete) specified by high amount of cement content with the use of silica fume and also small diameter aggregate developed by Eiffage (Karmout, 2009).

UHPC is gaining increasing interest in Germany. Based on an extensive research project, technical criteria and measures have been already developed to use regionally available raw materials for coarse and fine grained UHPC, to decrease the cement mass content and to use steel fiber mixtures and non-corrosive high strength plastic fibers to monitor the ductility depending on the requirements given by an individual design and construction (Schmidt & Fehling 2005).

The German Committee for Structural Concrete (DAfStB) (Grübl & Rühl, 1998), drew up a “state of the art report” on Ultra High Performance Concrete. The DAfStB is some part of the German Standardization Organization DIN being responsible for all codes, standards, and technical requirements related to the production and application of concrete and giving the rules for the design of concrete structures. The “German state of the art report" supports the technical knowhow and the experience with UHPC that have been published worldwide. It supports nearly all applications that exist here mainly on commercially available UHPC mixtures. The main principles and the characteristic behavior criteria are durability and the resistance against fire. A second part of report refers to the adequate design and construction of structures using UHPC. This report traditionally is first step towards a reliable technical guideline and a batter standard for UHPC (Karmout, 2009).

2.9 Relevant Material Property Characterization Studies

Ma et al. (2004), at University of Leipzig Germany completed a project on UHPC production with basalt particle size from 0.8 to 5 mm. The compressive strength reached the same value as reactive powder concrete when the maximum aggregate size was smaller than 1.0 mm. Using of the coarse aggregates led not only to cementitious paste volume reduction, but also required changing in process of mixing on consequent mechanical properties. UHPC included coarse aggregate was more easily homogenized and fluidized. Formulations without or with coarse aggregate showed close behavior under compressive loading, though with a bit difference in modulus of strain at peak stress and elasticity as well, which was dependent on the stiffness of aggregates. Lower paste volume fraction and physical resistance of stiffer basalt aggregate showed in a lower autogenous shrinkage of the UHPC containing coarse aggregates. The first purpose of adding coarse aggregates was to reduce the cement. Therefore, the costs of construction might be reduced. The Project had been undertaken where artificial aggregate was used to substitute with natural one, clinker-aggregates showed in rise of strength (around 20 MPa) compared to natural aggregates. Microstructure observation displays extra silica fume leads to important improvement because of particle size of silica fume which is 1/100 of a cement particle. Henceforth the space between cement particles could be filled by silica fume particles. So, voids and pores could be significantly decreased in mixture.

Stiel et al. (2004), have represented a study on the effect of steel fiber orientation on mechanical properties of UHPC. These researchers concentrated on innovated ultra high performance concrete marketed under name of CARDIFRC. The produced UHPC consisted of two different lengths of fibers and a total fiber content of six percent by volume. The research program concentrated on effect of ultra high performance concrete flow direction during the casting on compressive strength and flexural strength behaviors of the concrete. It was concluded steel fiber reinforcement tends to align with the direction of flow during casting.

Ma et al. (2004) studied compressive treatments of UHPC when loading parallel and perpendicular to flow during casting direction. The tests on compressive strength were done on 100 mm cubes and the three-point flexure tests were done on 100 X 100 mm prisms with 500 mm length. The cube compressive strength tests showed that preferential fiber direction has no significant effect either on the modulus of elasticity or on the compressive strength of UHPC. However, the three-point flexure tests results displayed that the peak equivalent flexural strength of the mixture prisms was reduced by a factor of more than 3 when the steel fibers were preferentially aligned perpendicular to the principal tensile forces.

Rougeau & Borys (2010), accomplished a research on different ultra-fines used to produce very high performance concrete and ultra-high performance concrete. The used ultra-fine powders were limestone microfiler (LM), metakaolin (MK), pulverized fly ash (PFA), micronized phonolith (PH), and siliceous microfiler (SM). The UHPC with limestone microfiler and micronized phonolith resulted in having a good fluidity. The UHPC with pulverized fly ash, metakaolin, and siliceous microfiler required more water and superplasticizer to reach the same workability. Notwithstanding, a significant higher dosage of superplasticizer in comparison of silica fume, the UHPC with metakaolin displayed poor workability with having slump of 17 cm. The fluidity of metakaolin blended cement became poorer than Portland cement at the same dosage of superplasticizer and with same water/cement ratio. UHPC with pulverized fly ash needed significant higher water content. All of the compressive strengths of UHPC were above 150 MPa except for those with pulverized fly ash. Higher performances were obtained with silica fume. Much higher strengths at periods ranging between 28 and 90 days have been noted, using silica fume.

In another investigation ultra-high performance concrete produced by structural engineering department of Kassel university, Germany was studied regarding its micro structural features when no steel fibers were incorporated. Especially measurements with mercury porosimetry, density with helium pycnometry, surface area determination with nitrogen sorption, and finally water vapor sorption were conducted. Parallel to normal hardened cement paste, porosity was strongly decreased and specific surface area was very low compared to fully hydrated cement paste, ordinary Portland cement with w/c = 0.4. Results of UHPC revealed that this material when compared to normal hardened cement paste was much denser and material with less porosity and from nitrogen sorption measurements of very low specific surface area measured compared to normal hardened cement paste (Schmidt & Fehling, 2005).

The permeability of steel fiber reinforced UHPC was investigated and compared with that of normal concrete by Hosseini et al. (2009). Research concentrated on making small cracks in 0.5 % and 1.0 % fiber reinforced concrete, therefore determination of permeability of concrete. Two principal results of the study were as follows. First, research confirmed the results of other investigators about cracks less than 0.1 mm. The width had little influence on permeability of OC. Second, research confirmed that fiber reinforcement decreases total permeability of strained section of concrete by changing crack mechanism from a few large width cracks size to many small cracks. As would be expected, concrete with a higher volume percentage of fiber reinforcement showed less permeability and consequently more durability.

Study has done by focus on creep and shrinkage treatments of UHPC by Elker et al. (2014). They indicated that shrinkage is primarily caused by self-desiccation of the concrete binder resulting in the irreversible collapse of C-S-H sheets. As UHPC contains a very low water/ cementitious ratio, this type of concrete completely self-desiccates between casting and the steam treatment. So, Ultra high performance concrete exhibits no post-treatment shrinkage. Respect to creep, Ma et al. (2004) restates previous research showing that the C-S-H phase is the only constituent in UHPC that exhibits creep. Also, they pointed out that concrete creep tends to be much more pronounced when it occurs as the concrete is desiccating. So, the collapsed C-S-H microstructure and lack of internal water both work to decrease the creep of UHPC.

2.10 UHPC Applications

As Ultra High Performance Concrete is been developed, the right market still has to be discovered to use its increased durability, strength, and flexural capacity. Up to now this versatile mixture has been used in many purposes like: pedestrian bridges artwork, acoustical panels, precast members, and a few highway bridges (Lee et al. 2013).

UHPC utilization in the USA has been restricted, but its international roots have caused many different applications in Asia, Europe, Canada, and Australia. While there are many of the applications which are related to the transportation industry, more and more uses for such innovative material are being innovated to not only reap the benefits of its strength, but also UHPC's durability (Wille & Boisvert-Cotulio, 2015).

Ultra-high-performance concrete (UHPC) is a combination of fine materials that produces a highly durable concrete with compressive strengths in excess of 100 MPa and as high as 250 MPa. Several different formulations are available and have been used in practical applications. Worldwide bridge-related applications include the following:

- Footbridge in Sherbrooke, Canada
- Two road bridges at Bourg Les Valence, France
- Footbridge in Seoul, Korea
- Footbridge at Sakata Mirai, Japan
- Footbridge at Lauterbrunner, Switzerland
- Tollgate at Millau Viaduct, France
- Road bridge at Shepherd's Creek, New South Wales, Australia

Chapter 3

RESEARCH PROGRAM, TEST METHODOLOGIES AND MATERIAL PROPERTIES

3.1 Design of Experiments (DOE)

As Laird. (2002) defined; DOE is a series of experiments in which variations are prepared to the input variables of a system or it is a process and the influences on response variables are measured. DOE is applicable to both computer simulation and physical processes models.

Experimental design is an effective method for maximizing the quantity of information gained from study while minimizing the amount of information to be received. Factorial experimental designs study effects of many different variables by varying them simultaneously instead of just changing one factor at a time. Factorial designs permit estimating of the sensitivity to each factor and to the combined effects of two or more variables (Friedman, 1994).

DOE, also called experimental design, is an organized and structured way of conducting and controlled analyzing tests to evaluate the factors that are affecting on a response variable. DOE specifies the specific setting levels of combinations of variables at which the individual runs in the tests are to be conducted. Multivariable testing method differs factors simultaneously. Since the factors are varied independently of each other, causal predictive model could be determined. Data gained from observational studies or other data not collected accordance with a DOE approach could only establish correlation, not causality. There are problems with traditional experimental methods of changing one factor at a time, i.e., its inefficiency and its inability to determine the exact effects that are caused by numerous factors acting in combination (Montgomery, 2008).

DOE was developed by Fisher (1925) at Rothamsted Experimental Position, an agricultural research station 25 miles north of London. In Fisher's first book on DOE, it was showed how valid conclusion can be plotted effectively from tests with normal fluctuations like soil conditions, temperature, and also rain fall, it was, in the presence of nuisance parameters. The known nuisance factors normally cause systematic biases in results group (e.g., batch – to – batch variation). Unknown nuisance factors usually reason accidental variability in results and are called noise. Although experimental design methods were firstly used in agricultural context, but later, this method has been applied in military and industry since 1940. Besse Day, employed at U.S. Naval Laboratory, used DOE to express difficulties like discovery the reason of bad welds at naval shipyard through Second World War (Telford, 2007). George Box, applied by Majestic Chemical Industries before coming to USA, is a leading developer of design of experiments procedures for the optimization of chemical processes (Telford, 2007). Later, W. Edwards trained statistical methods, consisting experimental design to the Japanese engineers and scientist in early 1950s (Montgomery, 2008). Genichi Taguchi was the most well-known Japanese scientist of this group, who was very famous for his quality improvement methods. One of the companies where Taguchi first applied his techniques was Toyota, Japan (Goh, 1993). Since the late 1970s, USA industry has become attracted again in quality improvement initiatives, now known as "Six Sigma" and "Total Quality" programs. DOE is considered an advanced methodology in the Six Sigma programs, which were pioneered at GE and Motorola (Basu, 2012).

3.1.1 Fundamental Principles

The fundamental principles in DOE are solutions to problems in experimentation posed by two types of nuisance factors and serve to increase the efficiency of experiments. The fundamental principles are as follows (Montgomery, 2008):

- Randomization,
- Replication,
- Blocking,
- Orthogonally, and
- Factorial experimentation.

Randomization is an approach protects against unknown bias distorting the outcomes of the experiment.

An example of a bias is tool drift in an experiment comparing a baseline procedure to a new procedure. If all tests using the baseline procedure are conducted first and then all tests using the new procedure are conducted, the detected difference between the procedures might be entirely due to tool drift. To guard against erroneous conclusions, the testing sequence of the baseline and new procedures should be in random order such as A, B, B, A, B, A, and so on.

The tool drift or any unknown bias should be "average out." Replication rises sample size and is a way to increase the precision of experiment. Replication increases signal to noise ratio when the noise originates from uncontrollable nuisance variables. Replicate is a complete repetition of a same experimental conditions, start with the initial setup. Blocking is an appropriate method for increasing precision by eliminating influence of recognized nuisance variables. For instance the recognized nuisance variables is “batch-to-batch” variability. In blocked design system, both of baseline and new procedures were also used to material samples from a batch, then to the samples from other batches. The difference between baseline procedures and new one is not effected by the batch-to-batch differences. Blocking is a constraint of completion randomization, meanwhile both of procedures are always used for each batch. Blocking method increases precision since the batch-to-batch variability will be removed from “experimental error” (Baş, D., & Boyacı, İ. H., 2007).

Orthogonality in experiment influences in factor influences being uncorrelated, therefore, will be further easily understood. These factors in orthogonal design of experiment are varied independently from each other. The results of data using this design could be brief by taking averages differences and could be illustrated by graphically selected groups of averages. Recently, with being of powerful computers, orthogonally is no longer needed anymore, but still it is a desirable property, since ease of explanation results. The factorial experiment is an appropriate methodology in which influences due to each combinations and variable of factors are assessed. Factorial design is geometrically constructed and vary all variables all together. Factorial design gathers information at the vertices of cube in X-dimensions (X is the Number of factors being studied). If information are obtained from all of vertices, design is a full factorial which is required 2^x runs. Subsequently, whole number of mixtures rises exponentially with number of variables studied, fractions of full factorial design could be created. By increasing the number of variables, the fractions amount become smaller as 1/2, 1/4, 1/8, 1/16, and 1/32. Fractional factorial method designs collect result from specific subset of whole possible vertices and require $2^x/2$ runs. If there are just three variables in experiment, geometry of DOE for full factorial experiment will need (2^3) experiments, and also 3/2 fractional factorial experiments will need 4 runs as shown in Figure 3.1. Factorial design, consisting fractional factorial, have increased accuracy over other design types which is because of having built in internal replication. Effect of factors are fundamentally difference between regular of all experiments at two levels for any variable which are called "high level (+1)" and "low level (-1)". Duplicates of same levels will not require in factorial design method, which seems like replication infraction principle in experimental designs. However, half of whole data results are obtained at low level and the other half are obtained at high level of any variable, resultant in very large amount of duplicates. Duplication is provided by variables which involved in design and make to have non-significant influences. Whereas, every variable is varied with respect to all of variables, results on all variables is obtained by any experiment. Actually, each result is used in analysis many times within, for the estimation of any interactions and effects. Additional effectiveness of two levels factorial design is coming from a fact that it spans factor space, which is half of design points at each end level, which was most practical way of determination if variable has any significant influence or not (Baş, D., & Boyacı, İ. H., 2007).

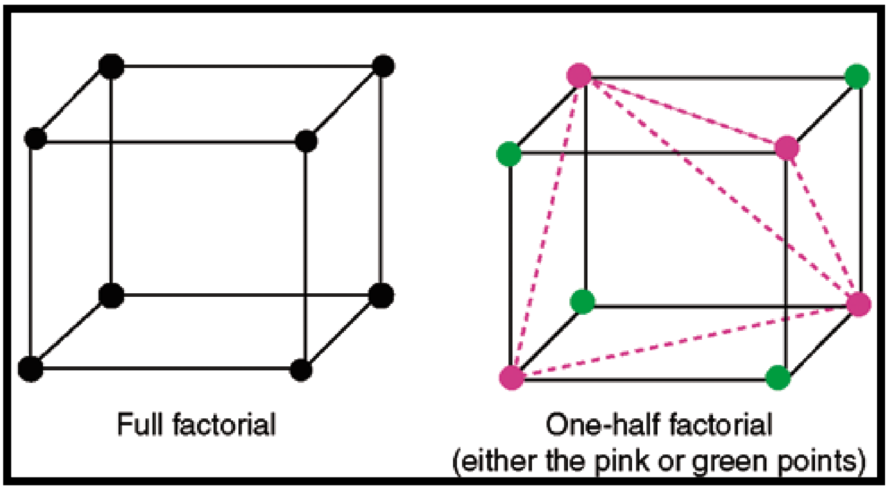

Figure 3.1: Full factorial (left) and 3/2 factorial in 3 dimensions (right)

3.1.2 Usages

The main uses of design of experiments are (Telford, 2007):

- Screening many factors,
- Discovering interactions among variables,
- Maintaining and establishing and quality control,
- Designing robust modeling, and
- Optimizing a process, including evolutionary operations.

Variable Interactions occur when influence on response of change in level of one variable from low (-1) level to high level (+1) depends on level of other variables. When interaction is presented between variables, combined influence of those variables on the response variable could not be forecasted from separate influences (Telford, 2007). The influence of variables acting in the combination could either be larger or fewer than would be estimated from each variable independently. Mostly, it is required to estimate a method with many input factors and with measured output factors. The process can be complex computer simulating modelling or engineering processing with different ingredient, pressure, temperature and many other factors as inputs (Loehlin, 2004). Screening experiment explains which input factors are causing main effect in the different responses. Each variable could also be named characterization testing or sensitivity analysis.

The found effects are efficiently using design of experiment like screening design (Telford, 2007). Optimizing a process includes determination shape of response. Regularly, screening design will be firstly completed to find out relatively few important variables. RSM has different levels on each of variables. This will produce more detail image of surface, particularly providing information upon which variables have curvature and area in response where peak and plateaus happen (Loehlin, 2004).

3.2 Response Surface Methodology (RSM)

Some part of this thesis addresses finding the prediction modeling of UHPC by using of response surface methodology, through carefully attention paid to quantification of mistake. This thesis is focused on the application of response surface methodologies to mechanical properties of UHPC.

The focus of this study is directly toward statistical models, or RSM for using mix design. Figure 3.2 conceptually illustrated area of models in which it is expected that RSM may be used. RSM may be hired with low effort and potentially have to be applied to both non-linear and linear problems.

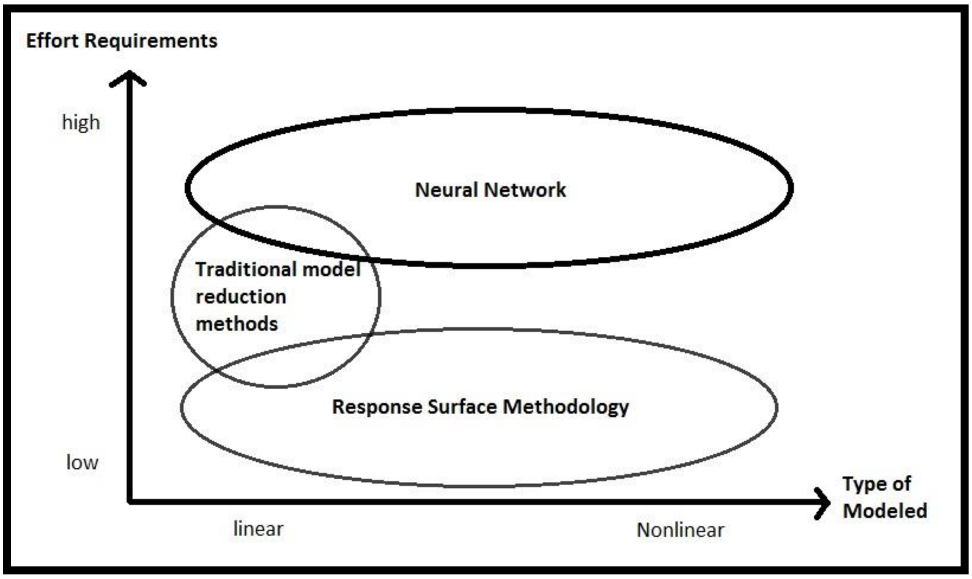

Figure 3.2: Conceptual plot of meta-models and problems they suit.

The concept of design space was presented as set of wholly possible experiments or simulations that interest analyst. That is set that contains all independent and controllable factors set at all possible levels (Fang, 2005). In case of RSM, design of experiments methodologies (often called response surface methods) are working. Already it was very popular in industrial and chemistry engineering communities, DOE is a statistical method used to “intelligently” control which simulation or physical experiments should be performed when the resources are rare (Montgomery, 2008).

DOE relies on ANOVA (analysis of variance) to choose a few results of full factorial set that effectively provide information about full response surface. Models will then be fix to the intelligently selected data using standard multiple regression methodologies resultant in different model type like polynomial, linear, quadratic models that relates input variables to output features. While the models are empirical in nature, they could rely on expertise of experimenter for assignment of model input parameters and choice of proper output responses.

Advantages of using RSM formulation are so many. Table 3.1 summarizes abilities of each of methods and it can be shown that RSM has many desirable qualities (Baş & Boyacı, 2007).

Table 3.1: Disadvantages and advantages of different modeling techniques.

	Neural Networks Methods	Response surface	Traditional Model Reduction Methods
Models linear	Yes	Yes	Yes
Models non-linear	Yes	Yes	No
Models stochastic	Yes	Yes	No
Data requirement	High	Low	Mid

It is empirical nature of RSM that makes them well suited to nonlinear simulation, because higher order models could be used to relate input factors to responses.

Relatively few data sets are needed to build a model relating inputs and outputs. Using of low order models will have an increasingly important part to play in forecasting modeling.

Furthermore, the issues of efficiently using small amounts of results, the polynomial modeling can make it particularly well suited to the mix design with maximum variables. If several responses are modeled, then separate model could be used with the optimization formulation and system inputs could be determined in this style. While input formulations could still be non-unique, they could be rated as those most likely to have caused output measuring.

3.3 Material Properties

Materials used in this study are listed below:

3.3.1 Cement

The type 2 Portland sulfate resisting slag cement of 42.5N was used which is controlled by European standard EN 197-1 (2002) cement composition. The amount of slag and clinker for manufactured cement in Cyprus were between 21-35% and 65-79%.

3.3.2 Fine aggregate

In this study limestone sand with maximum particle size of 5 mm was used. Sieve analysis was done based on ASTM C136 (1995) and controlled by ASTM C33 (2004) as shown in Figure 3.3.

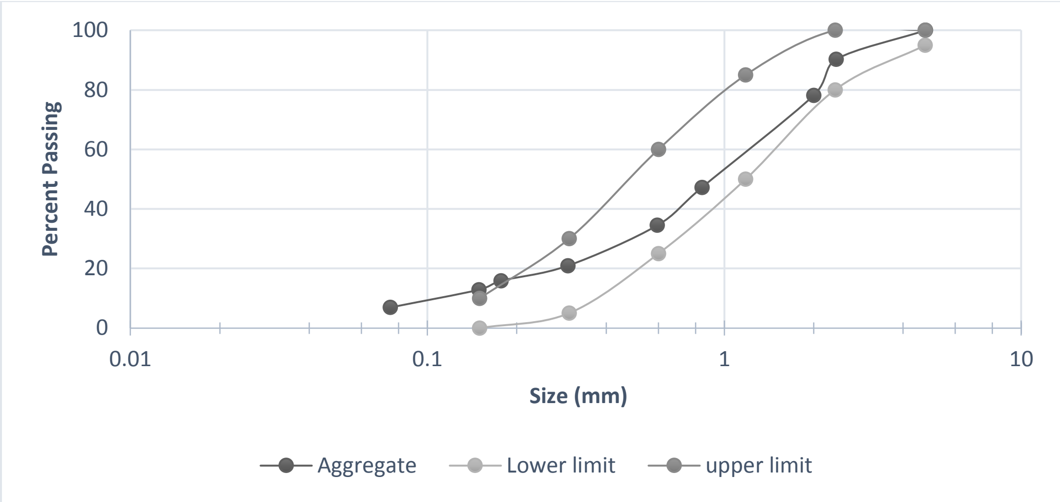

Figure 3.3: Particle size distribution of sand

3.3.3 Mixing Water

The water used for mixing and curing was ordinary tap water.

3.3.4 Superplasticizer

The superplasticizer was a polycarboxylic ether based with high range water reducing property. The new generation superplasticizer admixture developed for UHPC called GLENIUM 27 manufactured by BASF was used. The superplasticizer is consistent with EN 934-2 (2009).

3.3.5 Steel Fiber

The diameter and length of fiber was 0.55 mm and 13 mm with the tensile strength of 1345 MPa and young modulus of 21 GPa. The steel fiber was manufactured by Dramix, and confirmed by ASTM A820 (2001).

3.3.6 Silica Fume

A white undensified silica fume with more than 95% purity of silicon dioxide and particle sizes between 0.1-1 μm as pozzolanic material was used.

3.3.7 Quartz powder (Qp)

The crushed quartz powder was used as cement substitution with the particle size of less than 0.125 µm and more than 99.2 percent of SiO_2 component. The chemical analysis to find the purity percentage was done which is shown in Table 3.2.

