# INTEGRAL MIXING USING NANO SILICON FOR CONCRETE WATERPROOFING

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Dedicated to

My family and friends

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## ABSTRACT

Permeation of water and other aggressive fluids in concrete can result in degradation and other aesthetic problems. Consequently, these affect the service life of concrete structures. A number of research studies were undertaken to extend the service life of concrete infrastructures using various waterproofing agents. To this effect, a great deal of repair and maintenance cost can be avoided. The aim of this study is to investigate and establish waterproofing performance of nano silicon-based mortar. In this regard, nano silicon was characterized using Field Emission Scanning Electron Microscope (FESEM), Energy Dispersion Spectroscopy (EDS), Fourier Transformed Infrared (FTIR), X-Ray Diffraction (XRD), surface zeta potential and Water Contact Angle Test (WCA). Response Surface Methodology (RSM) was employed to establish the optimum mix ratio. The relationship between the experimental factors and response was modelled and validity of the model was further evaluated to ensure accurate predictions. To establish precision of the mathematical model, an experiment was planned based on Central Composite Design (CCD). The model was investigated using Analysis of Variance (ANOVA). Optimum mix ratio, necessary to increase resistance to water absorption was established at nano silicon dosage of 6.6% by weight of cement and w/c of 0.42. Furthermore, an appropriate experimental control test steps for producing waterproof cement mortar was designed. In this regard, necessary test methods from established standards were adopted to constitute supporting structure of the approach. Besides, the results were validated using macro and microstructure tests and indicated that water resistance to capillary absorption of cement mortar increased to 62%. Likewise, water absorption by immersion increased by 37%. Furthermore, resistance to water vapor transmission rate increased to 52%. On the other hand, resistance to gas permeability increased to 31% as compared to reference specimen. Moreover, while the volume of water permeable voids for nano silicon-based mortar was 16.9%, the total porosity of the same specimen was 14%. Macrostructure test indicated a good quality mortar specimen recorded an Ultra Sonic Pulse Velocity (UPV*)* value of 3623 (m/s). In addition, FESEM and XRD indicated the formation of a crystalline hydrophobic thin film layer of nano silicon within the pore structure of the mortar specimen. In conclusion, the nano silicon-based mortar has been proven to have a good resistance to water permeation.

# ABSTRAK

Penelapan air dan cecair agresif yang lain ke dalam konkrit dapat membawa kepada pemerosotan dan masalah estetik lain. Oleh yang demikian, ini memberi kesan kepada hayat perkhidmatan struktur konkrit. Beberapa penyelidikan telah dijalankan untuk melanjutkan hayat perkhidmatan infrastruktur konkrit menggunakan pelbagai agen kalis air. Bagi kesan tersebut, banyak kos pembaikan dan penyelenggaraan dapat dielakkan. Tujuan kajian ini adalah untuk mengkaji dan menantukan prestasi kalis air.mortar yang berasaskan nano silicon. Dalam hal ini, penentuan ciri nano silikon dilakukan dengan menggunakan Mikroskop Elektron Imbasan Pancaran Medan (FESEM), Spektroskopi Tenaga Penyerakan (EDS), Inframerah Transformasi Fourier (FTIR), Pembelauan Sinar-X (XRD), Keupayaan Permukaan Zeta dan Ujian Sudut Sentuhan Air (WCA). Oleh itu, Metodologi Permukaan Gerak Balas (RSM) digunakan untuk mewujudkan nisbah campuran yang optimum. Hubungan antara faktor-faktor eksperimen dengan tindak balas telah dimodelkan dan selanjutnya kesahan model dinilai untuk memastikan ramalan yang lebih tepat. Bagi mewujudkan ketepatan model matematik, eksperimen dirancang berdasarkan Reka Bentuk Komposit Pusat (CCD). Model ini dikaji menggunakan Analisis Varians (ANOVA). Nisbah campuran yang optimum perlu untuk meningkatkan rintangan kepada penyerapan air yang wujud pada dos nano silikon sebanyak 6.6% mengikut berat simen dan nisbah air kepada simen, iaitu 0.42. Selain itu, langkah-langkah ujian kawalan eksperimen yang sesuai bagi menghasilkan mortar simen kalis air telah dibangunkan. Dalam hal ini, kaedah ujian yang diperlukan untuk menghasilkan piawaian yang ditetapkan telah diterima pakai untuk membentuk struktur bagi menyokong pendekatan ini. Di samping itu, hasil kajian disahkan menggunakan ujian makro dan mikrostruktur yang menunjukkan bahawa rintangan air bagi penyerapan kapilari simen mortar telah meningkat kepada 62%. Begitu juga, penyerapan air dengan rendaman telah meningkat sebanyak 37%. Selain itu, rintangan kepada kadar penghantaran wap air telah meningkat kepada 52%. Di samping itu, rintangan terhadap kebolehtelapan gas telah meningkat kepada 31% berbanding dengan spesimen kawalan. Selain itu, jumlah air lompang telap bagi mortar berasaskan nano silikon pula 16.9% manakala jumlah keliangan spesimen yang sama adalah 14%. Ujian makrostruktur menunjukkan spesimen bagi mortar berkualiti baik merekodkan nilai Halaju Denyut Ultrasonik (UPV) sebanyak 3623 (m/s). Tambahan pula, FESEM dan XRD menunjukkan pembentukan lapisan filem nipis hidrofolik berkristal nano silikon dalam struktur liang spesimen mortar. Kesimpulannya, mortar berdasarkan nano silikon terbukti mempunyai ketahanan yang baik terhadap kemasukan air.