Table 3.2: Chemical analysis of quartz powder

Crushed quartz chemical analysis	
Component	Percentage
LOI	0.05
SiO_2	99.26
Al_2O_3	0.33
Fe_2O_3	0.027
TiO_2	0.023
CaO	0.01
MgO	0.08
Na_2O	0.01
K_2O	0.21

3.3.8 Quartz Sand (Qs)

The crushed quartz sand was used as an aggregate substitution which replaced by crushed limestone sand. It is in the form of yellowish-white with particle size between 0.125 µm and 200 µm. where the sieve analysis is given in Figure 3.4. The chemical analysis to find the purity percentage is shown in Table 3.2.

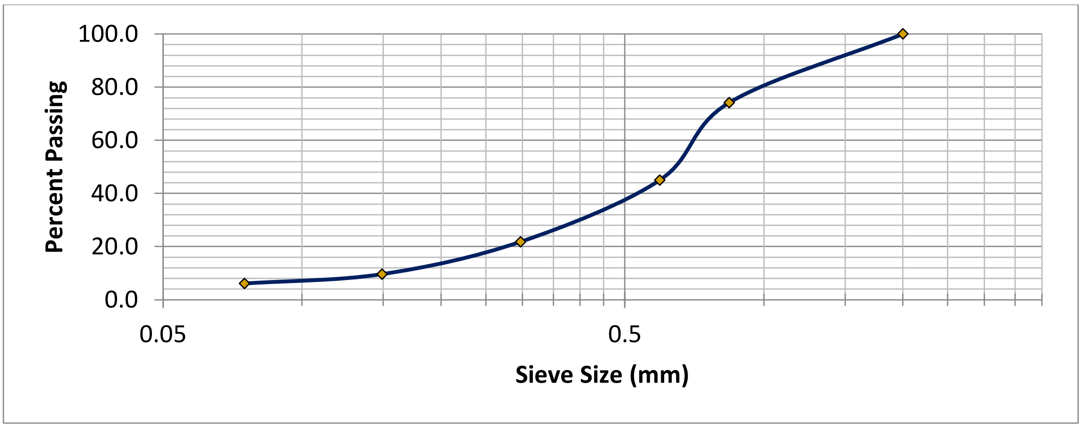

Figure 3.4: Particle size distribution of crushed quartz sand

Chapter 4

STATISTICAL MODELS FOR MECHANICAL PROPERTIES OF UHPC WITH LOCAL MATERIALS USING RESPONSE SURFACE METHODOLOGY (PHASE ONE)

4.1 Introduction

Concrete is still one of the most popular materials used in construction. However, it still has some inherent drawbacks like tensile strength or brittleness (Yoo et al. 2013a, b). Therefore, attention is paid to improve the properties of concrete for higher strength and ductility and tending to improve the durability resulted in innovation of several types of concrete (Zhang et al. 2014a, b). Ultra high performance concrete (UHPC) is one of the latest concrete that has unique properties (Wang, 2014) such as high compressive strength, exhibiting the tensile and flexural strength with increase in energy absorption (toughness), improved high durability, improved resistance against freezing- thawing and various chemical attacks (Ma et al., 2004a, b).

Despite increasing the concrete performance, the concrete performing in terms of CO_2 emissions and environmental effects should be also considered. In these decades, global warming and other significant ecological changes are increasing (Wille, 2015). For producing UHPC a large amount of binder or cement is required which has been reported by researchers to be more than 1000 kg/m^3 (Yu, 2014). Whereas, manufacturing 1 ton of cement produces approximately 900 kg/m^3 of carbon dioxide, finding the optimum amount of cement is significantly meaningful. On the other hand, some investigations showed some solution by cement replacement without significant decrease in performance (Yu, 2014).

Toughness is a quantity of energy absorption capacity, and it is used to describe the ability of UHPC to resist fracture when static, dynamic and impact loads are applied. Energy absorption or toughness capability could be calculated from the area under the load-deflection curve in flexure, which will be the total energy absorbed prior to complete separation of the specimen (Marar et al. 2011a, b).

Effects of steel fiber content and shape on flexural toughness of ultra high performance concrete was studied by Wu et al. (2016). The effect of just steel fiber orientation on flexural toughness were studied by Barnett et al. (2010). The effect of steel fiber and silica fume on flexural toughness were studied by Zhang et al. (2014). In most studies the single or some effects of concrete ingredients on flexural toughness were modeled and studied while in this study the effect of five independent variables together and the interactions between them on flexural toughness strength was modelled.

Statistical method based on experimental design is used for this research work. Response surface method is a combination between statistical and mathematical techniques (Mohammed et al. 2014a, b), which can be used for modeling and analyzing in order to find the relations between variables.

Response surface methodology (RSM) is a combination between mathematical and statistical techniques, it can be used for modeling and analyzing several variables which gives a good interpretation by finding the relationship between variables to achieve the optimum response. There are many methods which find the optimum mix proportions, for instance, De Larrad & Sedran (1994) used particle packing model for mix proportioning. Aldahdooh & Bunnori (2013) reported using RSM within two variables for evaluating UHPC binder content. In this research the RSM used for modeling and optimizing the mechanical properties of UHPC in normal curing and local materials with 5 variables which were w/c ratio, SF amount, cement amount, steel fiber amount, and superplasticizer.

Many studies were made on how to increase the toughness by different fiber properties (Tuan et al., 2014), but they didn't focus on the effect of some other ingredients and interaction between them. This research tried to monitor the effect of concrete ingredients, separately or together on flexural toughness as well as offering the model for energy absorption prediction.

The quality of the concrete in a structure is determined by the properties and proportions of the aggregate, cement, water and additives used, by the mixing and compaction procedure, by the curing of the concrete after placing and by the age at which the concrete is tested (Yu, 2014). In particular, the strength of concrete is greatly influenced by the water/cement ratio and the relative volume of air in the mix. Since aggregates, cement, water and air have different specific weights, the overall density of a concrete mix will depend upon the relative amounts of these materials (Zain, 2008. Wille, 2015. Wany, 2012). A relation between the strength and the density of a particular concrete mix may therefore be expected (Mattacchione, 1995). This relation, if it could be established, would be unique only for mixes with identical cement and aggregates, proportions, curing, age at testing and strength testing procedure (Arafa et al. 2010).

The density of concrete in a structure can be measured nondestructively using gamma radiation (Arafa et al. 2010), if the relation between strength and density for a particular concrete under the relevant curing conditions were known, the strength could be inferred from the value of density obtained (Ma et al., 2004). In order to decide whether or not the measurement of density could be used to give a reasonably accurate value of the strength of ultra high performance concrete, it is necessary to know the sensitivity of the strength of concrete to changes in the density (Senthil Kumar & Baskar, 2014). Arafa (2010) briefly showed increasing the strength by raising density and also Zain (2008) make prediction model by using density for normal concrete. In this research the effect of density with several different factors were studied.

4.2 Experimental Design

Design of experiment was done by using RSM. In this study the mechanical properties of UHPC was analyzed and the relation between variables were considered.

4.2.1 Methodology

In this research, based on RSM, the mechanical properties of UHPC with local materials at different levels as well as mix proportion for each response was considered and the interaction of variables was monitored. The response surface modeling used was central composition design with α=1 (face centered) and linear or quadratic models for responses. The interaction between variables and the effect on responses were analyzed by ANOVA. The statistical software "Design- Expert version 9.0.3", Stat-Ease, Inc., was used to analyze the experimental design.

In this study, the mechanical properties of UHPC was investigated as: 7 days compressive strength, 14-day compressive strength and 28-day compressive strength as well as splitting tensile and flexural strength test were denoted as responses and 5 variables including SF (A), superplasticizer content (B), steel fiber content (C), cement content (D), w/c ratio (E) were defined to explain the modeling. Based on previous studies and literature review, the range of variables are as follow: SF amount is from 15 to 30 percent of sand mass, superplasticizer content is from 4 to 8 percent of sand mass, steel fiber content is from 10 to 20 percent of sand mass, the cement amount is from 70 to 130 percent of sand mass, and w/c ratio is from 0.18 to 0.32. The variables with their level limitation are given in Table 4.1.

The flexural toughness test which was monitored through ASTM C1609 (2012) was also defined as the response and five above variables were defined to explain the modeling. Based on previous studies as reported by Yu et al. (2014), Máca et al. (2014), Wille et al. (2012), the range of variables were selected as follows: SF amount is from 15 to 30 percent of fine aggregate mass, the superplasticizer content is from 4 to 8 percent, the steel fiber content is from 10 to 20 percent, the OPC amount is from 70 to 130 percent of fine aggregate mass, and w/c ratio from 0.18 to 0.32.

Table 4.1: The variables with their levels

Variables	Assigned	Levels of Variables		
		-1	0	+1
Silica fume	A	15%	25%	30%
Superplasticizer	B	4%	6%	8%
Fiber	C	10%	15%	20%
Cement	D	70%	100%	130%
W/C Ratio	E	0.18	0.225	0.32

Percentages of variables are based on aggregate mass used

4.2.2 Specimen Preparation and Test Specimen

In this research, 45 batches were prepared (see Table 4.2) which were mixed in a drum rotating mixer. The first premix which included dry materials (cement, SF, sand) except steel fiber were blended in determined proportion for 5 minutes, then proportional amount of superplasticizer was added to suitable water as well as steel fiber, thereafter, water was added to premixed mixture and mixed to obtain homogeneous paste. Ten cubes with dimensions of 100mm were cast for compressive strength determination at three different ages (7, 14, and 28 days). Also, three 100 x 200mm (D x L) cylinders were cast for 28-day splitting tensile strength test, and finally three 100x100x500 mm beams were used for 28-day flexural strength test. After casting, all samples were compacted by vibration table and kept in the moist curing room for 24 hours. The specimen were then demolded and transferred to the curing water tank at 23 ± 2°C until testing.

Table 4.2: Design of experiments

	SF	SP	Fiber	Cement	w/c	Sand	SF	SP	Fiber	Cement	w/c
Mix no	A	B	C	D	E		A	B	C	D	E
1	-1	1	-1	-1	1	1	0.15	0.08	0.10	0.7	0.32
2	1	-1	-1	1	1	1	0.30	0.04	0.10	1.3	0.32
3	1	-1	1	-1	-1	1	0.30	0.04	0.20	0.7	0.18
4	0	0	1	0	0	1	0.20	0.06	0.20	1.0	0.22
5	1	-1	-1	-1	-1	1	0.30	0.04	0.10	0.7	0.18
6	0	0	0	0	-1	1	0.20	0.06	0.15	1.0	0.18
7	-1	1	-1	-1	-1	1	0.15	0.08	0.10	0.7	0.18
8	1	-1	1	1	-1	1	0.30	0.04	0.20	1.3	0.18
9	-1	0	0	0	0	1	0.15	0.06	0.15	1.0	0.22
10	-1	-1	1	-1	1	1	0.15	0.04	0.20	0.7	0.32
11	0	-1	0	0	0	1	0.20	0.04	0.15	1.0	0.22
12	1	1	-1	1	1	1	0.30	0.08	0.10	1.3	0.32
13	0	0	0	1	0	1	0.20	0.06	0.15	1.3	0.22
14	1	-1	1	-1	1	1	0.30	0.04	0.20	0.7	0.32
15	1	-1	1	1	1	1	0.30	0.04	0.20	1.3	0.32
16	-1	-1	-1	1	1	1	0.15	0.04	0.10	1.3	0.32
17	0	0	0	0	1	1	0.20	0.06	0.15	1.0	0.32
18	-1	-1	1	1	1	1	0.15	0.04	0.20	1.3	0.32
19	-1	-1	-1	1	-1	1	0.15	0.04	0.10	1.3	0.18
20	0	0	0	0	0	1	0.20	0.06	0.15	1.0	0.22
21	1	-1	-1	-1	1	1	0.30	0.04	0.10	0.7	0.32
22	0	0	0	0	0	1	0.20	0.06	0.15	1.0	0.22
23	-1	1	-1	1	1	1	0.15	0.08	0.10	1.3	0.32
24	1	1	1	1	-1	1	0.30	0.08	0.20	1.3	0.18
25	1	1	1	1	1	1	0.30	0.08	0.20	1.3	0.32
26	-1	1	1	-1	-1	1	0.15	0.08	0.20	0.7	0.18
27	-1	-1	1	1	-1	1	0.15	0.04	0.20	1.3	0.18
28	-1	-1	1	-1	-1	1	0.15	0.04	0.20	0.7	0.18
29	1	1	-1	-1	-1	1	0.30	0.08	0.10	0.7	0.18
30	1	1	1	-1	1	1	0.30	0.08	0.20	0.7	0.32
31	-1	-1	-1	-1	-1	1	0.15	0.04	0.10	0.7	0.18
32	1	1	1	-1	-1	1	0.30	0.08	0.20	0.7	0.18
33	1	-1	-1	1	-1	1	0.30	0.04	0.10	1.3	0.18
34	-1	-1	-1	-1	1	1	0.15	0.04	0.10	0.7	0.32
35	1	1	-1	1	-1	1	0.30	0.08	0.10	1.3	0.18
36	-1	1	1	1	1	1	0.15	0.08	0.20	1.3	0.32
37	-1	1	1	-1	1	1	0.15	0.08	0.20	0.7	0.32
38	-1	1	-1	1	-1	1	0.15	0.08	0.10	1.3	0.18
39	1	1	-1	-1	1	1	0.30	0.08	0.10	0.7	0.32
40	0	1	0	0	0	1	0.20	0.08	0.15	1.0	0.22
41	1	0	0	0	0	1	0.30	0.06	0.15	1.0	0.22
42	0	0	0	-1	0	1	0.20	0.06	0.15	0.7	0.22
43	0	0	-1	0	0	1	0.20	0.06	0.10	1.0	0.22
44	0	0	0	0	0	1	0.20	0.06	0.15	1.0	0.22
45	-1	1	1	1	-1	1	0.15	0.08	0.20	1.3	0.18

4.2.3 Compressive Strength Test

To determine compressive strength of specimens, 100 mm size cubes were tested. Concrete compression machine based on ASTM C109 (2002) with 3000 kN in capacity was used. Three samples for each test age were tested. The compressive strength of specimens were determined from 41 to 95 MPa for 7 days, 45.3 to 103 MPa for 14 days, and 47 to 110 MPa for 28days.

4.2.4 Tensile Strength Test

Two types of indirect tension tests were implemented: flexural strength and splitting tensile strength of cylinders. The tests were carried out on 28-days age specimen.

4.2.5 Flexural Strength Test

ASTM C1609 (2012) standard was used for this test. This test involves four point flexural loading. The beam size was 100x100x500 mm with a span length of 300 mm and load distance of 100 mm.

4.2.6 Splitting Tensile Strength

Splitting tensile test was performed in accordance with ASTM C496 (2004). The specimen size for doing spitting size was 100x200 mm (DxL) cylinder. Compression testing machine was used to do this experiment.

4.2.7 Flexural Toughness Strength

The ASTM C1609 (2006) standard was used in performing the test. This test involves four point flexural loading. The beam size which was 100*100*500 mm with the span of 300 mm and load distance of 100 mm. One sample with third-points within deflection measurement under universal machine loading with two LVDT at the middle of the span and two sides of beams is shown in Figure 4.1. The flexural toughness is the area under the load versus net deflection curve when deflection ranges from 0 to 2 mm (l/150).

Figure 4.1: Flexural toughness test

4.3 Results and Discussion of Results

The effects of five variables (silica fume content, superplasticizer content, steel fiber content, cement content, and w/c ratio) on the mechanical properties (compressive and tensile strength) as well as, flexural toughness of UHPC were analyzed by using the response surface method.

4.3.1 Mechanical Properties

Table 4.3 shows the results of compressive strength at 7, 14, and 28-days, splitting tensile strength, and modulus of rupture. Each result was derived by average of three specimens.

Table 4.3: Mix design amounts and responses of UHPC mixtures

Mix no	Sand kg	SF kg (A)	SP kg (B)	Steel Fiber kg (C)	Cement kg (D)	Water kg (E)	Compressive strength (MPa)			Tensile (MPa) Y_4	Rupture (MPa) Y_5
							7 Y_1	14 Y_2	28 Y_3		
1	50	7.5	4	5.0	35	13.6	58.0	65.0	71.0	5.5	7.02
2	50	15.0	2	5.0	65	25.6	42.0	48.0	49.0	4.0	4.95
3	50	15.0	2	10.0	35	9.0	88.5	96.0	103.0	9.0	11.46
4	50	10.0	3	10.0	50	13.5	75.0	86.0	97.0	10.0	10.50
5	50	15.0	2	5.0	35	9.0	87.0	91.0	97.0	9.0	11.07
6	50	10.0	3	7.5	50	10.8	66.0	91.0	102.0	6.0	6.62
7	50	7.5	4	5.0	35	7.6	77.8	91.0	102.0	6.1	8.50
8	50	15.0	2	10.0	65	14.4	95.0	103.0	110.0	5.5	8.75
9	50	7.5	3	7.5	50	12.9	81.5	94.0	106.0	6.2	8.28
10	50	7.5	2	10.0	35	13.6	76.0	83.0	85.0	5.4	7.00
11	50	10.0	2	7.5	50	13.5	71.5	80.8	83.4	5.0	7.47
12	50	15.0	4	5.0	65	25.6	64.5	74.6	77.0	3.5	4.00
13	50	10.0	3	7.5	65	16.8	72.2	81.5	86.0	5.0	9.47
14	50	15.0	2	10.0	35	16.0	42.6	53.3	57.3	4.6	7.33
15	50	15.0	2	10.0	65	25.6	41.0	45.3	47.0	3.9	6.27
16	50	7.5	2	5.0	65	23.2	67.0	72.0	81.5	4.6	5.00
17	50	10.0	3	7.5	50	19.2	73.1	85.3	93.5	4.7	9.25
18	50	7.5	2	10.0	65	23.2	61.3	67.5	71.7	4.9	10.74
19	50	7.5	2	5.0	65	13.0	85.0	99.5	104.5	6.2	11.25
20	50	10.0	3	7.5	50	13.5	61.6	67.1	73.0	4.5	7.84
21	50	15.0	2	5.0	35	16.0	44.8	56.0	63.6	4.6	7.20
22	50	10.0	3	7.5	50	13.5	76.0	80.0	86.0	5.1	9.56
23	50	7.5	4	5.0	65	23.2	69.0	80.0	82.0	4.0	5.00
24	50	15.0	4	10.0	65	14.4	78.0	85.0	93.0	7.0	10.00
25	50	15.0	4	10.0	65	25.6	56.2	64.1	70.8	4.7	7.61
26	50	7.5	4	10.0	35	7.6	74.0	79.0	82.9	6.4	11.64
27	50	7.5	2	10.0	65	13.0	94.6	97.0	105.8	8.5	12.14
28	50	7.5	2	10.0	35	7.6	91.3	101.5	109.0	10.2	15.00
29	50	15.0	4	5.0	35	9.0	80.0	87.0	99.0	5.1	7.00
30	50	15.0	4	10.0	35	16.0	66.6	73.5	85.0	5.0	6.60
31	50	7.5	2	5.0	35	7.6	83.0	94.0	109.0	8.0	12.40
32	50	15.0	4	10.0	35	9.0	76.0	79.5	87.0	8.5	9.31
33	50	15.0	2	5.0	65	14.4	75.9	94.8	95.7	4.9	9.37
34	50	7.5	2	5.0	35	13.6	54.0	58.7	68.9	5.2	7.70
35	50	15.0	4	5.0	65	14.4	72.0	80.0	88.0	4.4	8.40
36	50	7.5	4	10.0	65	23.2	62.5	75.5	87.0	5.5	5.67
37	50	7.5	4	10.0	35	13.6	67.4	73.0	79.0	5.2	9.24
38	50	7.5	4	5.0	65	13.0	89.0	92.2	93.9	4.7	9.61
39	50	15.0	4	5.0	35	16.0	61.1	72.0	86.0	5.5	6.97
40	50	10.0	4	7.5	50	13.5	73.0	83.0	86.0	4.5	7.00
41	50	15.0	3	7.5	50	14.6	73.5	85.0	95.0	5.0	7.00
42	50	10.0	3	7.5	35	10.1	70.0	79.0	83.0	6.5	8.88
43	50	10.0	3	5.0	50	13.5	73.7	84.0	95.2	7.0	8.54
44	50	10.0	3	7.5	50	13.5	70.0	76.0	82.0	5.4	11.03
45	50	7.5	4	10.0	65	13.0	75.8	86.0	91.3	5.4	9.47

The interaction and correlation between variables and responses was calculated by ANOVA analysis of variance. For the modeling, linear model, two-factor interaction, and quadratic models were considered to find best predictive model. In each model, the significant parameters were detected and then, by backward elimination technique the insignificant terms were eliminated and the final regressions for each were performed. Consequently, the quadratic model was selected for all responses. The quality of prediction models were determined by coefficient of multiple determination R^2, which shows the total deviation of the variables from the prediction model. The p-value (probability of errors) with 95% confidence level and statistical significant test at 5% and also lack of fit with p-value greater than 0.05 was performed for model validations.

Table 4.4 shows that all quadratic models were significant according to t-test ($P < 0.05$) and F-value of 13.44, 14.19, 15.43, 11.74, and 13.10 and lack of fit with given P-value implies which are insignificant. In addition, the model coefficient of determination R^2 has a reliable confidence with 0.87, 0.88, 0.88, 0.88, and 0.83 for the different responses. The predicted R^2 of 0.7, 0.73, 0.75, 0.67, and, 0.69 are in reasonable agreement with adjustment in R^2 of 0.81, 0.82, 0.82, 0.81, and 0.77 for all responses, whereas, the differences is less than 0.2. The adjusted R-squared compares the explanatory power of regression models that contain different numbers of predictors. The adjusted R-squared is a modified version of R-squared that has been adjusted for the number of predictors in the model. The adjusted R-squared increases only if the new term improves the model more than would be expected by chance. It decreases when a predictor improves the model by less than expected by chance. The adjusted R-squared can be negative, but it’s usually not. It is always lower than the R-squared.

The predicted R-squared indicates how well a regression model predicts responses for new observations.

Table 4.4: Analysis result of regression models

Response	R^2	Adj-R^2	Pre-R^2	F-Value	Lack of fit	Model P-value
Compressive strength 7 days	0.87	0.81	0.70	13.44	0.81	<0.0001
Compressive Strength 14 days	0.88	0.82	0.73	14.19	0.61	<0.0001
Compressive strength 28 days	0.88	0.82	0.75	15.43	0.54	<0.0001
Splitting tensile strength	0.88	0.81	0.67	11.74	0.30	<0.0001
Modulus of Rupture	0.83	0.77	0.69	13.10	0.92	<0.0001

The performance of offered prediction models with mechanical responses (7, 14, and 28 days compressive strength, splitting tensile strength, and modulus of rupture) for mixture experimental design of UHPC are illustrated in Figures 4.2- 4.6.

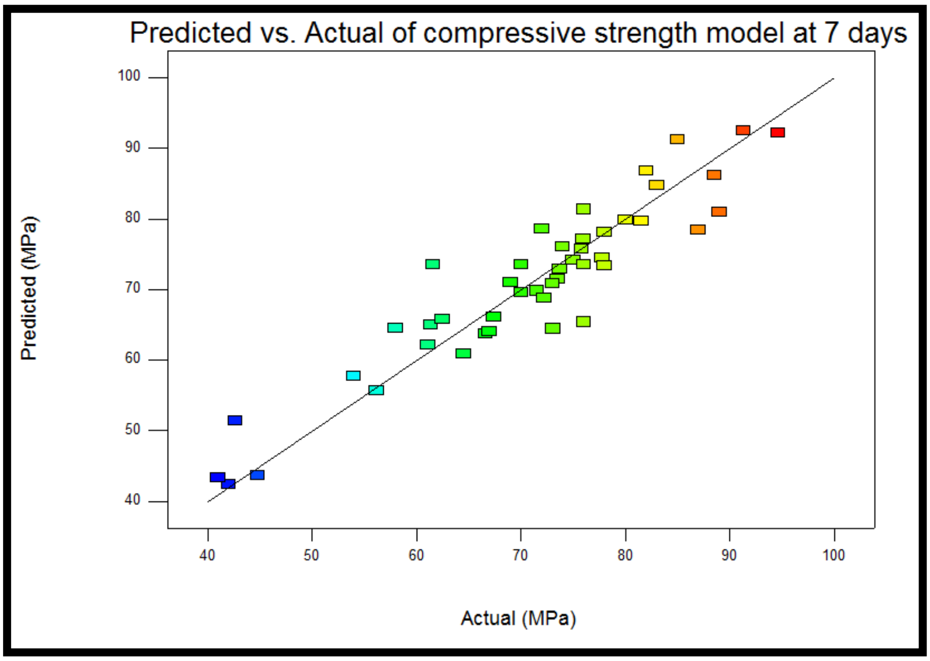

Figure 4.2: Prediction efficiency of offered model for 7-day compressive strength

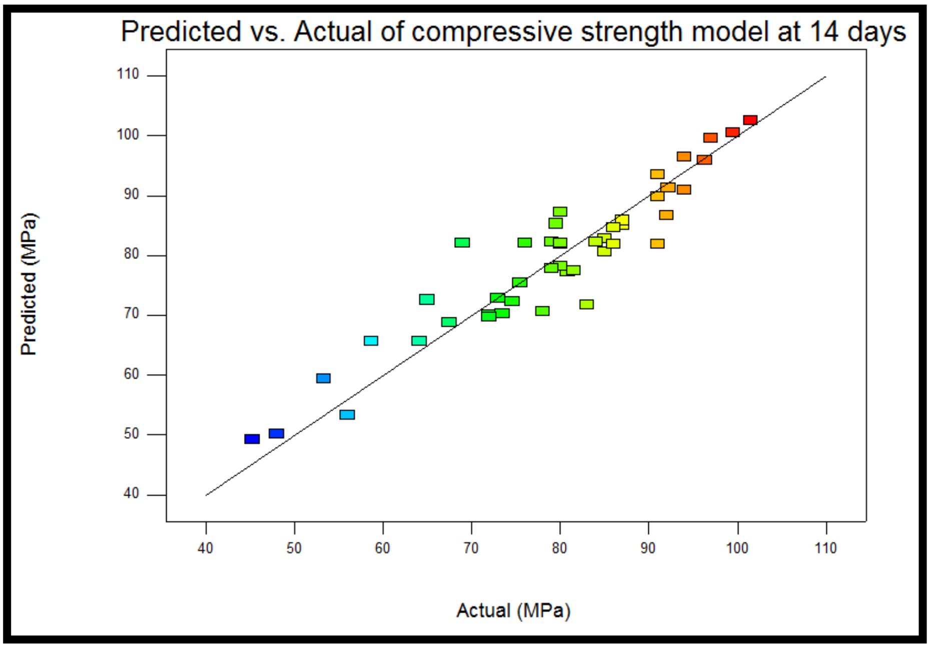

Figure 4.3: Prediction efficiency of offered model for 14-day compressive strength

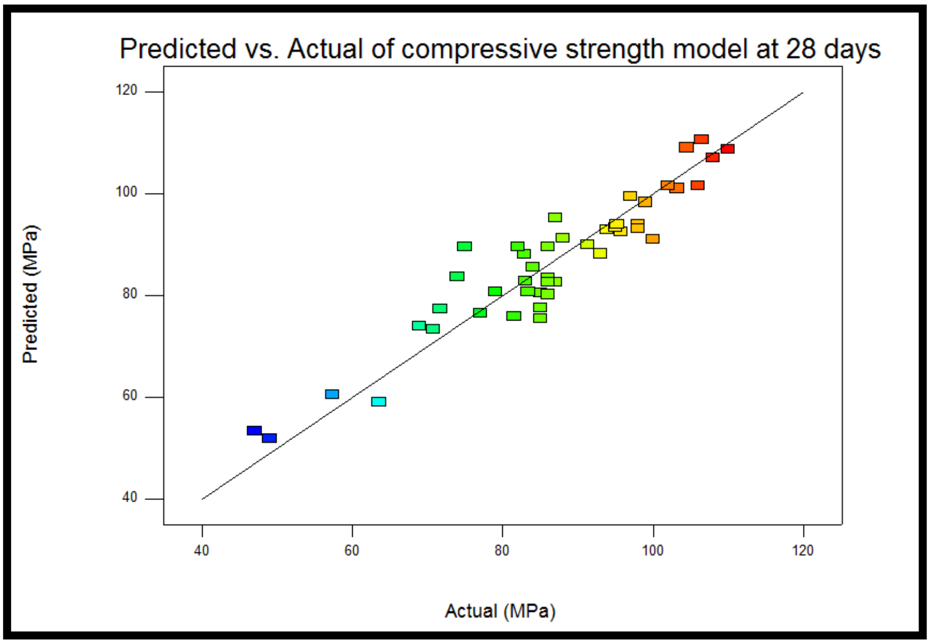

Figure 4.4: Prediction efficiency of offered model for 28-day compressive strength

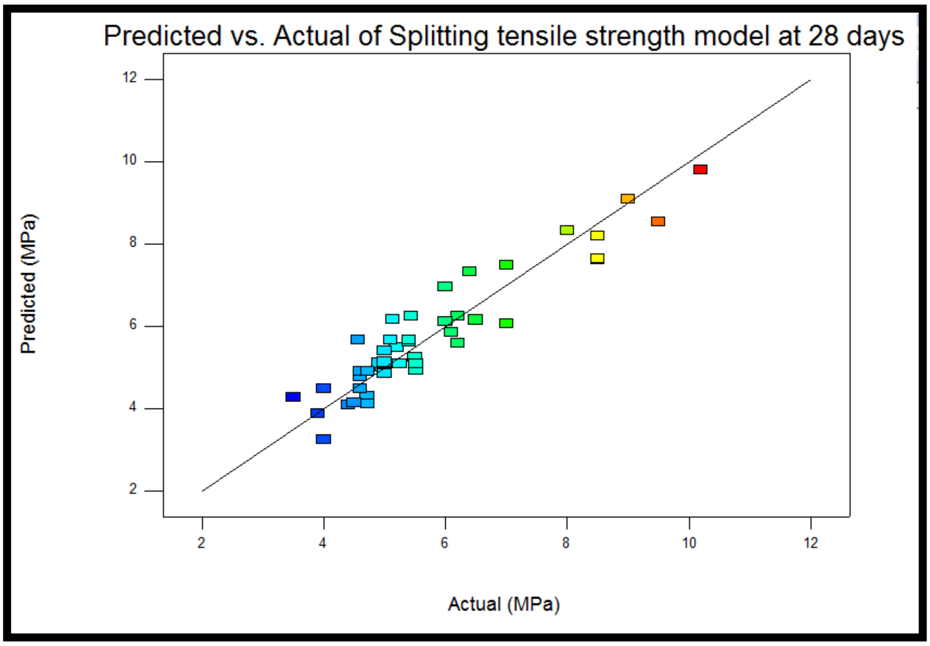

Figure 4.5: Prediction efficiency of offered model for splitting tensile strength

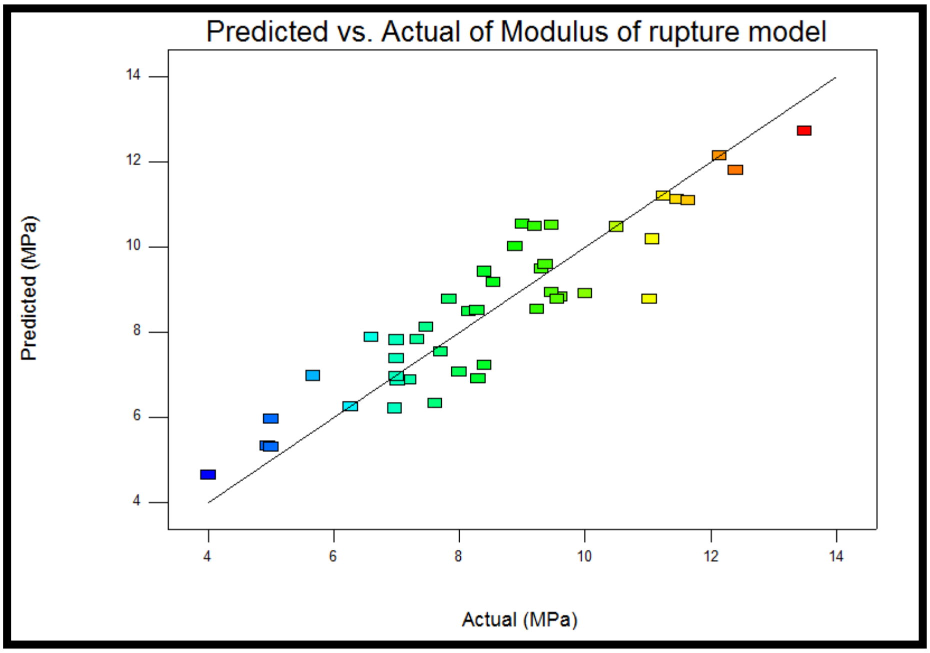

Figure 4.6: Prediction efficiency of offered model for modulus of rupture

Table 4.5 and Table 4.6 listed the finalized prediction models to reach the desired performance of compressive and tensile strength of UHPC in terms of real mixture ingredient. Probability factor is given for each parameter, in Table 4.5 and it is clear that linear B, C, and D which have high P-value are not statistically significant factors at the stipulated level of 5% for 7, 14, and 28-day compressive strengths. Moreover, linear A and E are statistically significant factors for all ages of compressive strength as shown in Table 4.5. The quadratic A, B, C, D, E are not statistically significant factors at the stipulated level of 5%, however, the quadratic A, B, C, and E are statistically significant factors at the stipulated level of 5% for 28-day compressive strength. The significance of some two-way interaction terms are given at 7, 14, and 28-day compressive strength in Table 4.5. A significant two-way interaction explains that the simple effect of a variable is not same at all levels of other variables. The 2-way interaction of A with B, D, E (AB, AD, AE), B with C and E (BC, BE), and C with D (CD) are statistically significant factors at the stipulated level of 10% for 7-day compressive strength. At 14 days compressive strength, 2-ways interactions of A with B and D (AB, AD), B with D (BD), and C with D (CD) are statistically significant factors at the stipulated level of 10%. Also at 28-day compressive strength, the 2-way interactions of A with B, D, E, (AB, AD, AE), and B with E (BE) are statistically significant factors at the stipulated level of 10%, those two-way interactions or quadratic variables which were not used in the given models were insignificant.

Table 4.5: Estimated parameters for models at 7, 14, 28-day compressive strength

	Compressive 7 days		Compressive 14 days		Compressive 28 days	
Parameters	Estimate	Prob > f	Estimate	Prob > f	Estimate	Prob > f
Constant	73.55		82.05		89.63	
A	-4.10	0.000183	-4.10	0.000208	-4.19	0.000419
B	0.51	0.600039	0.43	0.662289	0.99	0.358075
C	0.62	0.522755	-0.13	0.89202	-0.37	0.729417
D	-0.41	0.669074	-0.16	0.865862	-1.30	0.229922
E	-11.20	<0.0001	-11.46	<0.0001	-12.00	<0.0001
AB	2.91	0.006164	2.45	0.020269	3.72	0.001868
AD	-1.93	0.060132	-1.78	0.083709	-2.26	0.046967
AE	-1.92	0.061712	-1.42	0.164175	-1.85	0.100197
BC	-1.55	0.127327	-1.43	0.160621	-1.14	0.304248
BE	4.29	0.000154	5.39	0.188816	6.45	<0.0001
CD	-1.68	0.099306	-1.73	<0.0001	----	-----
BD	---	---	1.34	0.093411	-----	-----
A^2	2.04	0.559331	4.86	0.155532	7.85	0.048624
B^2	-3.21	0.361439	-4.24	0.212705	-7.95	0.046197
C^2	---	----	----	----	3.95	0.309034
D^2	-4.36	0.217797	-4.39	0.197407	-8.15	0.041307
E^2	2.09	0.549816	----	----	-----	----

Estimated Parameters within probability values for splitting tensile strength and modulus of rupture are given in Table 4.6 and it is clear that linear factors A, B, C, D, E are statistically significant factors at the stipulated level of 10% with having probability value of 0.06, 0.005, 0.0001, <0.0001, <0.0001 respectively, for splitting tensile strength and 0.003, 0.002, 0.0007, 0.004, <0.0001, respectively, for rupture modulus. The quadratic B, C for splitting tensile strength and quadratic B for rupture modulus are statistically significant factors at the stipulated level of 10%. About 2-ways interactions, as it is given in table 4.6, the interaction between A and B (AB), B and E (BE), C and E (CE), and, D with E (DE) are statistically significant factors at the stipulated level of 10% for splitting tensile strength, and interaction between C and D (CD) is statistically significant factor at the stipulated level of 5%.

Table 4.6: Estimated parameter of obtained models for splitting tensile strength and modulus of rupture

	Splitting Tensile strength		Modulus of Rupture	
Parameters	estimate	Prob>f	estimate	Prob>f
Constant	5.67		8.77	
A	-0.23	0.063382	-0.57	0.002885
B	-0.36	0.004972	-0.58	0.002424
C	0.53	0.000141	0.66	0.000715
D	-0.65	<0.0001	-0.54	0.004209
E	-1.00	<0.0001	-1.71	<0.0001
AB	0.26	0.044944		
AD	-0.13	0.299533	----	-----
AE	----	----	0.24	0.195130
BD	0.14	0.279485		
BE	0.48	0.000508	-----	----
CD	0.12	0.340114	0.42	0.025262
CE	-0.33	0.011287	----	----
DE	0.27	0.035389	-0.25	0.182927
A^2	-0.32	0.481555	-0.83	0.197593
B^2	-1.17	0.013697	-1.24	0.059639
C^2	2.33	0.000014	1.05	0.108133
D^2	-0.17	0.711158	0.70	0.274339
E^2	-0.56	0.219598	------	-----

Figure 4.7 and Figure 4.8 show the contours effect of cement and silica fume amount and also effect of SP amount and steel fibers on 7-day compressive strength, respectively. As it is clearly shown, increasing rate of silica fume from 0.15 to 0.3 of aggregate mass decreased the 7-day compressive strength where the amount of SF was changed from 15% to 43% (by weight of cement). Šerelis et al. (20015) found that the best ratio for silica fume was 15% (by weight of cement) for UHPC, moreover, Figure 4.7 shows that the rate of cement was not very significant. So increasing the cement is not effective on 7-day compressive strength as Aldahdooh et al. (2013) reported increasing the binder will not enhance the strength because the capillary porosity will increase by increasing the amount of cement. Effect of steel fiber with SF is given in Figure 4.8 that shows: effect of steel fiber is negligible for increasing the 7-day

compressive strength as Salam (2015) found that there is a small improvement in compressive strength by adding fibers.

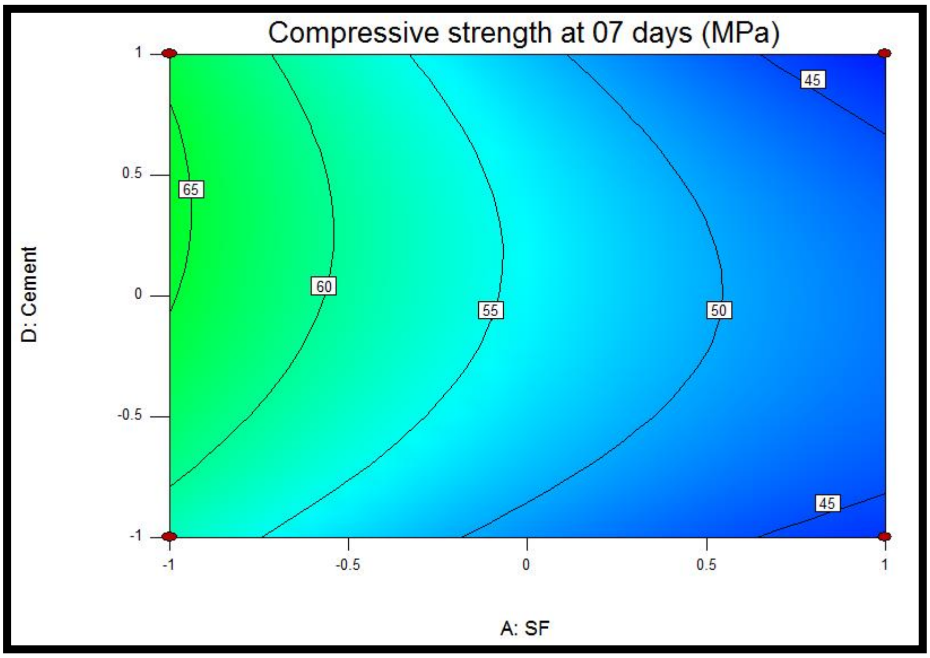

Figure 4.7: Contour plot of 7-day compressive sttength changes, X1=SF amount and X2=cement amount

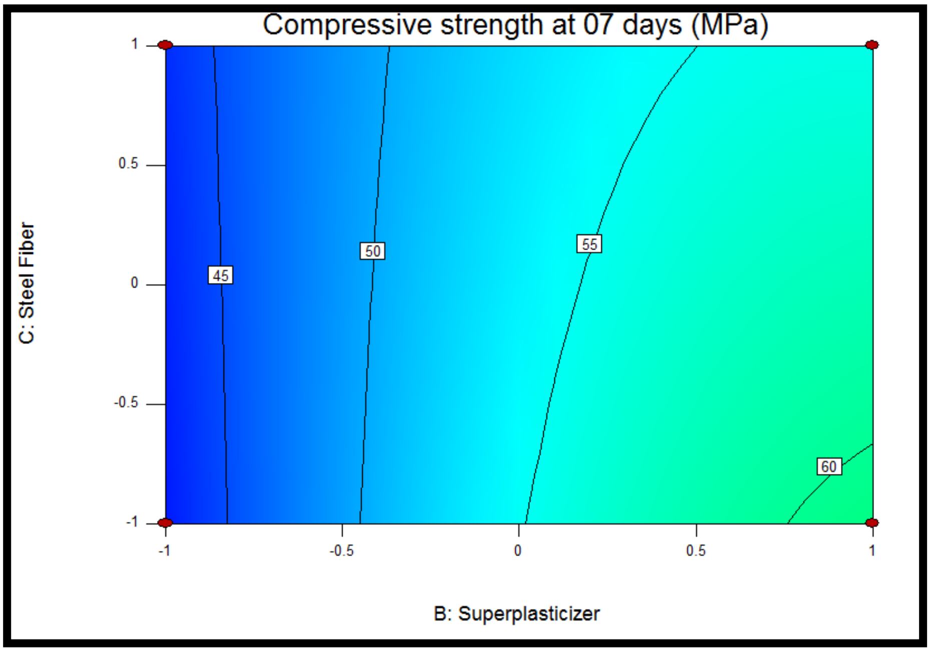

Figure 4.8: Contour plot of 7-day compressive sttength changes, X1=steel fiber amount and X2=superplasticizer amount

The combiation effects of SF and w/c ratio is given in Figure 4.9. Decreasing the w/c ratio and amount of silica fume increases the 7-day compresive strength significantely. There is a common belief that decreasing the w/c ratio increases the compressive strength of concrete. The effect of w/c ratio with superplsticizer on 7-day compressive strength are inversely correlated which is shown in Figure 4.10. The effect of only superplasticizer is not very significant on 7 days compressive strength as shown in the given models but the correlation between superplasticizer and w/c ratio was found very meaningful and effective on 7 days compressive strength.

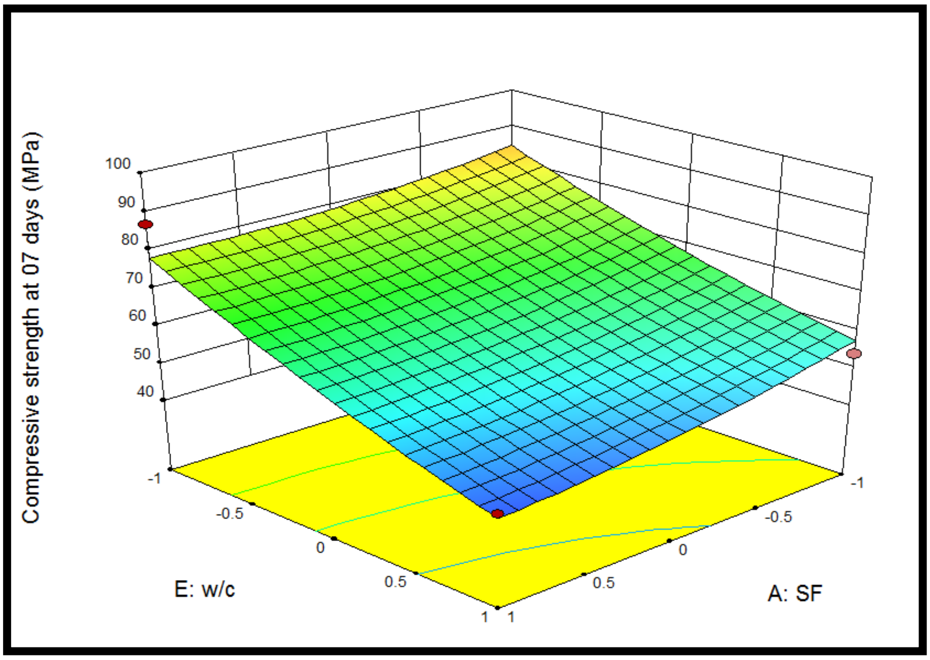

Figure 4.9: Response surface plot of X1 = SF amount, X2 = w/c, SP = -1, Fiber = -1 and Cement = -1 on 7 day compressive strength

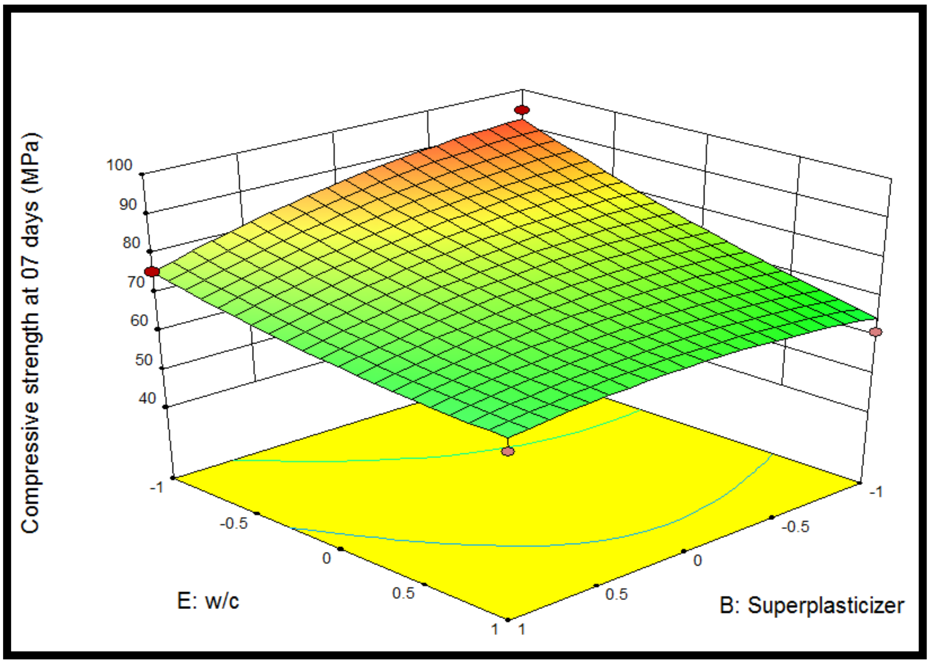

Figure 4.10: Response surface plot of X1 = Superplasticizer amount, X2 = w/c, SF = -1, Fiber = 1 and Cement = 1 on 7 day compressive strength

The contours effect of w/c ratio and amount of silica fume and contours effect of SP amount and steel fibers on 14 days compressive strength are shown in Figure 4.11 and Figure 4.12, respectively. An increase of silica fume from 0.15 to 0.3 of aggregate mass decreases at the 14 days compressive strength where amount of SF started from 15% to 43% (by weight of cement). 14-day compressive strength is increased by decreasing the w/c ratio which were defined in this study between 0.18 and 0.32 with decreasing the porosity which is shown in Figure 4.11. Moreover, Figure 4.12 demonstrates that the steel fiber amount is not very significant on 14-day compressive strength, thus, increasing the fiber content is not effective on 14 days compressive strength. Model shows that increasing the superplasticizer amount (0.04 to 0.08 of aggregate mass) has a direct effect on 14-day compressive strength.

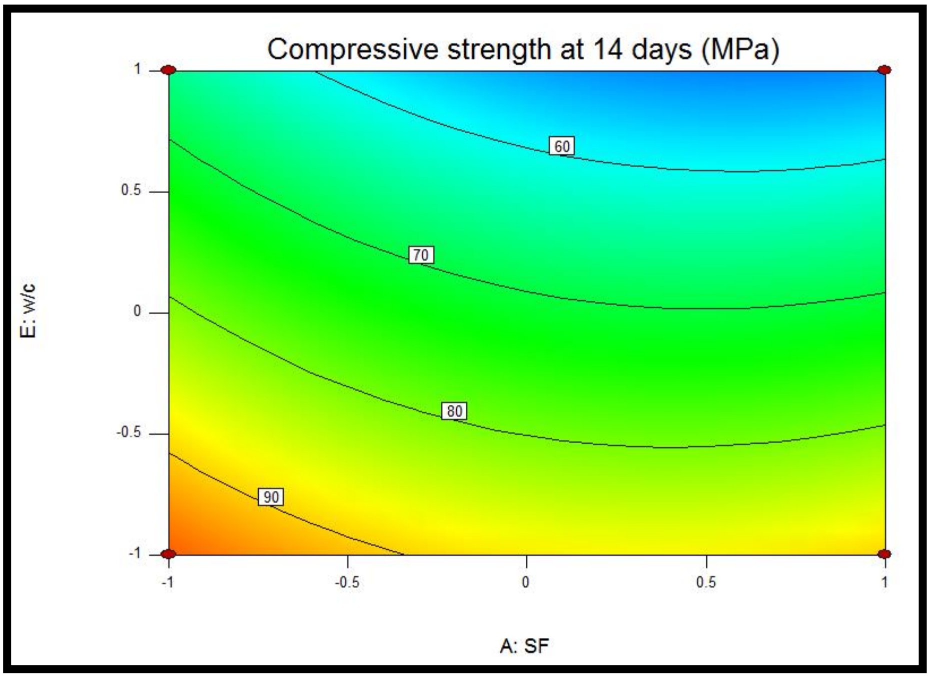

Figure 4.11: Contour plot of 14 day compressive sttength changes, X1 = SF amount and X2 = w/c ratio

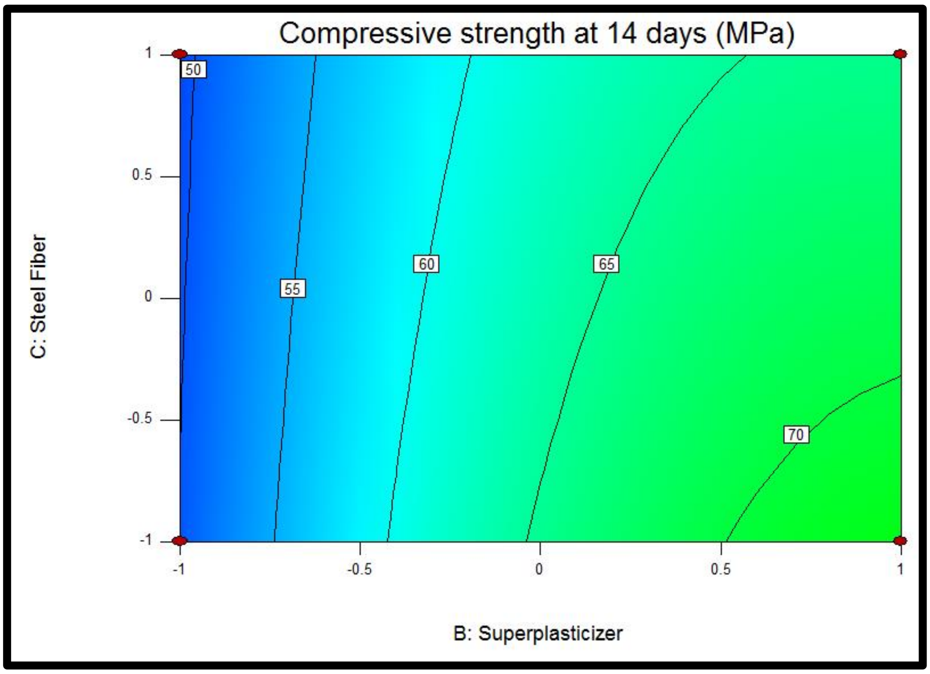

Figure 4.12: Contour plot of 14 day compressive sttength changes, X1 = Superplasticizer amount and X2 = steel fiber amount

The response surface of 14-day compressive strength is given in Figure 4.13 and Figure 4.14. As shown in Figure 4.13, the response surface due to cement amount and superplasticizer has increased at low level of superplasticizer amount, and 14 day compressive strength was negligible increased in the middle level of cement amount. A decrease in superplasticizer and w/c ratio increases 14-day compressive strength which is given in Figure 4.14. Schmidt (2004), reported that increasing of compressive strength due to reducing the w/c ratio is because of decreasing of capillary pore volume. Adding extra superplasticizer amount can segregate the concrete therefore the strength will be reduced. Thus, in Figure 4.14, the maximum strength is at low level of w/c ratio and superplasticizer amount.

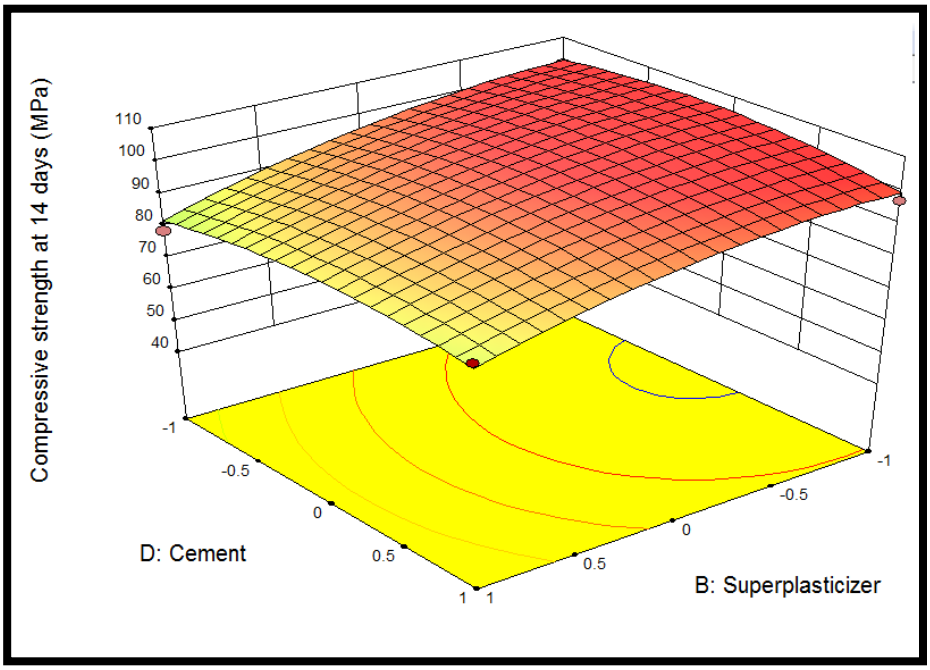

Figure 4.13: Response surface plot of X1 = SP amount, X2 = Cement, SF = -1, Fiber = 1 and w/c = -1 on 14 day compressive strength

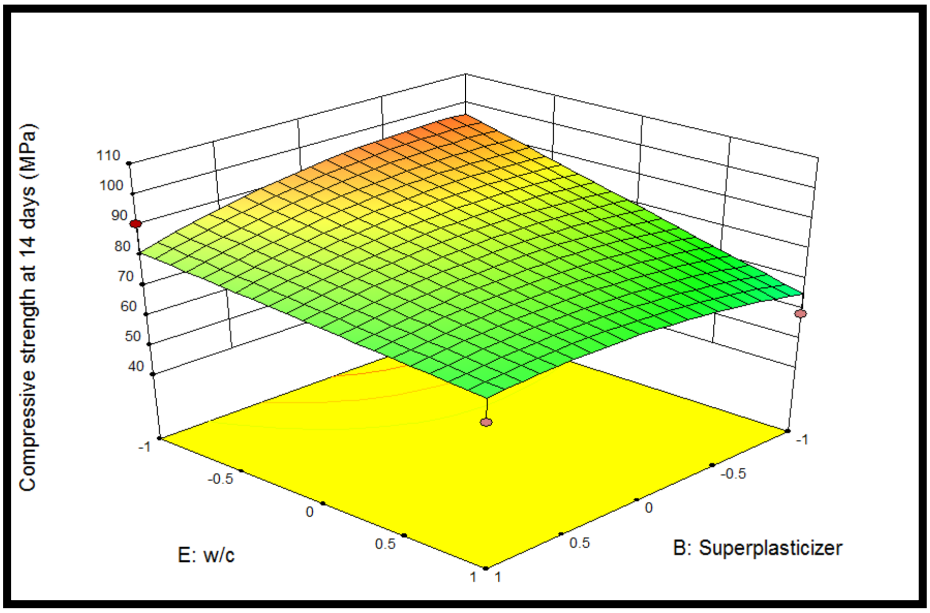

Figure 4.14: Response surface plot of X1 = Superplasticizer amount, X2 = w/c, SF = -1, Fiber = -1 and Cement = -1 on 14-day compressive strength

Figure 4.15 shows the contour effect of amount of silica fume and w/c ratio on 28-day strength. Therefore, it is clear that decreasing w/c ratio has a significant effect on 28-day compressive strength. Silica fume content has inversely effected 28-day compressive strength where by decreasing amount of silica fume, 28 days strength is reduces. The corrolation between w/c ratio and SP amount is significant as it is shown in Figure 4.16. The highest 28 days compressive strength crosses the low level SP and w/c ratio. In Figure 4.17, the interaction between amount of silica fume and OPC is plotted. A decrease of SF improves the 28-day compressive strength as Ghafari et al. (2015) reported in their model. Cement content increment (0.7 to 1.3 by weight of aggregate) was not significant at 28-day compressive strength.

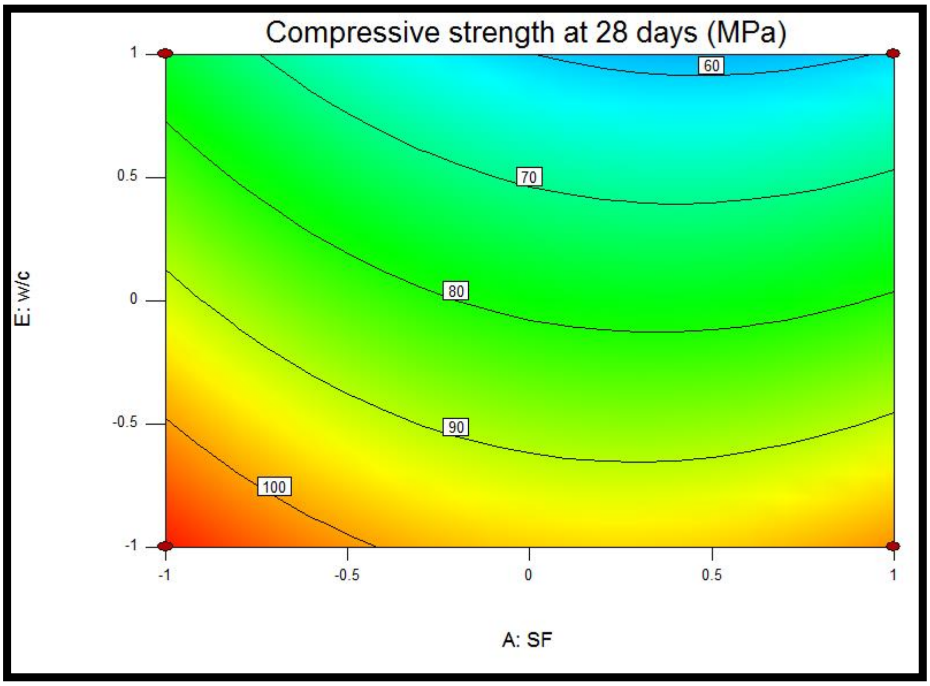

Figure 4.15: Contour plot of 28 day compressive sttength changes, X1 = SF amount and X2 = w/c ratio

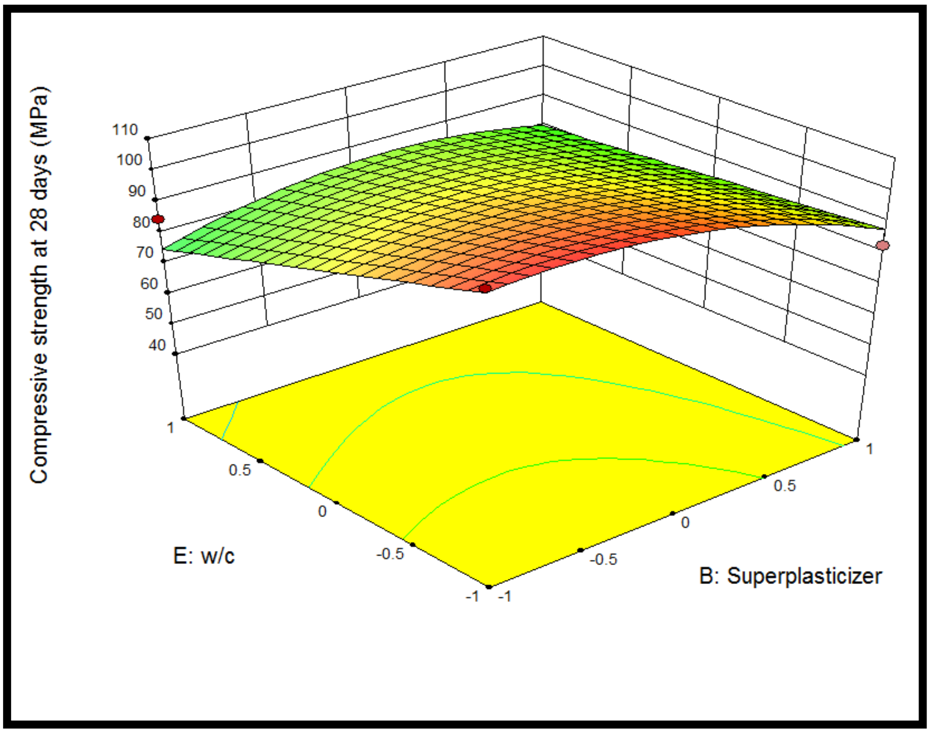

Figure 4.16: Response surface plot of X1 = Superplasticizer amount, X2 = w/c, SF = -1, Fiber = 1 and Cement = -1 on 28 day compressive strength

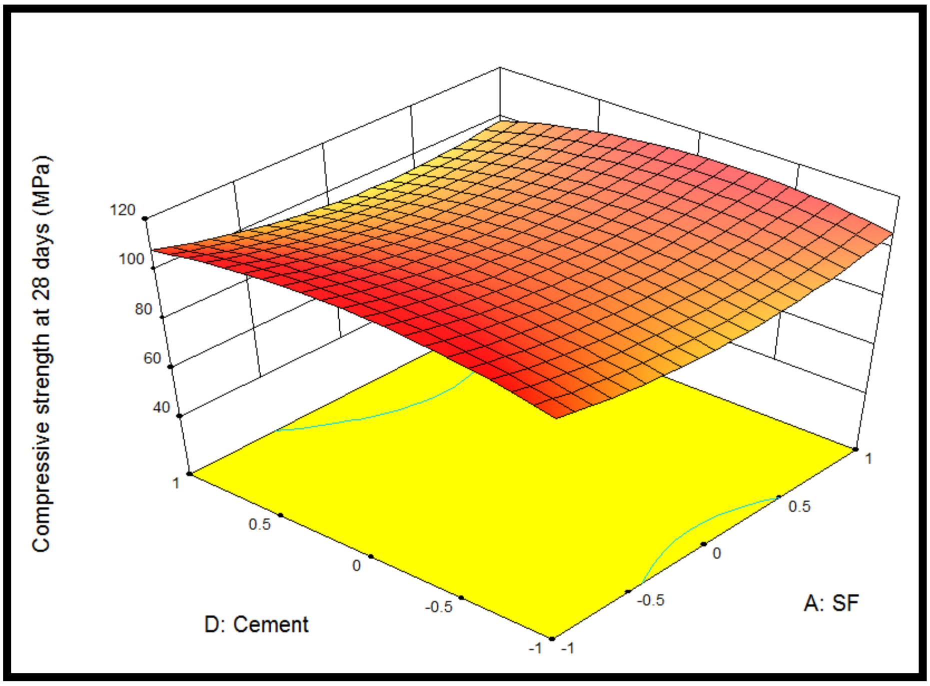

Figure 4.17: Response surface plot of X1 = SF amount, X2 = Cement, Superplasticizer = 0, Fiber = 1 and w/c = -1 on 28 day compressive strength

According to Figure 4.18, decreasing w/c ratio from 0.32 to 0.18 significantly improves of splitting tensile strength. Senthil Kumar & Baskar (2014), modeled the inverse effect of w/c ratio on splitting tensile strength. Amount of cement inversely affected on tensile strength, by increasing amount of cement and decreases recorded for splitting tensile strength as plotted in Figure 4.18 and Figure 4.19. Decreasing SF content increases the splitting tensile strength as shown in Figure 4.19.

Figure 4.20 shows the interaction of steel fiber (0.1 to 0.2 by weight of aggregate) and w/c ratio (0.18 to 0.32). It can be seen that both have significant role in splitting tensile strength improvement in the studied range. As can be seen, Figure 4.20 shows, the maximum splitting tensile is achieved by crossing of lowest level of w/c ratio and highest level of steel fiber content. It is a well-known that the splitting tensile strength increases by increasing amount of steel fibers. The interaction of cement amount and superplasticizer is given in Figure 4.21 which shows clearly that by decreasing the cement amount (0.7 to 1.3 weight of aggregate) and superplasticizer (0.04 to 0.08 weight of aggregate), maximum splitting tensile strength is obtained.

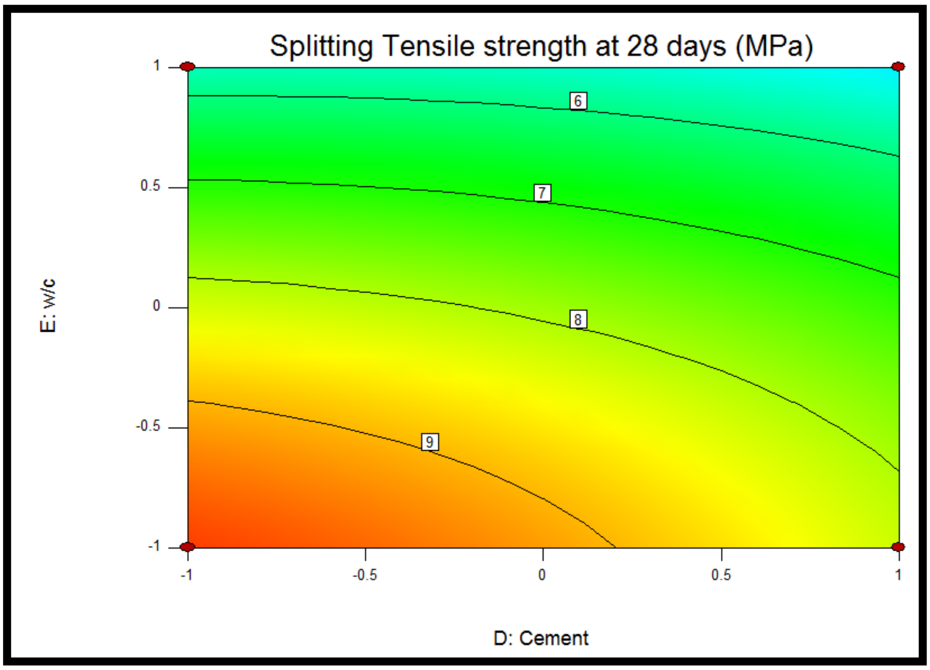

Figure 4.18: Contour plot of 28 day compressive strength changes, X1= Cement amount and X2 = w/c ratio

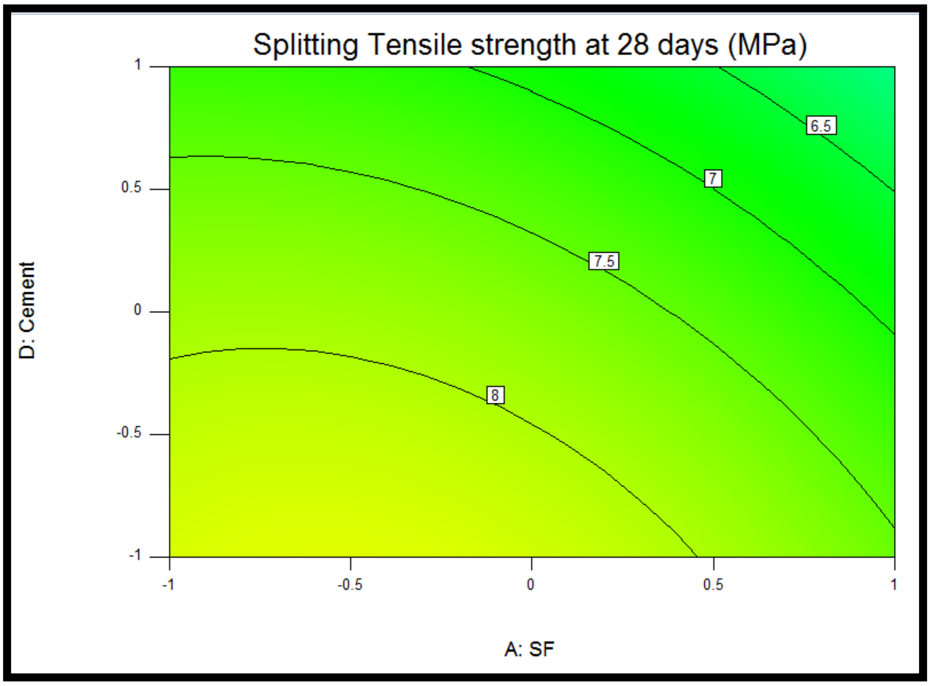

Figure 4.19: Contour plot of splitting tensile strength changes, X1 = SF and X2 = Cement

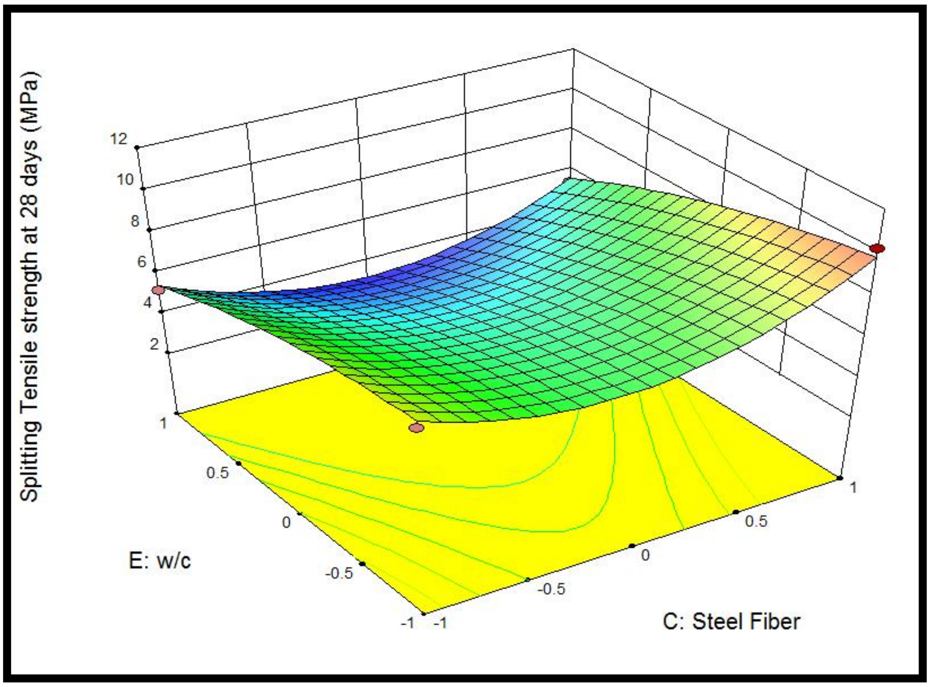

Figure 4.20: Response surface plot of X1 = Fiber amount, X2 = w/c, SP = -1, SF= -1 and Cement = -1 on splitting strength

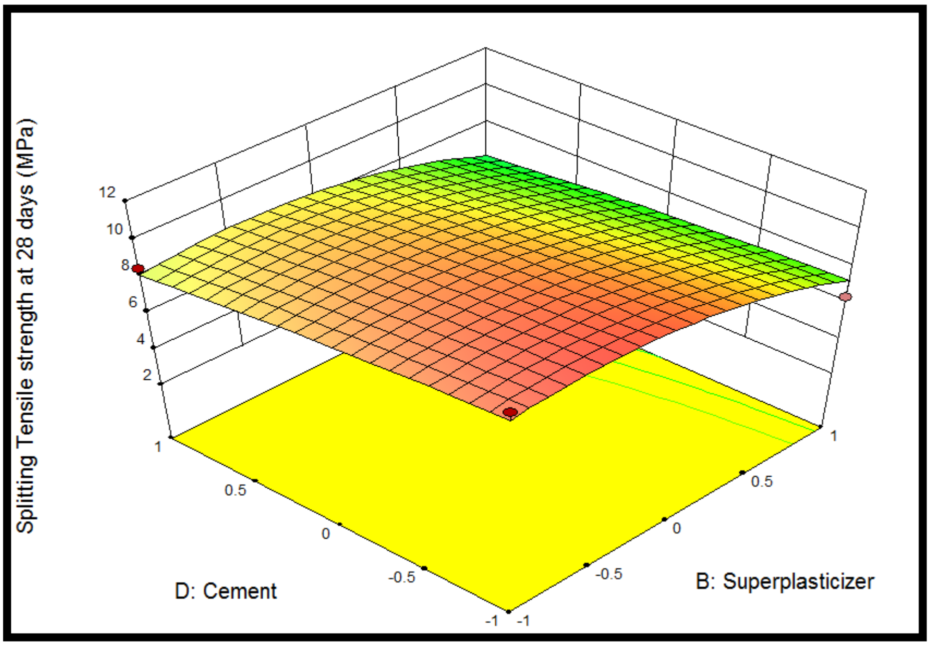

Figure 4.21: Response surface plot of X1 = SP, X2 = Cement, SF = -1, Fiber = 1 and w/c = -1 on splitting strength

It is clear that w/c ratio is very effective on modulus of rupture of UHPC according to Figure 4.22 and Figure 4.23. By decreasing w/c ratio (from 0.32 to 0.18) the modulus of rupture increases. Also, by decreasing the silica fume amount (from 0.20 to 0.30 by weigh of sand), the modulus of rupture increases (Figure 4.22). Decreasing the cement content from 1.30 to 0.70 improves the modulus of rupture of UHPC as shown in Figure 4.23.

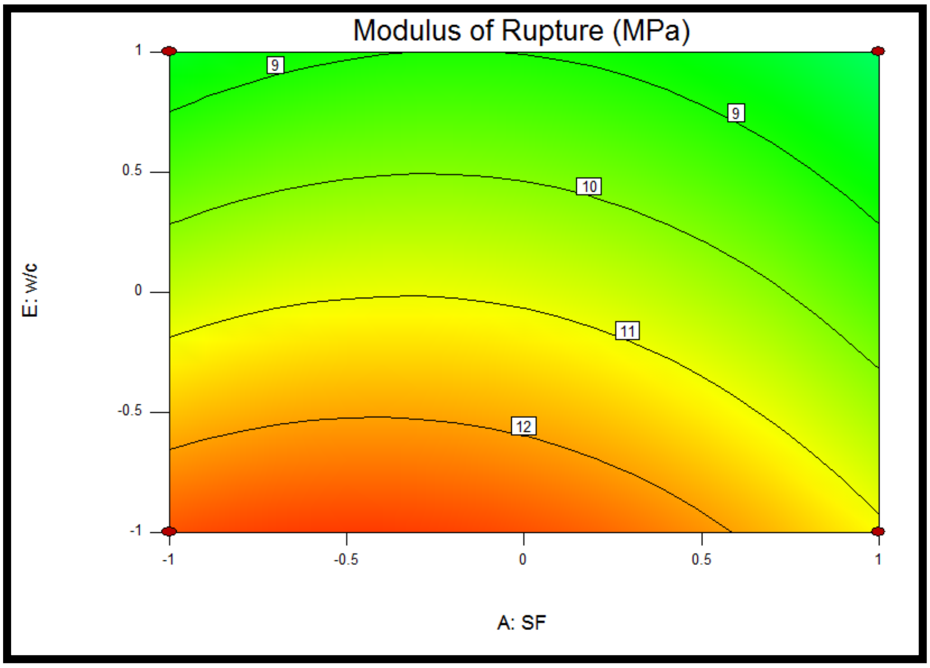

Figure 4.22: Contour plot of rupture module changes, X1 = SF amount and X2 = w/c ratio

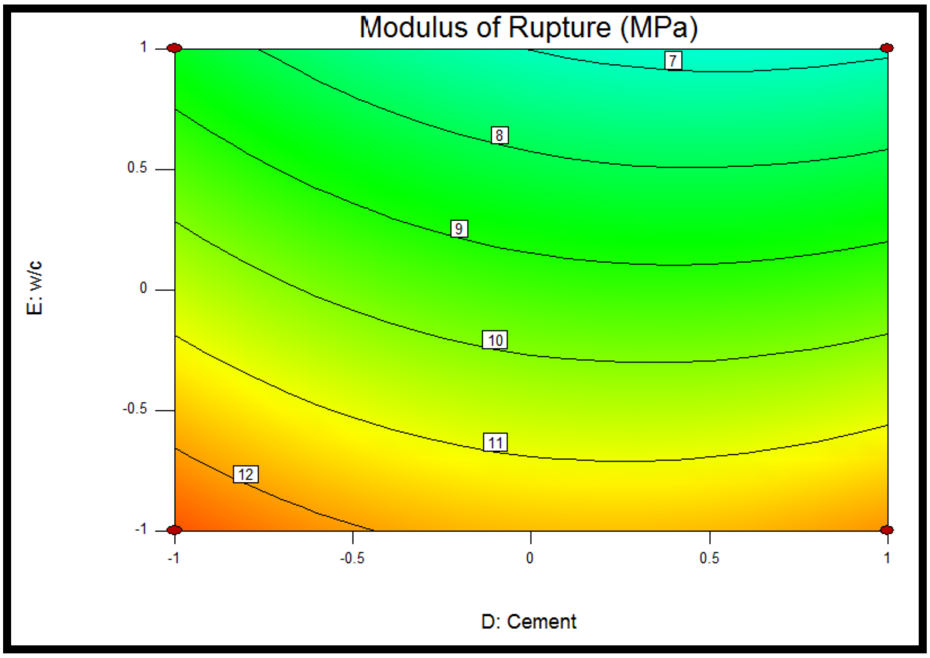

Figure 4.23: Contour plot of rupture module changes, X1 = Cement amount and X2 = w/c ratio

The response surface of modulus of rupture between the variables of steel fiber and superplasticizer amount is shown in Figure 4.24. It is obvious that the modulus of rupture increases by increasing steel fiber content from 0.1 to 0.2 (by weight of aggregate) and decreasing superplasticizer amount from 0.08 to 0.04 (by weight of aggregate). The interaction of superplasticizer and w/c ratio on modulus of rupture was given in Figure 4.25. It is derived that the interaction of w/c ratio and superplasticizer amount is very significant for modulus of rupture, the lowest level of superplasticizer and lowest level of w/c ratio result in maximum modulus of rupture as shown in Figure 4.25. Thus, by reducing the rate of superplasticizer from 0.08 to 0.04 (by aggregate mass) and decreasing the w/c ratio from 0.32 to 0.18, the modulus of rupture is increases.

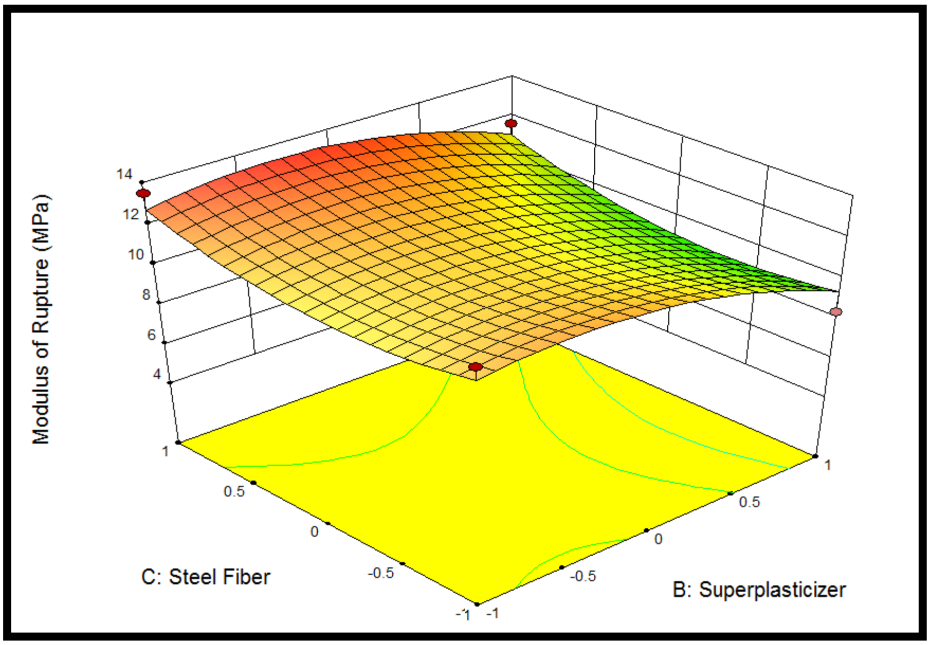

Figure 4.24: Response surface plot of X1 = SP, X2 = Fiber, SF = -1, Cement = -1 and w/c = -1 on rupture module

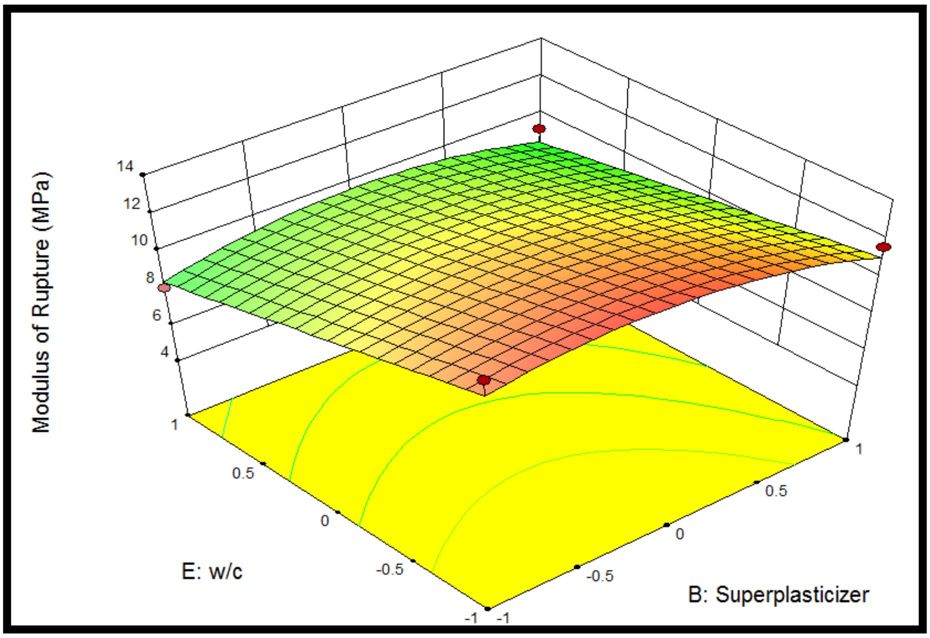

Figure 4.25: Response surface plot of X1 = SP, X2 = w/c, SF = -1, Fiber = 1 and Cement = -1 on rupture module

4.3.2 Flexural Toughness

The effects of five variables (silica fume content, superplasticizer content, steel fiber content, OPC content, and w/c ratio) on flexural toughness of UHPC was analyzed by using the response surface method. For producing the model 45 points (results) were selected such as 32 points for model making, 3 points for replication, and 8 points to consider the lack of fit.

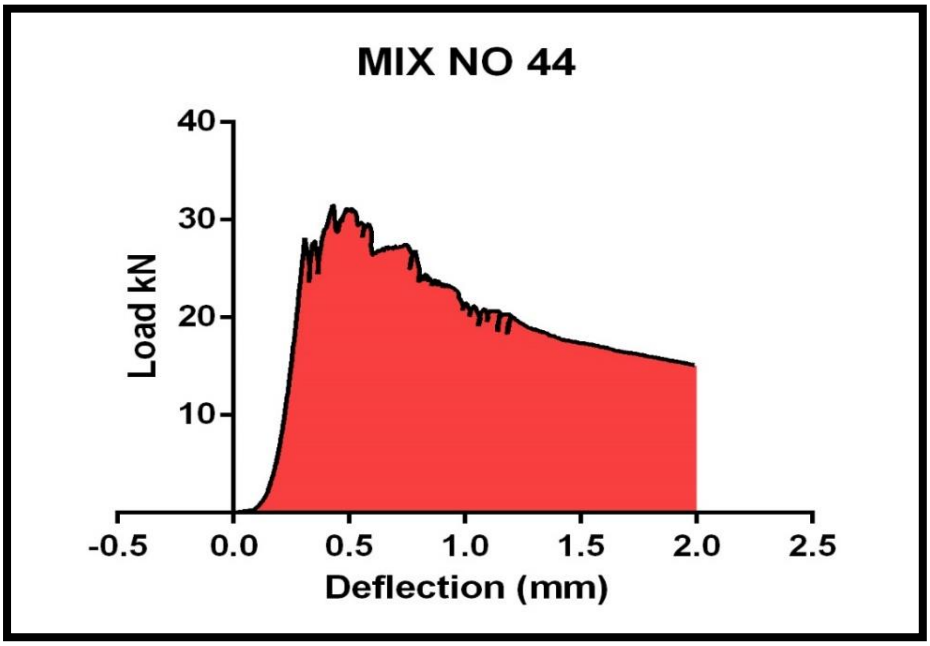

Figure 4.26: Load- deflection of mix No 44

Flexural toughness is obtained from the area under the load versus net deflection curve when deflection changes from 0 to 2 mm which is given as an example load-deflexion curve of mix no 44 is shown in Figure 4.26. In order to calculate the area under the load-deflection curve, Prism GraphPad Software Version 6.0 was used. Table 4.7 shows the results of using five different variables on the mechanical properties of UHPC. Each result was derived by the average of 3 specimens. This experiment was conducted in accordance with ASTM C1609 (2006).

Table 4.7: Mix design amounts and flexural toughness of UHPC

Mix no	Sand (kg)	Silica Fume (kg) A	Super-plasticizer (kg) B	Steel Fiber (kg) C	Cement (kg) D	Flexural Toughness (kN.mm) Y
1	50	7.5	4.0	5.0	35	39.24
2	50	15.0	2.0	5.0	65	17.56
3	50	15.0	2.0	10.0	35	43.28
4	50	10.0	3.0	10.0	50	38.00
5	50	15.0	2.0	5.0	35	30.36
6	50	10.0	3.0	7.5	50	39.00
7	50	7.5	4.0	5.0	35	51.11
8	50	15.0	2.0	10.0	65	35.51
9	50	7.5	3.0	7.5	50	40.00
10	50	7.5	2.0	10.0	35	57.00
11	50	10.0	2.0	7.5	50	32.48
12	50	15.0	4.0	5.0	65	15.69
13	50	10.0	3.0	7.5	65	38.20
14	50	15.0	2.0	10.0	35	36.85
15	50	15.0	2.0	10.0	65	21.18
16	50	7.5	2.0	5.0	65	15.00
17	50	10.0	3.0	7.5	50	40.59
18	50	7.5	2.0	10.0	65	44.64
19	50	7.5	2.0	5.0	65	31.85
20	50	10.0	3.0	7.5	50	32.00
21	50	15.0	2.0	5.0	35	11.93
22	50	10.0	3.0	7.5	50	40.00
23	50	7.5	4.0	5.0	65	13.00
24	50	15.0	4.0	10.0	65	48.00
25	50	15.0	4.0	10.0	65	25.00
26	50	7.5	4.0	10.0	35	61.00
27	50	7.5	2.0	10.0	65	56.25
28	50	7.5	2.0	10.0	35	82.00
29	50	15.0	4.0	5.0	35	34.00
30	50	15.0	4.0	10.0	35	27.39
31	50	7.5	2.0	5.0	35	58.74
32	50	15.0	4.0	10.0	35	33.88
33	50	15.0	2.0	5.0	65	34.45
34	50	7.5	2.0	5.0	35	29.00
35	50	15.0	4.0	5.0	65	46.89
36	50	7.5	4.0	10.0	65	24.00
37	50	7.5	4.0	10.0	35	39.62
38	50	7.5	4.0	5.0	65	47.30
39	50	15.0	4.0	5.0	35	40.62
40	50	10.0	4.0	7.5	50	28.00
41	50	15.0	3.0	7.5	50	30.06
42	50	10.0	3.0	7.5	35	46.78
43	50	10.0	3.0	5.0	50	36.45
44	50	10.0	3.0	7.5	50	27.78
45	50	7.5	4.0	10.0	65	40.00

The interaction and correlation between the variables and the flextural toughness was calculated by ANOVA (analysis of variance). For the modeling, linear model, two-factor interaction, and quadratic models were considered to find best predictive model. In each model, the significant parameters were detected and then, by the backward elimination technique the insignificant terms were eliminated and the final regressions for each were performed. Consequently, the quadratic model was selected for response. The quality of prediction models were determined by coefficient of multiple determination R^2, which shows the total deviation of the variables from the prediction model. The p-value (probability of errors) with 95% confidence level and statistical significant test at 5% and also lack of fit with p-value greater than 0.05 was performed for model validations.

Table 4.8 shows the ANOVA results for the response parameters. The results illustrated that the model was significant at the 5% confidence level because the P values was less than 0.05. Furthermore, the large p-value of 0.59 for lack of fit (>0.05) of response demonstrates that the F-value was not significant, implying significant model correlation between the variables and process response. The model coefficient of determination R^2 has a reliable confidence with 0.85. The predicted R^2 of 0.63 in reasonable agreement with adjust R^2 of 0.78, whereas, the difference is less than 0.2.

Table 4.8: Analysis result of regression models

Response	R^2	Adj-R^2	Pre-R^2	F-Value	Lack of fit	Model P-value
Flexural toughness	0.85	0.78	0.63	11.97	0.59	<0.0001

Figure 4.27 shows the normal plot of the residual value of flexural toughness, which was used to determine the model satisfactoriness. Based on the adequacy of the model, the residuals from the least square fit were important, as shown in Figure 4.27. The constructed plot of the studentized residual versus the normal percentage of probability was satisfied because flexural toughness residual plot agreed well with the straight line, as shown in Figure 4.28. Consequently, it could be mentioned that this model is reliable enough.

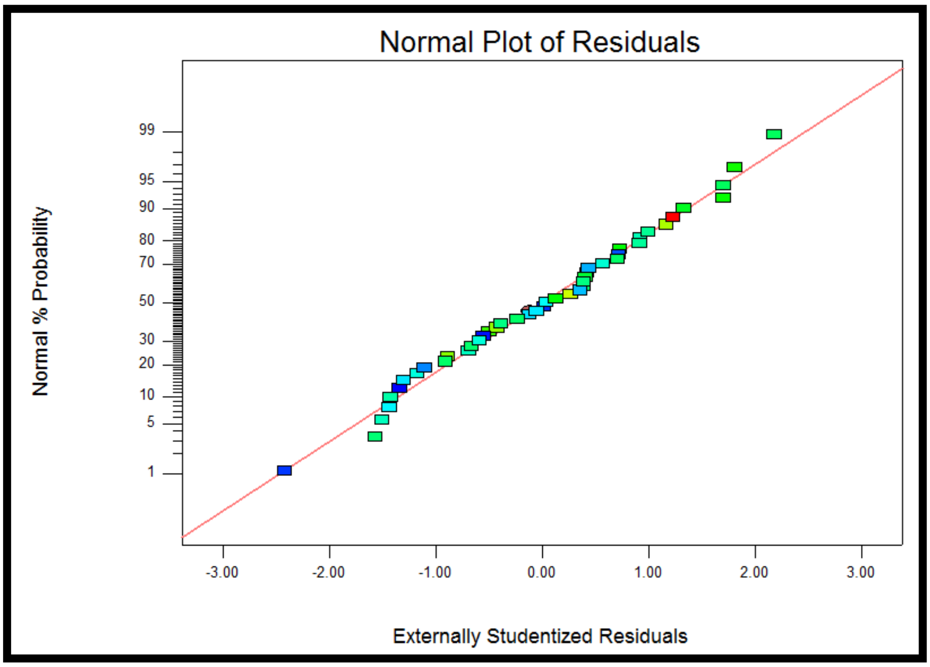

Figure 4.27: Normal plot of residual value of flexural toughness

Table 4.9 listed the finalized prediction model to reach the desired performance of flexural toughness of UHPC. Probability factor is given for each parameter, in Table 4.9, it is clear that linear B with a high P-value are not statistically significant factors at the stipulated level of 5%. Moreover, linear A, B, C and E are statistically significant factors Table 4.9. The significant of some two-way interaction terms are given in Table 4.9. A significant two-way interactions explain that the simple effect of a variable is not the same at all levels of other variables. The 2-way interaction of A with B, C, D

(AB, AC, AD), B with C (BC), and D with E (DE) are statistically significant factors at the stipulated level of 10% for Flexural strength. The quadratic value for A, D, E are significantly important.

Table 4.9: Parameter estimated for model

	Con	A	B	C	D	E	AB	AC	AD	AE	BC	DE	B^2	D^2	E^2
MODEL ESTIMATE	35.64	-5.8	-0.6	4.72	-4.9	-8.1	3.1	-2.4	4.1	1.7	-4.2	-1.6	-6.70	5.5	2.9
Prob > F	-----	0.0	0.5	0.00	0.0	0.0	0.0	0.0	0.0	0.1	0.0	0.1	0.09	0.16	0.5

The effect of five concrete mix design parameters (amount of silica fume, amount of superplasticizer, amount of cement, amount of steel fiber, and w/c ratio) on flexural toughness has been considered employing response surface methodology. The effects of variables on response can be presented graphically by 3D plotting of response value versus variables.

Figure 4.28 and Figure 4.29 show the contours effect of SF and steel fiber amount and also effect of SP amount and steel fibers at fixed variables on flexural toughness, respectively. As it is clearly shown, increasing rate of silica fume from 0.15 to 0.3 of fine aggregate mass decreases the flexural toughness where the amount of SF was changed from 15% to 43% (by weight of cement). Effect of steel fiber with SF is given in Figure 4.28 shows that the effect of steel fiber is very significant for increasing the energy absorption (Pyo et al. 2015). Effect of SP is variable, by increasing the SP amount the toughness is increases until it reaches to a code of around 0, beyond this level of SP toughness decreases. Alsadey (2012) reported that this phenomenon happen since over dosage of SP will cause segregation and bleeding, affecting the uniformity and cohesiveness of the UHPC mixture. Therefore, flexural toughness will reduce if the dosage is beyond the optimum amount. Consequently, the energy absorption will be decreased by segregation of concrete ingredients as well as fibers.

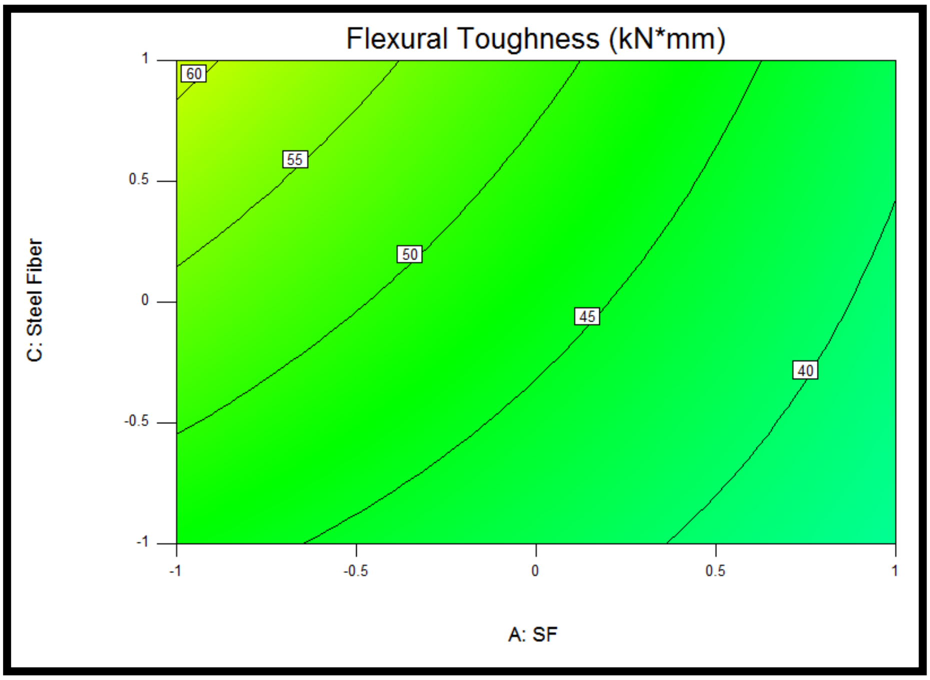

Figure 4.28: Contour plot of flexural strength changes, X1=SF amount and X2=Steel fiber

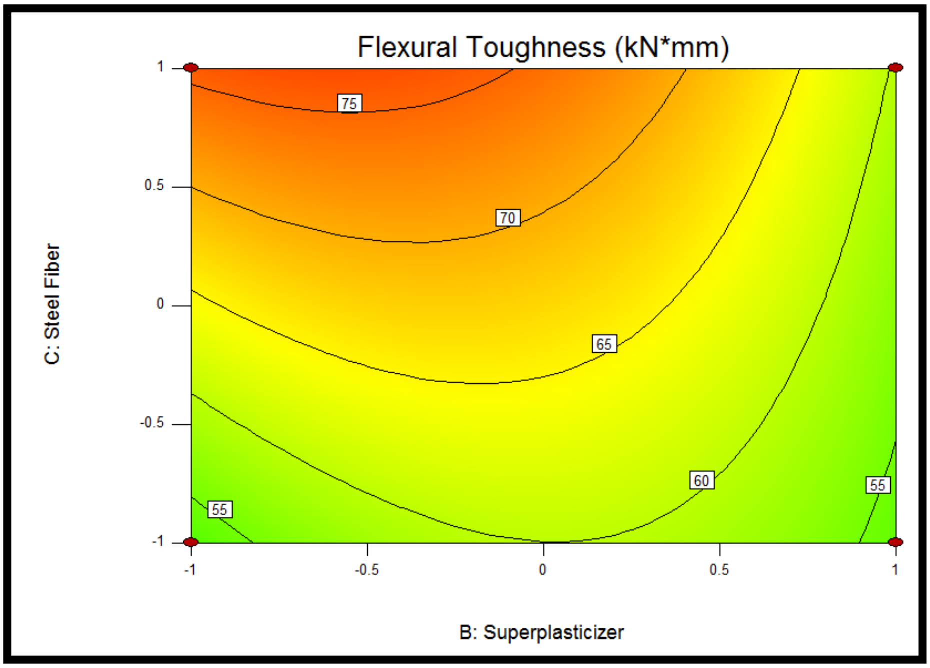

Figure 4.29: Contour plot of flexural toughness changes, X1=superplasticizer amount and X2=steel fiber

The combined effects of cement and SF ratio are given in Figure 4.30. Decreasing the w/c ratio and amount of silica fume and cement causes a significant increase in the energy absorption (Wille & Boisvert-Cotulio, 2015). The effect of SF with superplsticizer on toughness is inversly corrolated as shown in Figure 4.30. The effect of superplasticizer is not very significant on toughness as shown in Figures 4.30 and 4.31.

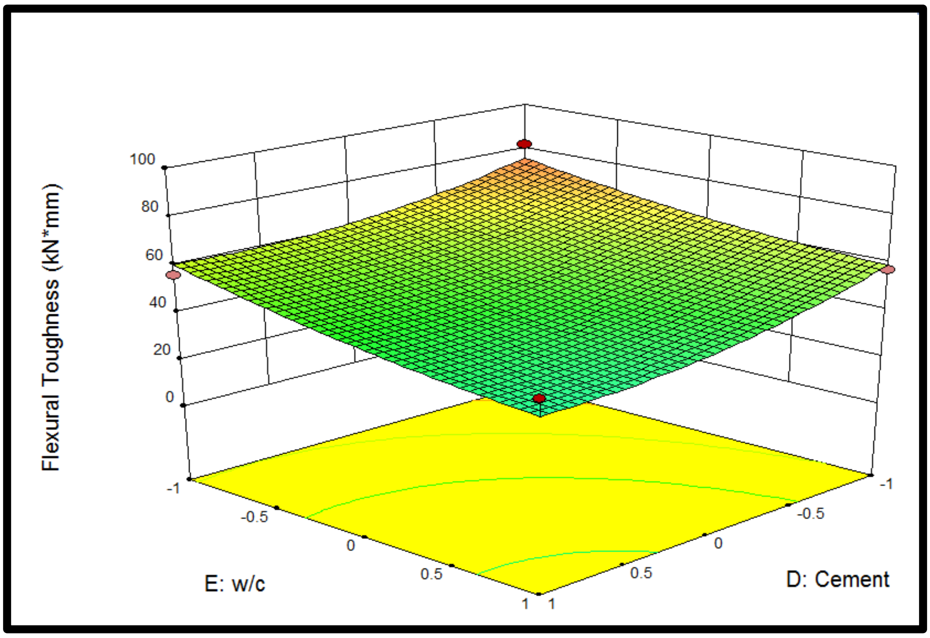

Figure 4.30: Response surface plot of X1 = Cement amount, X2 = w/c, SF = -1, Superplasticizer = -1 and steel fiber= 1 on flexural toughness

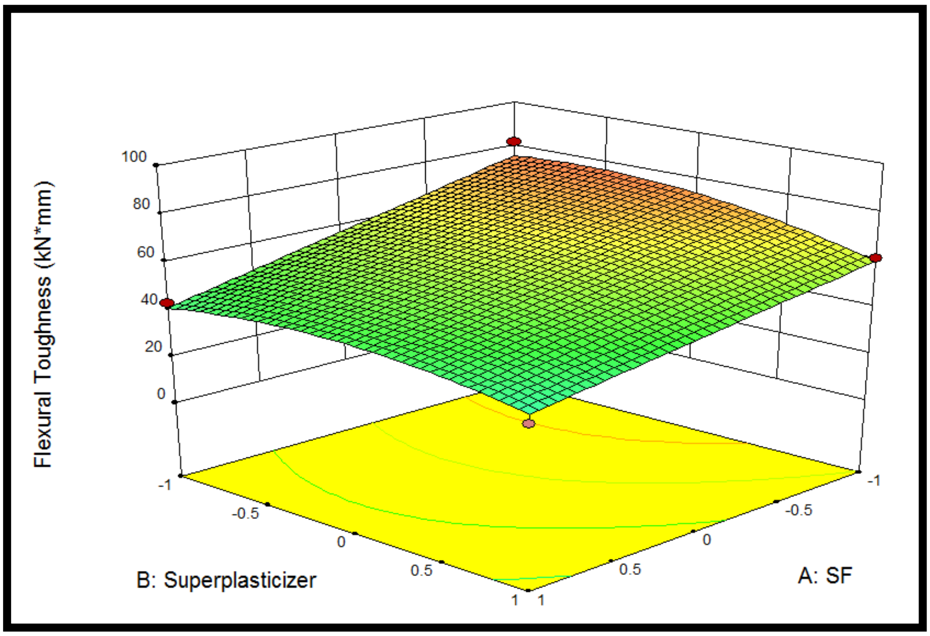

Figure 4.31: Response surface plot of X1 = SF, X2 = Superplasticizer amount, Steel fiber = 1, Cement amount = -1 and w/c = -1 on flexural toughness

4.3.3 Density

Table 4.10 shows the results of density and compressive strength at 7, 14, 28 days with using five different variables. Each result was derived by average of 3 specimens.

Table 4.10: Mix proportions and responses of UHPC

Mix no	Sand (kg)	Silica Fume (kg) A	Super-plasticizer (kg) B	Steel Fiber (kg) C	Cement (kg) D	Water (kg) E	Compressive strength (MPa)			Density (kg/m^3)		
							7	14	28	7	14	28
1	50	7.5	4	5	35	13.6	58	65	71	2120	2100	2070
2	50	15	2	5	65	25.6	42	48	49	1980	1990	1980
3	50	15	2	10	35	9	88.5	96.3	103.2	2560	2570	2590
4	50	10	3	10	50	13.5	75	86	97	2330	2310	2330
5	50	15	2	5	35	9	87	91	97	2400	2370	2380
6	50	10	3	7.5	50	10.8	66	91	102	2375	2340	2385
7	50	7.5	4	5	35	7.65	77.8	91	102	2380	2390	2410
8	50	15	2	10	65	14.4	95	103	110	2480	2450	2470
9	50	7.5	3	7.5	50	12.9375	81.5	94	106	2370	2375	2395
10	50	7.5	2	10	35	13.6	76	83	85	2210	2200	2200
11	50	10	2	7.5	50	13.5	71.5	80.8	83.4	2300	2290	2285
12	50	15	4	5	65	25.6	64.5	74.6	77	2050	2010	1960
13	50	10	3	7.5	65	16.875	72.2	81.5	86	2380	2300	2260
14	50	15	2	10	35	16	42.65	53.3	57.35	2140	2140	2130
15	50	15	2	10	65	25.6	41	45.3	47	1970	1960	1920
16	50	7.5	2	5	65	23.2	67	72	81.5	2120	2140	2200
17	50	10	3	7.5	50	19.2	73.1	85.3	93.5	2190	2170	2180
18	50	7.5	2	10	65	23.2	61.35	67.5	71.7	2230	2230	2240
19	50	7.5	2	5	65	13.05	85	99.5	104.5	2410	2440	2420
20	50	10	3	7.5	50	13.5	61.6	67.1	73	2220	2230	2190
21	50	15	2	5	35	16	44.8	56	63.6	2090	2100	2160
22	50	10	3	7.5	50	13.5	76	80	86	2230	2200	2200
23	50	7.5	4	5	65	23.2	69	80	82	2260	2240	2190
24	50	15	4	10	65	14.4	78	85	93	2300	2350	2300
25	50	15	4	10	65	25.6	56.2	64.1	70.8	2170	2130	2200
26	50	7.5	4	10	35	7.65	74	79	82.9	2470	2420	2420
27	50	7.5	2	10	65	13.05	94.6	97	105.8	2460	2480	2480
28	50	7.5	2	10	35	7.65	91.3	101.5	109	2530	2480	2500
29	50	15	4	5	35	9	80	87	99	2360	2230	2300
30	50	15	4	10	35	16	66.6	73.5	85	2190	2250	2250
31	50	7.5	2	5	35	7.65	83	94	109	2530	2520	2530
32	50	15	4	10	35	9	76	79.5	87	2500	2500	2500
33	50	15	2	5	65	14.4	75.95	94.8	95.7	2400	2400	2380
34	50	7.5	2	5	35	13.6	54	58.7	68.9	2220	2160	2210
35	50	15	4	5	65	14.4	72	80	88	2350	2360	2380
36	50	7.5	4	10	65	23.2	62.5	75.5	87	2270	2280	2290
37	50	7.5	4	10	35	13.6	67.4	73	79	2350	2390	2390
38	50	7.5	4	5	65	13.05	89	92.2	93.9	2400	2400	2390
39	50	15	4	5	35	16	61.1	72	86	2150	2160	2160
40	50	10	4	7.5	50	13.5	73	83	86	2300	2300	2290
41	50	15	3	7.5	50	14.625	73.5	85	95	2320	2300	2330
42	50	10	3	7.5	35	10.125	70	79	83	2230	2230	2230
43	50	10	3	5	50	13.5	73.7	84	95.2	2290	2270	2310
44	50	10	3	7.5	50	13.5	70	76	82	2120	2170	2100
45	50	7.5	4	10	65	13.05	75.8	86	91.3	2370	2370	2350

Density and compressive strength range were between 1970 to 2560 kg/m^3 and 41 to 94.6 MPa at 7 days, 1960 to 2570 kg/m^3 and 45.3 to101.5 MPa at 14 days, and 1920 to 2590 and 47 to 110 MPa at 28 days, respectively. Figure 4.32 shows density versus compressive strength at 7 days. The figures 4.32 shows the significance of density on compressive strength at 7 days, by increasing the density the compressive strength of ultra high performance increases gradually. It can be said that one of the most important factor on compressive strength of concrete is w/c ratio. By increasing the rate of w/c ratio the density decreases and also the compressive strength also decreased, it can be concluded that by increasing the density, the compressive strength will increase. Also, by increasing the density the voids and spaces between concrete will decrease then this decreasing lead to increase the compressive strength. Also adding fiber with high density leads to increase the compressive strength. Many types of function like polynomial, logarithmic, logit, exponential, power, and linear were tested to find the best R^2 at 7 days. The best curve with highest R^2 of 0.6837 were obtained by using linear function with the equation of Y= 0.0731x- 96.454 was given in Figure 4.32.

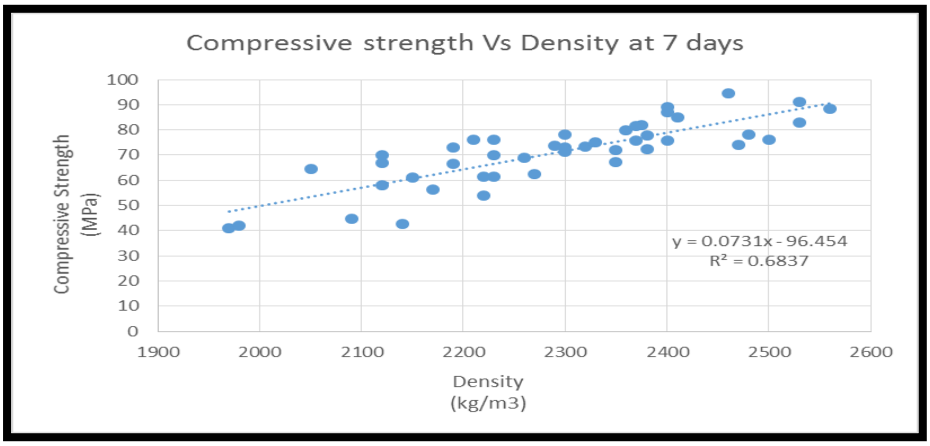

Figure 4.32: Compressive strength versus density at 7 days

Figure 4.33 shows the relation between density and compressive strength at 14 days. As it is shown in Figure 4.33 increasing the density significantly increases the compressive strength in the different mix designs which shows the direct effect of density on 14 days compressive strength. The best function to find the relation between density and 14 days compressive strength was linear function with the formula of Y=0.0758x- 93.868 and R^2 of 0.6766.

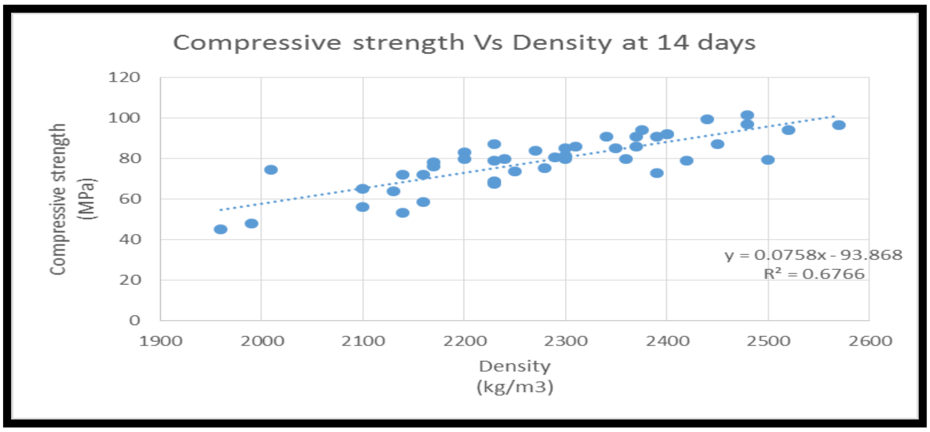

Figure 4.33: Compressive strength versus density at 14 day

Density versus compressive strength of ultra high performance concrete at 28 days is given in Figure 4.34. the compressive strength is increasing as density increases. Increasing the density is due to decreasing the pores and it can be happened by using filler, decreasing the w/c ratio. Many regressive function were used to find the best curve to give prediction model. But the linear function was the best model with the highest R^2 of 0.6609 and the Formula of Y=0.0791x- 94.295. This model is valid for the mixes made with 1.0 sand, 0.15-0.30 silica fume amount, 0.70-1.30 cement amount, 0.10- 0.20 steel fiber, 0.04- 0.08 superplasticizer (all values are by sand by weight mass) and 0.18- 0.32 water cementitious ratio with the normal curing condition.

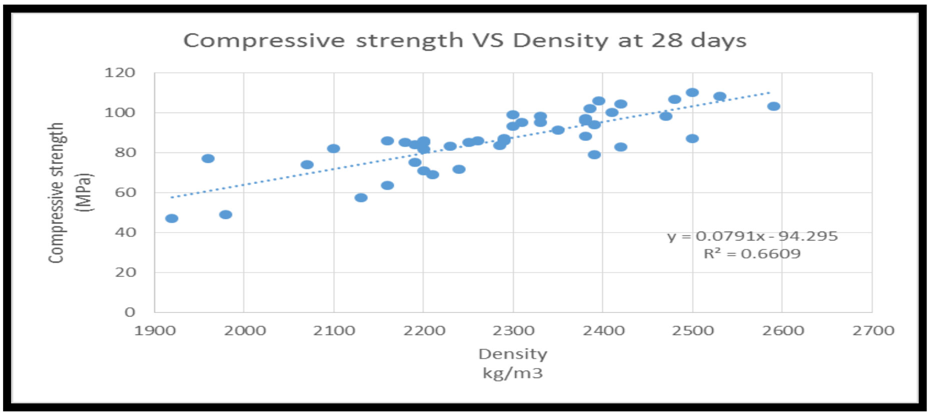

Figure 4.34: Compressive strength versus density at 28 day

Chapter 5

EFFECT OF QUARTZ POWDER, QUARTZ SAND AND CURING ON MECHANICAL PROPERTIES OF UHPC USING RESPONSE SURFACE MODELLING (PHASE TWO)

5.1 Introduction

Ultra High Performance concrete (UHPC) is a superior composite with the special properties in ductility, compressive and tensile strength (Wang, 2014). UHPC is a matrix of main ingredient materials like fine aggregate, fiber, superplasticizer, and large dosage of cement and silica fume (Reddy, 2014). However, by adding some other materials like quartz powder (Qp) and Quartz sand (Qs) and using different methodologies like applying higher curing temperature can improve the properties of UHPC can be improved (Wang, 2014).

At the beginning, UHPC with name of reactive powder concrete (RPC) or ultra-high performance ductile concrete (UHPdC) in 1990s was developed by Richard and Cheyrezy, that introducing of UHPdC considered as one of the amazing developments in the field of concrete technology (Richard & Cheyrezy, 1995). Later on, many researches on the UHPC were done to improve the performance (Aldahdooh et al, 2013). Prem et al. (2015) worked on strength of UHPC with and without fiber in different water curing temperature and reported that the optimum thermal curing of UHPC is 48 hours after demolding. Reda et al. (1999) reported that thermal curing converts weak calcium hydroxide (CH) to strong calcium silicate hydrate (C-S-H) gel during hydration.

Ambily et al. (2013) was developed the particle packing to increase compressive strength of UHPC. Yazici (2007) worked on the effect of combining silica fume, pulverized granulated blast furnace slag with Portland cement, quartz powder and basalt aggregate with three different curing regimes. Reddy et al. (2014) studied the macro mechanical properties of UHPC by using quartz powder as an aggregate and silica fume with different curing regimes.

Response surface methodology (RSM) is a mixture between mathematics and statistics techniques, and could be used for analyzing and modeling different factors to reach a good interpretation by finding the relations between variables to achieve the optimum response (Kumar et al. 2012). De Larrard & Sedran (1994) by using particle packing model found the mix proportion or Yu et al. (2014) modified Anderasen and Anderson particle packing model.

UHPC is normally consuming cement more than three times of normal strength concrete by large amount of ordinary cement between 900-1000 kg/m^3which is concluding using more energy and producing more carbon dioxide (Wille, 2015). This research is trying to model and analyze the effect of quartz powder as cement substitution in side of considering the effect of quartz sand as aggregate substitution and thermal water curing separately and together on mechanical properties of UHPC using response surface methodology.

5.2 Experimental Design

Design of experiment was done by using RSM. In this study the mechanical properties of UHPC was analyzed and the relation between variables were considered.

5.2.1 Methodology

In this research, based on RSM, effect of Qp, Qs and Curing temperature on mechanical properties of UHPC and the interaction of variables were monitored. The response surface modeling used was central composition design with α=1 (face centered) and linear or quadratic models for responses. The interaction between variables and the effect on responses were analyzed by ANOVA. The statistical software "Design- Expert version 9.0.3", Stat-Ease, Inc., was used to analyze the experimental design. Design of experiment deisgn is given in Table 5.1.

Table 5.1: Design of Experiments

Mix no	Qp (A)	Qs (B)	Curing (C)	Qp % (A)	Qs % (B)	Curing ^{0}C (C)
1	0	0	1	10	25	85
2	0	0	0	10	25	55
3	0	0	0	10	25	55
4	-1	-1	1	0	0	85
5	1	1	-1	20	50	25
6	0	0	-1	10	25	25
7	-1	1	-1	0	50	25
8	-1	0	0	0	25	55
9	-1	1	1	0	50	85
10	1	0	0	20	25	55
11	1	1	1	20	50	85
12	-1	-1	-1	0	0	25
13	1	-1	1	20	0	85
14	1	-1	-1	20	0	25
15	0	1	0	10	50	55
16	0	-1	0	10	0	55

In this research, mechanical properties of UHPC was investigated as: the 7-days compressive strength, 14-day compressive strength, 28-day compressive strength as well as splitting tensile were denoted as responses and 3 variables as quartz powder (A), quartz sand (B), different water curing temperatures, (C) are defined to explain the modeling. Based on previous studies and literature review, the range of variables are as follow: quartz powder is from 0 to 20 percent of cement mass as cement substitution, the Quartz Sand content from 0 to 50 percent of sand mass as sand substitution, and three water curing temperature is from 25 to 85 ^{0}C. The variables with their level limitation are given in Table 5.2.

Table 5.2: The variables with their levels

Variables	Code	Low level -1	Intermediate level 0	High level +1
Quartz powder	A[a]	0%	10%	20%
Quartz sand	B[b]	0%	25%	50%
Water curing	C[c]	25^{oc}	55^{oc}	85^{oc}

[a] percentage of aggregate substitution by aggregate mass, [b] percentage of cement substitution by cement mass, [c] different water curing temperature

5.2.2 Mix Proportion

Details of mix proportions are given in Table 5.3.

Table 5.3: UHPC mix proportion

Mix	Aggregate (kg/m^3)	Silica Fume (kg/m^3)	Cement (kg/m^3)	Steel Fiber (kg/m^3)	Super plasticizer (kg/m^3)	Water (kg/m^3)	W/C Ratio
Amount	1244	187	870	250	50	190	0.18

5.2.3 Specimen Preparation and Test Specimen

In this part of the thesis, 16 batches were prepared (Table 5.4) which were mixed in a drum rotating mixer. The first premix which included dry materials (cement, SF, sand, and if there was quartz) except steel fibers were blended in a determined proportion for 5 minutes, then proportional amount of superplasticizer was added to suitable water as well as steel fiber, thereafter, water was added to the premixed mixture and mixed to obtain homogeneous fresh concrete. Ten 100 mm cubes were casted for compressive strength determination for testing at three different days (7, 14, and 28-day). Also, three 100*200 mm (D*L) cylinders were casted for 28-day splitting tensile strength. After casting, all samples were compacted by vibration table and kept in the moist curing room for 24 hours. They were then molded out and transferred to the appropriate curing water tank temperature in different levels at 25, 55, 85 ºC for 48 hours, then were kept on water curing tank at 25 ± 2 °C until testing.

Table 5.4: Mix proportions of UHPC

NO	Crushed limestone	Silica Fume	Cement	Steel Fiber	Super plasticizer	Water	W/C	Qp	Qs	water curing temperatu
#	kg/m^3	kg/m^3	kg/m^3	kg/m^3	kg/m^3	kg/m^3	--	kg/m^3	kg/m^3	^0C
1	933	187	783	250	50	190	0.18	87	311	85
2	933	187	783	250	50	190	0.18	87	311	55
3	933	187	783	250	50	190	0.18	87	311	55
4	1244	187	870	250	50	190	0.18	0	0	85
5	622	187	696	250	50	190	0.18	174	622	25
6	933	187	783	250	50	190	0.18	87	311	25
7	622	187	870	250	50	190	0.18	0	622	25
8	933	187	870	250	50	190	0.18	0	311	55
9	622	187	870	250	50	190	0.18	0	622	85
10	933	187	696	250	50	190	0.18	174	311	55
11	622	187	696	250	50	190	0.18	174	622	85
12	1244	187	870	250	50	190	0.18	0	0	25
13	1244	187	696	250	50	190	0.18	174	0	85
14	1244	187	696	250	50	190	0.18	174	0	25
15	622	187	783	250	50	190	0.18	87	622	55
16	1244	187	783	250	50	190	0.18	87	0	55

5.2.4 Compressive Strength Test

To determine compressive strength of specimens, 100 mm UHPC cubes were tested. Concrete compression testing machine conformed ASTM C109 (2002) with a 3000 kN in capacity was used. Three samples for each day were tested.

5.2.5 Splitting Tensile Strength

Splitting tensile was performed through the ASTM C496 (2004) Standard test method for splitting tensile strength of cylindrical concrete specimens. The specimen was 100*200 mm (d*l) cylinder tested at 28-days. Standard concrete compression machine was used to do this experiment.

5.3 Results and Discussion of Results

The effects of three variables (quartz powder (Qp), quartz sand (Qs), thermal water curing) on the mechanical properties (compressive and tensile strength) of UHPC was analyzed by using response surface method. For producing the model, 16 points were selected such as 8 points for model making, 2 points for replication, and 6 points to consider the lack of fit.

Table 5.5 shows the results of using three different variables in mechanical properties of UHPC, compressive strength at 7, 14, 28-day, splitting tensile. Each result was derived by average of 3 specimens.

Table 5.5: Responses result of UHPC mixtures

Qp	Qs	Curing Regimes	Compressive Strength (MPa)			Splitting tensile strength (MPa)
			7-day	14-day	28-day	
-1	-1	-1	91.0	101.0	111.0	17.0
		1	116.8	121.0	122.0	15.2
	0	0	115.0	119.0	118.0	15.0
	1	-1	82.7	99.0	107.0	11.9
		1	120.0	123.0	124.0	14.1
0	-1	0	118.0	120.0	122.9	16.0
	0	-1	91.0	105.7	116.5	16.6
		0	120.8	121.5	123.4	14.7
			120.0	122.0	124.0	14.0
		1	122.0	122.0	124.0	17.9
	1	0	120.0	118.5	125.2	11.5
1	-1	-1	90.0	105.0	114.0	14.2
		1	125	124.4	124.0	15.8
	0	0	120.6	119.7	123.9	15.5
	1	-1	90.0	103.4	120.3	16.0
		1	132.7	129.0	131.5	18.0

The correlation and interaction between variables were calculated by ANOVA (analysis of variance). For the modeling, three types of modelling were checked. Linear model, two-factor interaction, and quadratic models. In each model, the significant parameters were detected, therefore, by backward elimination technique the insignificant terms were eliminated and the final regressions for each were done. As a result, the quadratic model was selected for all responses. The quality of prediction models were determined by coefficient of multiple determination R^2, which shows the total deviation of the variables from the prediction model. The p-value (probability of errors) with 95% confidence level and statistical significant test at 5% and also lack of fit with p-value greater than 0.05 was performed and checked for model validations.

Table 5.6 shows the analysis result of regression models. Compressive models were significant according to t-test ($P < 0.05$) and F-value and lack of fit with given P-value implies which are insignificant. Furthermore, the model coefficient of determination R^2 has a reliable confidence with 0.87, 0.88, 0.88, 0.88, and 0.83 for different responses. The predicted R^2 of 0.996, 0.975, and 0.984, are in reasonable agreement with adjustment in R^2 of 0.992, 0.975, and 0.965 for compressive strength models, whereas, the differences are less than 0.2.

Table 5.6: Analysis result of regression models

Response	R^2	Adj-R^2	Pre-R^2	F-Value	Lack of fit	Model P-value
Compressive strength 7-day	0.996	0.992	0.972	237	0.28	<0.0001
Compressive strength 14-day	0.975	0.960	0.935	76.71	0.13	<0.0001
Compressive strength 28-day	0.984	0.965	0.870	53.00	0.26	<0.0001
Splitting tensile strength	0.830	-	-	-	insignificant	0.094>0.05

Analysis of variance showed that the three used variable (quartz powder, quartz sand, thermal water curing) did not have meaningful significant effect on 28 days splitting tensile, whereas, the P value was bigger than 0.05.

The performance of offered prediction models with mechanical responses (7, 14, 28 days compressive strength) for mixture experimental design of UHPC are illustrated in Figure 5.1- Figure 5.3.

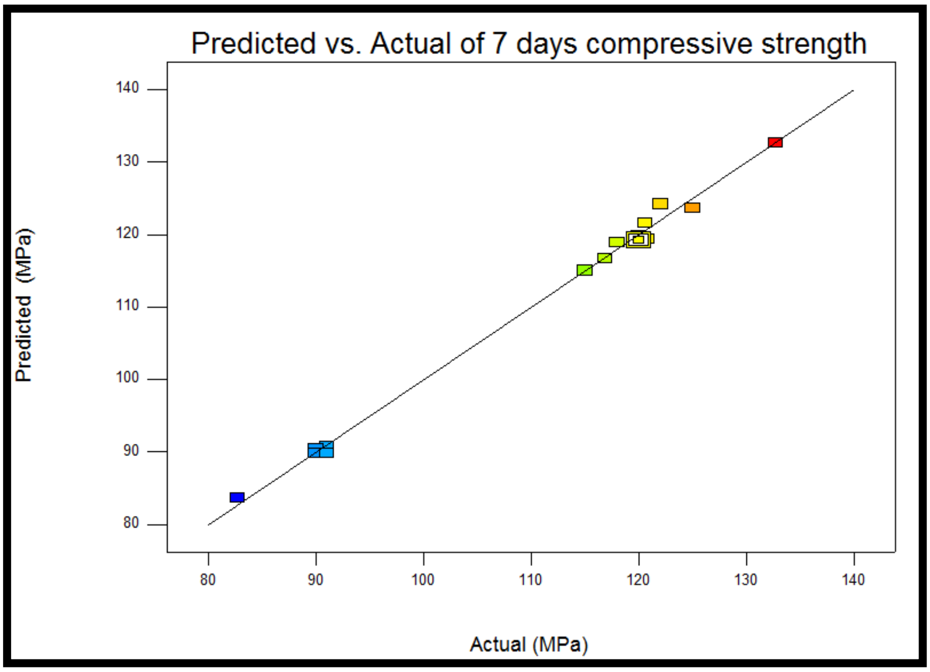

Figure 5.1: Prediction of efficiency of offered model for 7-day compressive strength

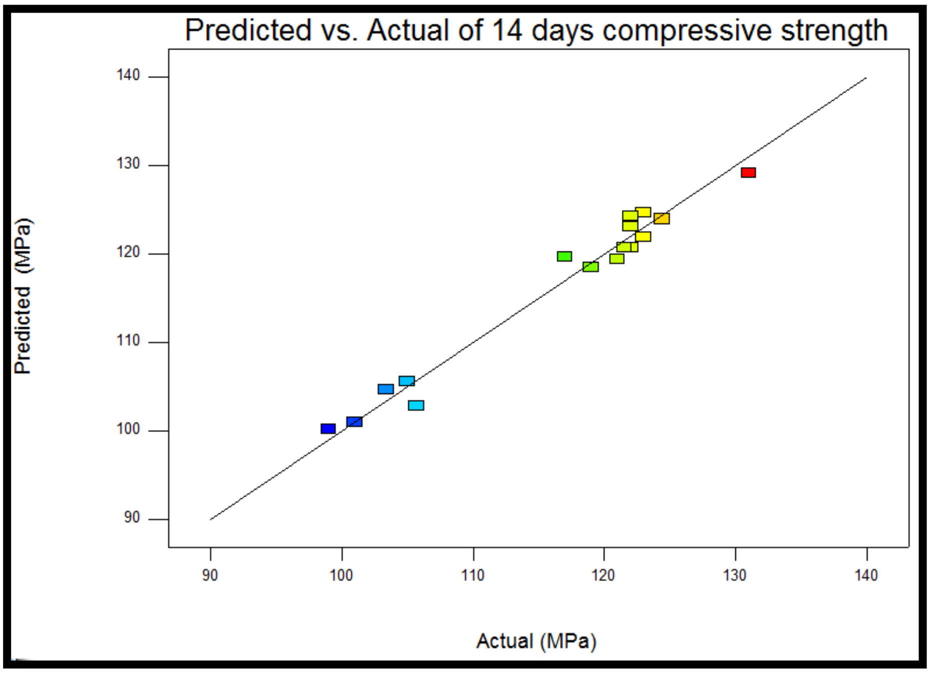

Figure 5.2: Prediction efficiency of offered model for 14-day compressive strength

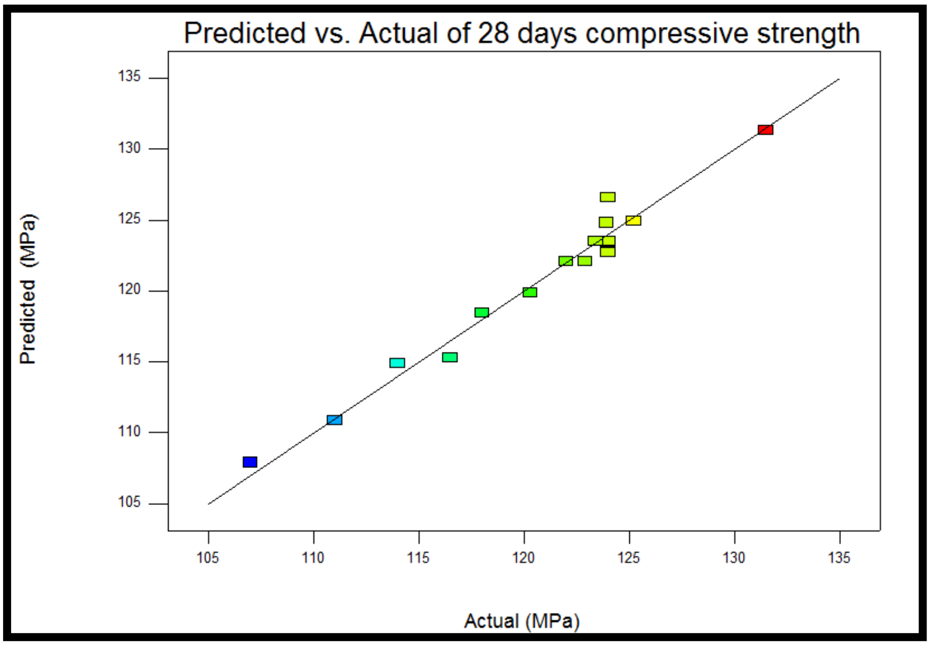

Figure 5.3: Prediction efficiency of offered model for 28-day compressive strength

Table 5.7 lists the finalized prediction models to reach the desired performance of compressive and tensile strength of UHPC. Probability factor is given for each parameter, in Table 5.7, linear variable A and C are statistically significant factors for all ages compressive strength as shown in Table 5.7. The quadratic B is not a statistically significant factors at the stipulated level of 5%, however, the quadratic C is statistically significant factors at the stipulated level of 5% for all ages compressive strength. The significant of some two-way interaction terms are given in 7, 14, 28 days compressive strength in Table 5.7. A significant two-way interactions explains that the simple effect of a variable is not same at all levels of other variables. The 2-ways interaction of A with B, C (AB, AC), and B with C (BC) are statistically significant factors at the stipulated level of 10% for 7 days compressive strength. In 14 days compressive strength, 2-way interactions of B with C (CD) is statistically significant factors at the stipulated level of 10%. Also in 28 days compressive strength, the 2-way interactions of A with B (AB) is statistically significant factor at the stipulated level of 10%.

Table 5.7: Parameter estimated for models at 7, 14, 28 days compressive strength

	Compressive strength (7 days)		Compressive strength (14 days)		Compressive strength (28 days)	
Parameters	**Estimate**	**Prob > f**	**Estimate**	**Prob > f**	**Estimate**	**Prob > f**
Constant	119.44	---	120.75	---	123.53	---
A	3.28	0.000157	2.28	0.003871	3.17	0.000258
B	0.46	0.341733	1.10	0.101637	1.41	0.019536
C	17.19	2.07E-09	10.73	0.000000	5.67	0.000006
AB	1.61	0.014647	---	---	1.98	0.006922
AC	1.82	0.008218	---	---	-0.85	0.148097
BC	2.39	0.001968	1.53	0.049413	0.90	0.128920
A^2	-1.12	0.216351	---	---	-1.89	0.064973
C^2	-12.42	1.36E-06	-7.20	0.000028	-2.59	0.020013

Effect of three parameters (quartz powder, quartz sand, different water curing temperaturess) on mechanical properties (7, 14, 28 days compressive strength, and 28 days splitting tensile strength) have been considered employing response surface methodology. Effect of variables on responses can be presented graphically by plotting of response value versus variables in different dimensions. The study shows the effect of each variable singularly and with other variables.

The quartz powder pozzolanic reactive is very low and slow. To enhance its reactivity, high heat or high pH is needed. That is why the correlation between different water curing temperatures was a bit significant in compressive strength modeling of UHPC. On the other side, the quartz powder can be used as filler (Sahani & Ray, 2014). Thereby, by reducing the initial porosity of the mixture the final strength will be increased(Sahani & Ray, 2014). The Different water curing temperatures was very effective on compressive strength of UHPC. the effect of Different water curing temperatures regimes was more highlighted in 7 days compressive strength in compression by 14, 28 days compressive strength. Raising the temperature increases the rate of hydration, so, the thermal water curing influences more on the early ages. Thermal curing regime enhance shaping of hydrated structures (Yu et al., 2014, Wang et al., 2015).

5.3.1 Effect of Variables on 7 Days Compressive Strength

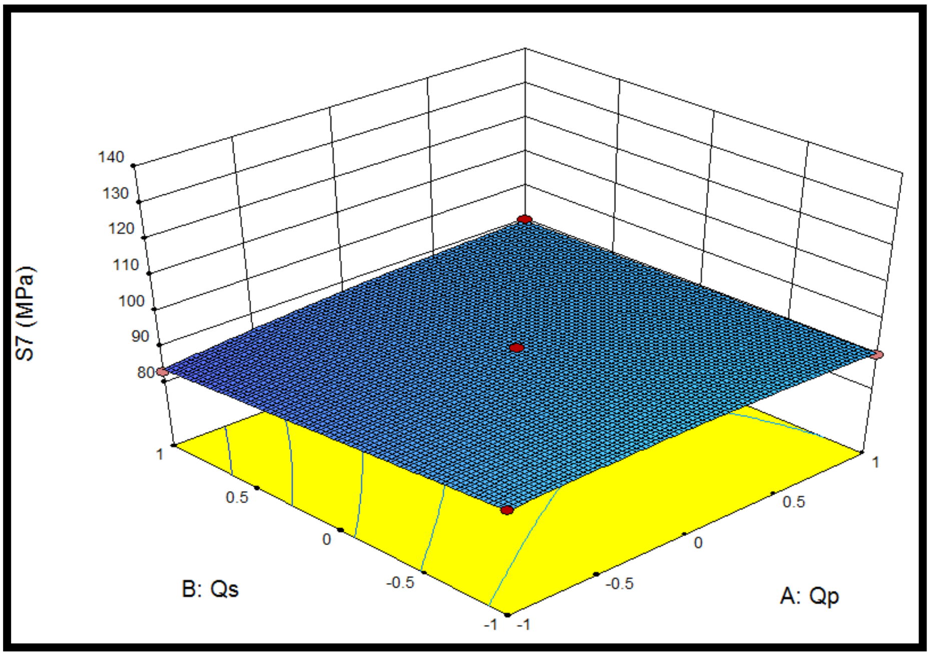

Figure 5.4: Response surface plot of X1=A:Qp amount, X2=B:Qs, and water Curing level of -1 on 7-day compressive strength

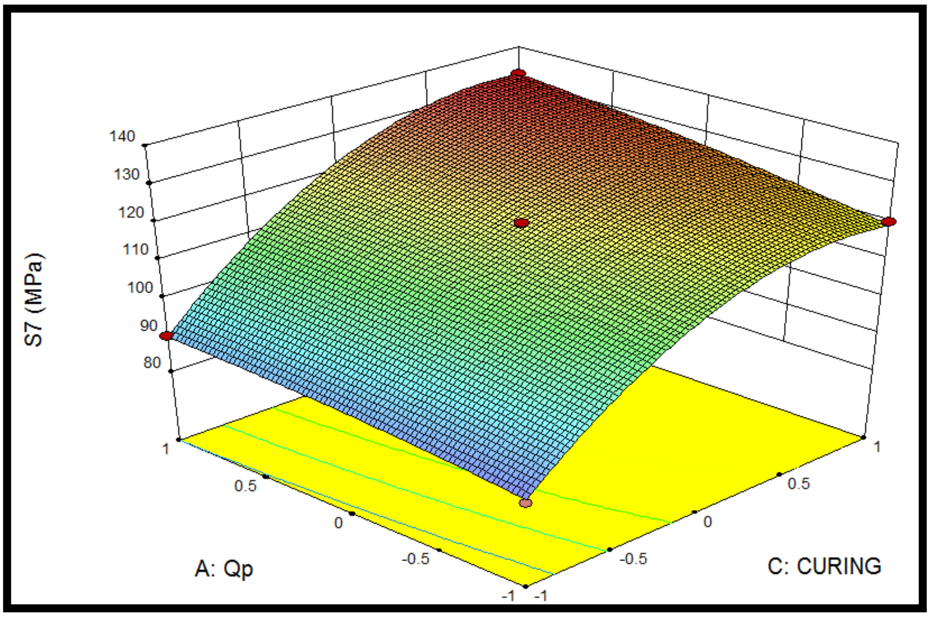

Figure 5.5: Response surface plot of X1=C:Curing level, X2=A;Qp, and Qs level of 1 on 7-day compressive strength

Figure 5.4 shows the 3D plot of Qp and Qs when curing is in lowest level (-1). The minimum value of 7-day compressive strength could be seen when all variables values are in mininmum level (-1). But, Figure 5.5 shows the 3D plot of Qp and thermal water curing with maximum level Qs. Highest value of 7 days compressive strenght above 45% increase could be seen when all variables are in maximum level (1). It is concluded that these three variables play important roles to increase the 7 days compressive strength.

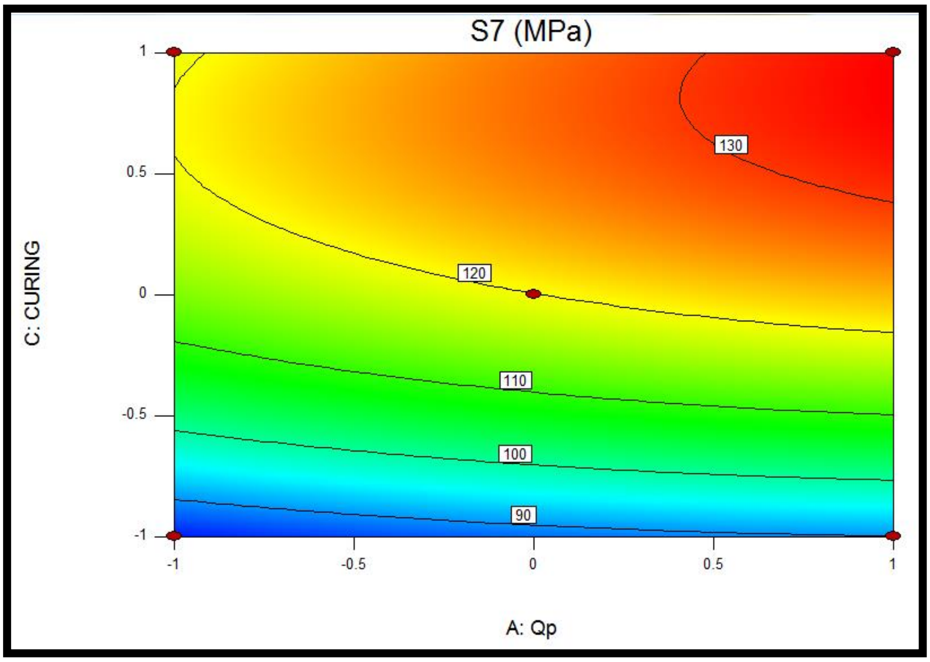

Figure 5.6: Contour plot of 7 days compressive sttength changes, X1=A:Qp amount and X2=C:Curing level

The effect of two way interaction (themal water curing regimes with quartz powder and different water curing temperatures with quartz sand) on 7 day compressive strength is given in Figure 5.6. The result significantly shows the effect of curing temperature on 7-day compressive strength, and also the possivite effect between quartz sand and quartz powder on 7 day compressive strength is shown in Figure 5.7 on 7-day compressive strength of UHPC. Increasing Qp and Qs increase 7-day compressive strength of UHPC.

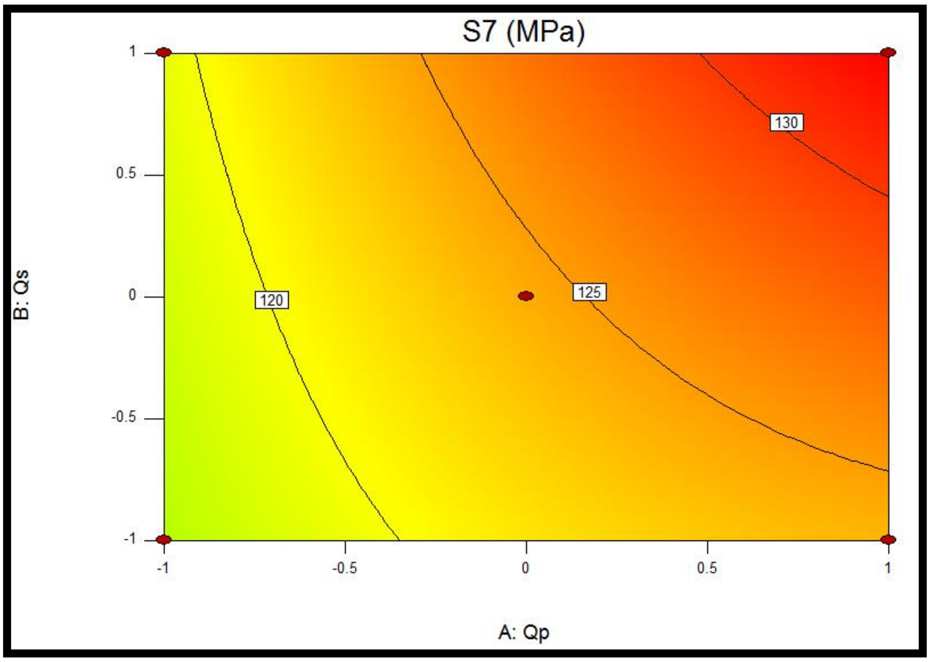

Figure 5.7: Contour plot of 7 days compressive sttength changes, X1=A:Qp amount and X2=B:Qs

5.3.2 Effect of Variables on 14 Days Compressive Strength

The effect of two variables at Qp=1 on 14 days compressive strength is monitored as 3D plot in Figure 5.8. The figure clearly shows the positive effect of variables on the 14 days compressive strength. The maximum value of 14-day compressive when they are in the highest level. 28% increasing on 14 days compressive strength of UHPC was found upon increasing the variable from the lower level to the maximum level.

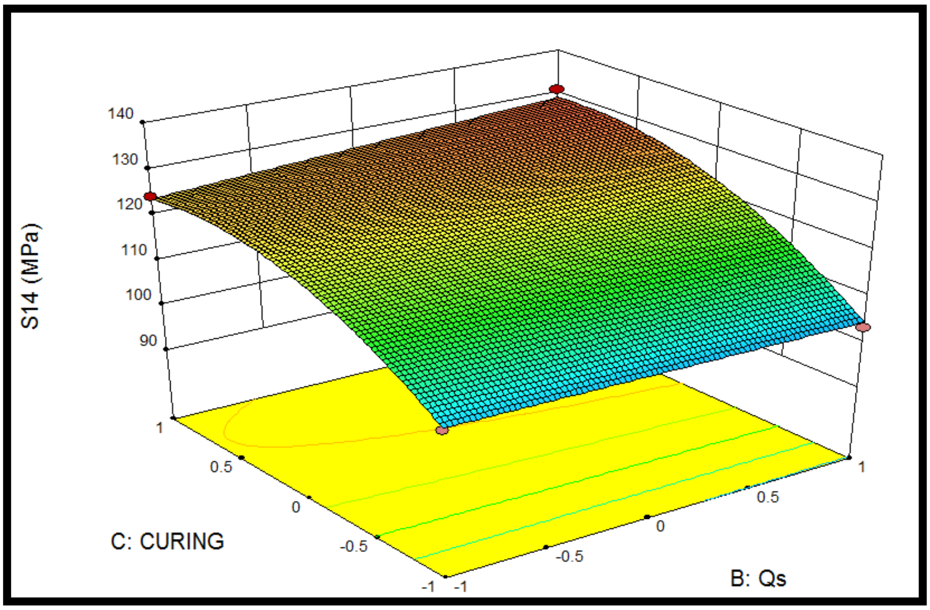

Figure 5.8: Response surface plot of X1 =B:Qs amount, X2:C= Curing level, and Qp level of 1 on 14 day compressive strength

The single effect of Qp on 14-day compressive strength is given in Figure 5.9. Substituting cement by quartz powder has effect on 14 days compressive strength. It can be explained because of low hydration activity and acting as filler inside of mixture.

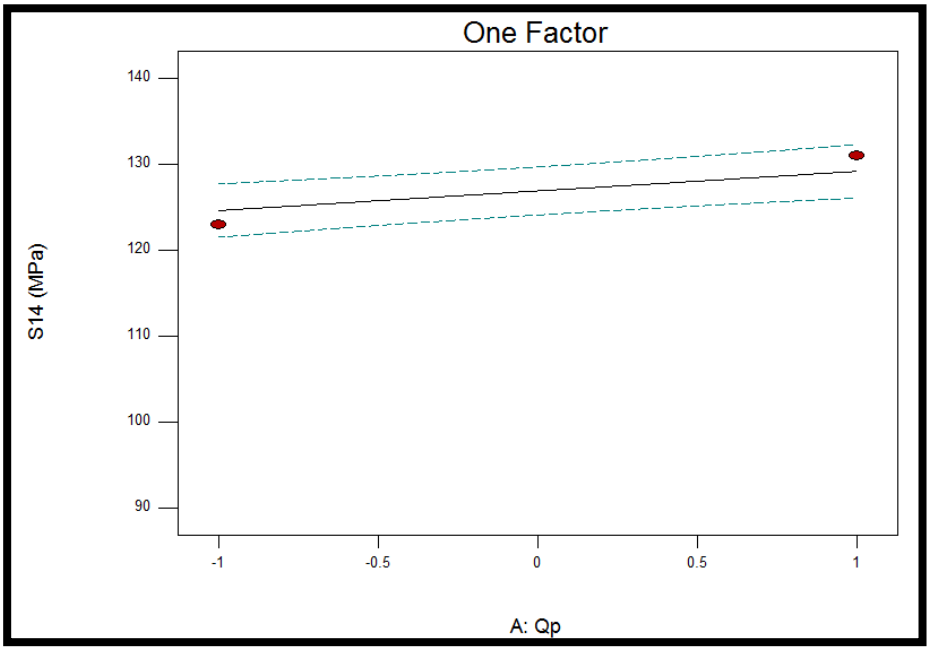

Figure 5.9: Effect of A: Qp on 14-day compressive strength

Replacing quartz powder by crushed limestone sand has positive effect on 14 days compressive strength which is shown in Figure 5.9.

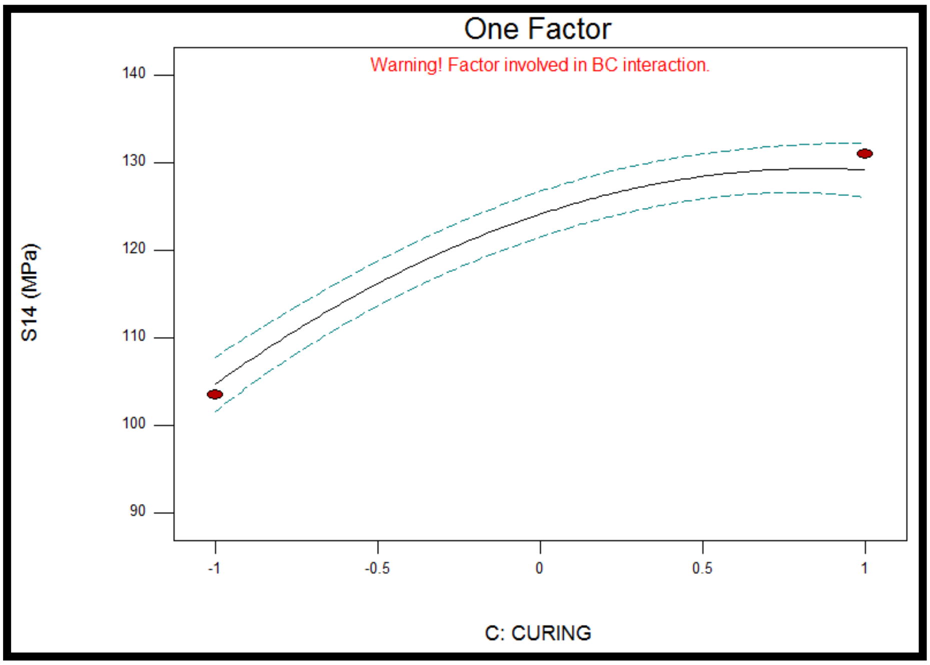

Figure 5.10: Effect of Water curing A:Qp =1 and B:Qs=1 on 14-day compressive strength

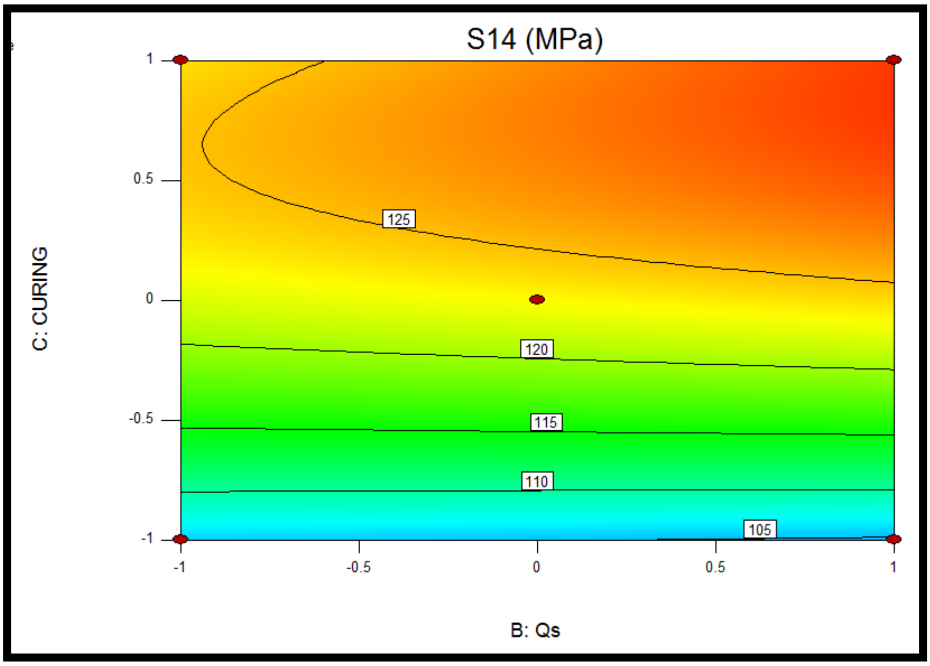

Figure 5.11: Contour plot of 14-day compressive strength changes, X1=B:Qs and X2=C: Curing level

The effect of single curing on 14-day compressive strength is illustrated in Figure 5.10. As it is clear that thermal water curing significantly affects 14-day compressive strength.

Effect of two interaction-way of Qs and curing is shown in Figure 5.11. Increasing the curing temperature and substituting the quartz sand with crushed limestone aggregate raise the 14-day compressive strength of UHPC.

5.3.3 Effect of Variables on 28 Days Compressive Strength

The effect of variables on 28 days compressive strength are shown in Figure 5.12-Figure 5.15. The Figure 5.12 shows the positive effect of variables on 28 days compressive strength of UHPC as highest level of variables together gives the maximum 28 days compressive strength.

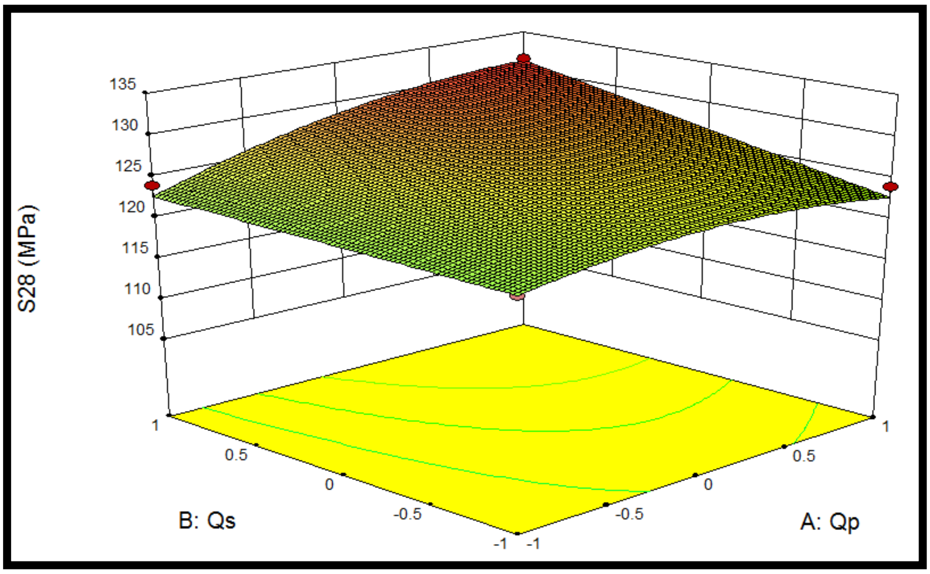

Figure 5.12: Response surface plot of X1 =A:Qp amount, X2 =B:Qs, and water Curing level of 1 on 28 day compressive strength

The effect of single Quartz powder on 28 compressive strength is given in Figure 5.13. It shows that by increasing quartz powder as cement substitution, the 28 days compressive strength rate is increasing.

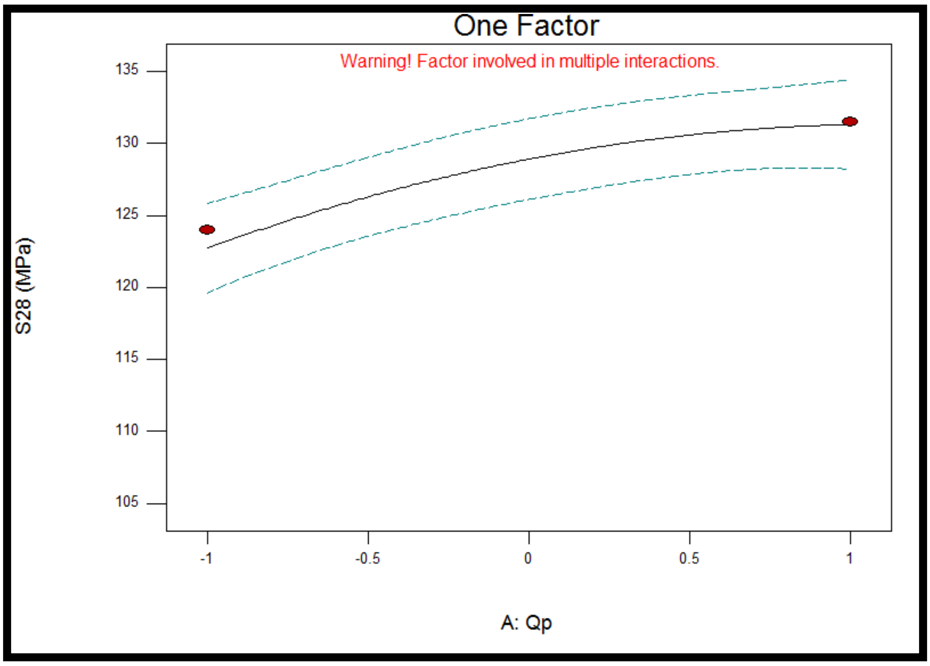

Figure 5.13: Effect of A:Qp with Qs =1 and water curing=1 on 28 day compressive strength

Two interaction of themal water curing regimes and quartz powder is shown in Figure 5.14 which is concluded that by substituting the cement with quartz power and increasnig the water curing tempreature the compressive strength is mainly increased.

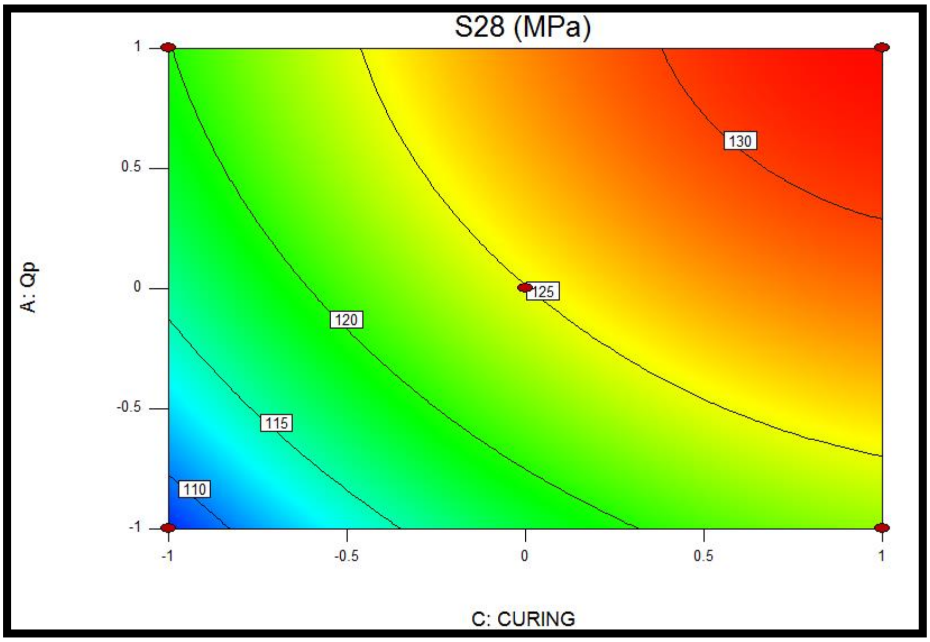

Figure 5.14: Contour plot of 28 days compressive sttength changes, X1=B: curing level and X2=A: Qp

The effect of single factor of curing on 28 days compressive strength is shown in Figure 5.15. It is obviously clear that by increasing the curing temperature, the 28 days compressive strength is increased.

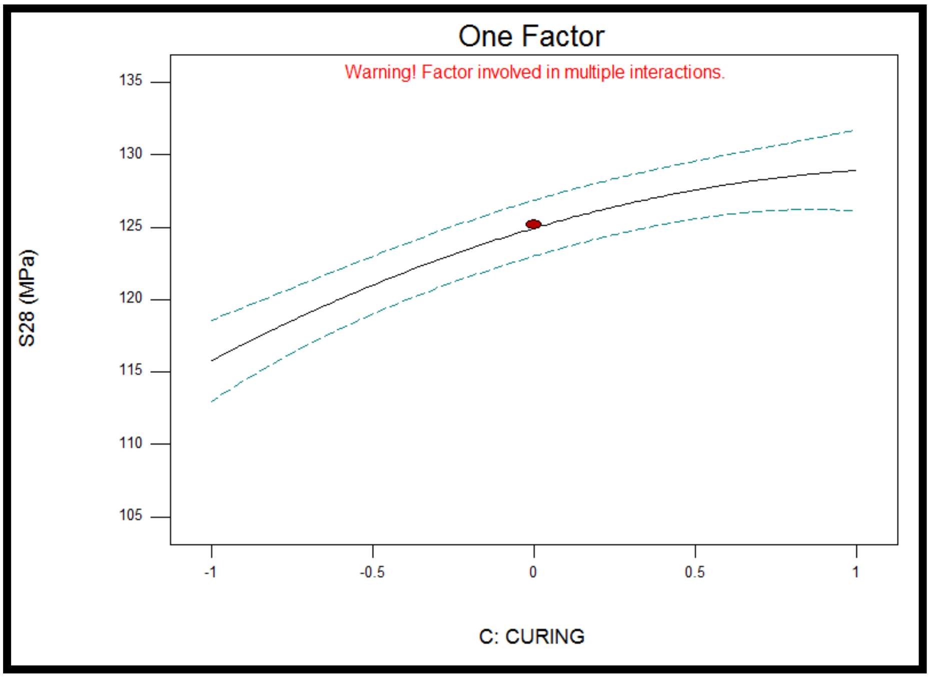

Figure 5.15: Effect of C: curing with Qp =0 and Qs=1 on 28 day compressive strength

Chapter 6

CONCLUSION AND RECOMMENDATIONS

6.1 Conclusion of part 1: Statistical Models for Mechanical Properties of UHPC with Local Materials Using Response Surface Methodology

The effects of five independent variables (including: silica fume, steel fibers, cement, superplasticizer, and w/c ratio on the mechanical properties of UHPC with local materials were investigated by using central composition response surface methodology and quadratic models for responses were performed. In this experimental study, interaction and correlation of the five variables were analyzed. The validity and significance of models and factors were analyzed by ANOVA. A total of 45 batches were produced to provide models 7, 14, and 28-day compressive strength, splitting tensile strength, 28-day flexural toughness and 28-day modulus of rupture. The most important findings of the study are given below:

- Quadratic model with R^2 of above 0.83 was obtained for 7, 14, and 28 days compressive strength, splitting tensile strength and modulus of rupture.
- Increasing the amount of cement paste did not necessarily increase the mechanical properties of UHPC.
- The increase in silica fume content (15% to 43% in the cement mass) had negative effect on compressive and tensile strength properties of UHPC.
- From the ANOVA statistical modeling, it was found that the interaction of w/c ratio with superplasticizer content was significantly important for improving

the mechanical properties such as compressive strength and tensile strength of concrete.

- The effect of steel fibers on tensile strength was highlighted but it was negligible for compressive property.
- It was proved that the w/c ratio is the most important factor for improving the mechanical properties of concrete according to adjusted variable values.
- Quadratic model with R^2 of above 0.85 was obtained for flexural toughness.
- Increasing amount of cement paste did not necessarily increase the energy absorption of UHPC.
- The increase in silica fume content (15% to 43% of cement mass) had negative effect on flexural toughness.
- The effect of w/c ratio (0.18 to 0.32) on flexural toughness was the most highlighted one.
- Increasing the fiber (10% to 20% of aggregate mass) content increased the energy absorption.

Also, relation of density and 7, 14, 28 days compressive strength with considering the effect of five independent variables (amount of silica fume, amount of steel fibers, amount of cement, amount of superplasticizer, and w/c ratio) was investigated. In this experimental study, a total of 45 batches were prepared to produce three valid models (7, 14, 28 day compressive strength). The most important findings of the study are as given as: Increasing the density increased the compressive strength of ultra high performance concrete in regarding of its mix proportions. The linear model was the best function type to predict compressive strength by having density. Compressive strength of ultra high performance concrete can be predicted by using nondestructively using non-destructive methodology for the given range of mix design. Linear predication models of 7, 14, 28-day compressive strength were found to have R^2 of 0.6837, 0.6766, and 0.6609, respectively.

6.2 Conclusion of part 2: Effect of Quartz Powder, Quartz Sand and Curing on Mechanical Properties of Ultra High Performance Concrete Using Response Surface Modelling

The effect of three controllable variables (quartz powder, quartz sand, different water curing temperatures) on mechanical properties of UHPC with local materials were investigated by using central composition response surface methodology and quadratic models for responses were performed. Besides, interaction and correlation of three variables were performed. The significance of model and factors were analyzed by ANOVA. A total of 16 batches were prepared to produce three valid models (7, 14, 28-day compressive strength, splitting tensile strength). The most important findings of the study are as given below:

Quadratic models with R^2 of above 0.975 were obtained for 7, 14, 28-day compressive strength. The result showed the variables did not have a main effect on 28-day splitting tensile strength despite, having R^2 of 0.83 which is shown the accuracy of using ANOVA.

Increasing 7, 14, and 28-day compressive strength treatment of UHPC were occurred by replacing the quartz powder with cement which causes to decrease the cement consuming up to 20% and produce a more environmental friendly.

Partial substitution of crushed limestone sand by quartz sand modified the compressive strength at all. Change of thermal water curing temperature significantly influences 7, 14, and 28-day compressive strength of UHPC.

6.3 Recommendations for future studies

1. This study was performed for mechanical properties of UHPC, therefore, the investigation on the micro-structure of UHPC is also very important.
2. It is important to study the durability of UHPC in different weather conditions.
3. The cement used in this study was type 2 Portland sulfate resisting slag class 42.5. Effect of other cement types on the performance of UHPC is needed.
4. The effect of different curing types like steam and curing inside of water curing on mechanical properties of UHPC is very important.
5. Using Nano-technology and Nano-materials in order to increase the performance of UHPC is strictly recommended.

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