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# LIST OF ABBREVIATIONS

| | | |
|---|---|---|
| A | - | Acrylic |
| Adj R | - | Adjusted Regression Coefficient |
| ASTM | - | American Society for Testing and Materials |
| ANOVA | - | Analysis of Variance |
| BS | - | British Standard |
| CCD | - | Central Composite Design |
| CNS | - | Colloidal Nano Silica |
| Comp Str | - | Compressive Strength |
| CV | - | Coefficient of Variation |
| Cor | - | Total Corrected |
| DoE | - | Design of Experiment |
| EDS | - | Energy Dispersive Spectroscopy |
| FTIR | - | Furrier Transformed Infrared |
| FESEM | - | Field Emission Scanning Electron Microscope |
| GCRC | - | German Committee of Reinforced Concrete |
| NCA | - | Natural Aggregate Concrete |
| NS | - | Nano Silicon |
| NMR | - | Nuclear Magnetic Resonance |
| OPC | - | Ordinary Portland Cement |
| PRESS | - | Predicted Residual Sum Of Squares |
| PU | - | Polyurathane |
| predR | - | Predicted Regression Coefficient |
| SEM | - | Scanning Electron Microscope |
| R | - | Regression Coefficient |
| RAC | - | Recycled Aggregate Concrete |
| RH | - | Relative Humidity |
| RSM | - | Response Surface Methodology |

| | | |
|---|---|---|
| RILEM | - | International Union of Laboratories and Experts in Construction Materials, System and Structures |
| TEM | - | Transmission Electron Microscope |
| UPV | - | Ultrasonic Pulse velocity |
| W/C | - | Water Cement ratio |
| WCA | - | Water Contact Angle |
| WVTR | - | Water Vapour Transmission Rate |
| XRD | - | X-Ray Diffraction |

# LIST OF SYMBOLS

| | | |
|---|---|---|
| A | - | Nano Silicon dosage |
| B | - | Magnitude of water cement ratio |
| K | - | Number of variables |
| $\beta 0$ | - | Constant term |
| $\beta i$ | - | Coefficient of linear parameter |
| $\chi i$ | - | Represents the variable factor |
| $\varepsilon$, | - | Residual associated with experiment |
| $\beta ij$ | - | Coefficient of interaction |
| $\beta ii$ | - | Coefficient of quadratic parameters |
| $W_T$ | - | Weight od specimen at time T |
| $W_O$ | - | Initial weight of specimen at time T |
| $I$ | - | Absorption |
| $S$ | - | Capillary coefficient |
| $T$ | - | Time |
| $L_1$ | - | Average length of the test surface |
| $G$ | - | Weight change |
| $m*$ | - | Rate of mass loss |
| $K$ | - | Permeability coefficient |
| $\eta$ | - | Dynamic viscosity |
| $P_1$ | - | Inlet pressure |
| $P_2$ | - | Outlet pressure |
| $Q$ | - | Flow rate |
| | | |
| A | - | Nano Silicon dosage |

# LIST OF APPENDICES

# CHAPTER 1

# INTRODUCTION

## 1.1 Background of the Research

Concrete is the most widely consumed material after water in the construction industry. According to Schutter and Audenaert (2004), though concrete is a rigid and porous material, these networks of interconnected pores interact with the environment. Consequently, it becomes susceptible to ingress of water and other aggressive fluids. Lulu et al. (2001) asserted that water ingress causes degradation and deterioration of concrete structures over time. Likewise, Chen et al. (2013) confirmed that infrastructures situated within an environment with relatively high humidity or close to water table are prone to deterioration due to the ingress of water.

Concrete is essentially a water-resistant material. However, Aldea et al. (1999) stated that water still permeates the exposed concrete structures such as pavement and bridge deck. This permeaition causes corrosion problems of the reinforcing steel bars and poor aesthetic of building façade. According to Neville (2002), variations in the ingredients mixed during its preparation can affect the degree of water-resistance and porosity. Also, type and quantity of interconnected pores in the concrete, as well as their spread within the matrix, largely influence the permeability. And this was also confirmed by Schutter and Audenaert (2004). Consequently, the service life of concrete material is adversely affected (Dai et al., 2010). Therefore, it becomes necessary to inspect and maintain concrete structure over time periodically. The inspection and maintenance techniques used for infrastructures have compelled attention. In this regard, the critical examination and subsequent maintenance of not so readily accessible infrastructures proved difficult due to the lack of funds required to cover the phenomenal costs

To this effect, concrete infrastructures need to be protected and thus, prolong the life span. Muhammad et al. (2015) reported that in an attempt to avoid traditional approach of detection and control of water seepage related problems, different approches were adopted by many researchers to develop waterproof concrete

According to National Corporation of Highway Research Program (NCHRP-244, 1981), waterproof material should not absorb more than 2.5% moisture in comparison to control specimen. However, German Committee on Reinforce Concrete (GCRC, 1991) recommends that waterproof concrete should not absorb more than 50% of the moisture/water in comparison to reference specimen. On the other hand, Basheer et al. (1997) asserted that to date, there were no universally adopted criteria for rating water resistance penetration. Alternatively, British Standard (BS EN 14695-2010) recommends that waterproof concrete is one that prevents passage of water from one plane to another. Consequently, a great deal of repair and maintenance cost could substantially be avoided. To this effect, Zhu et al. (2013) reported that the use of waterproof concrete plays a critical role in improving the performance of concrete infrastructure by extending their service life. According to Muhammad et al. (2015), this can be achieved through a method of external membrane, external coating and integral method as indicated in Figure 1.1.

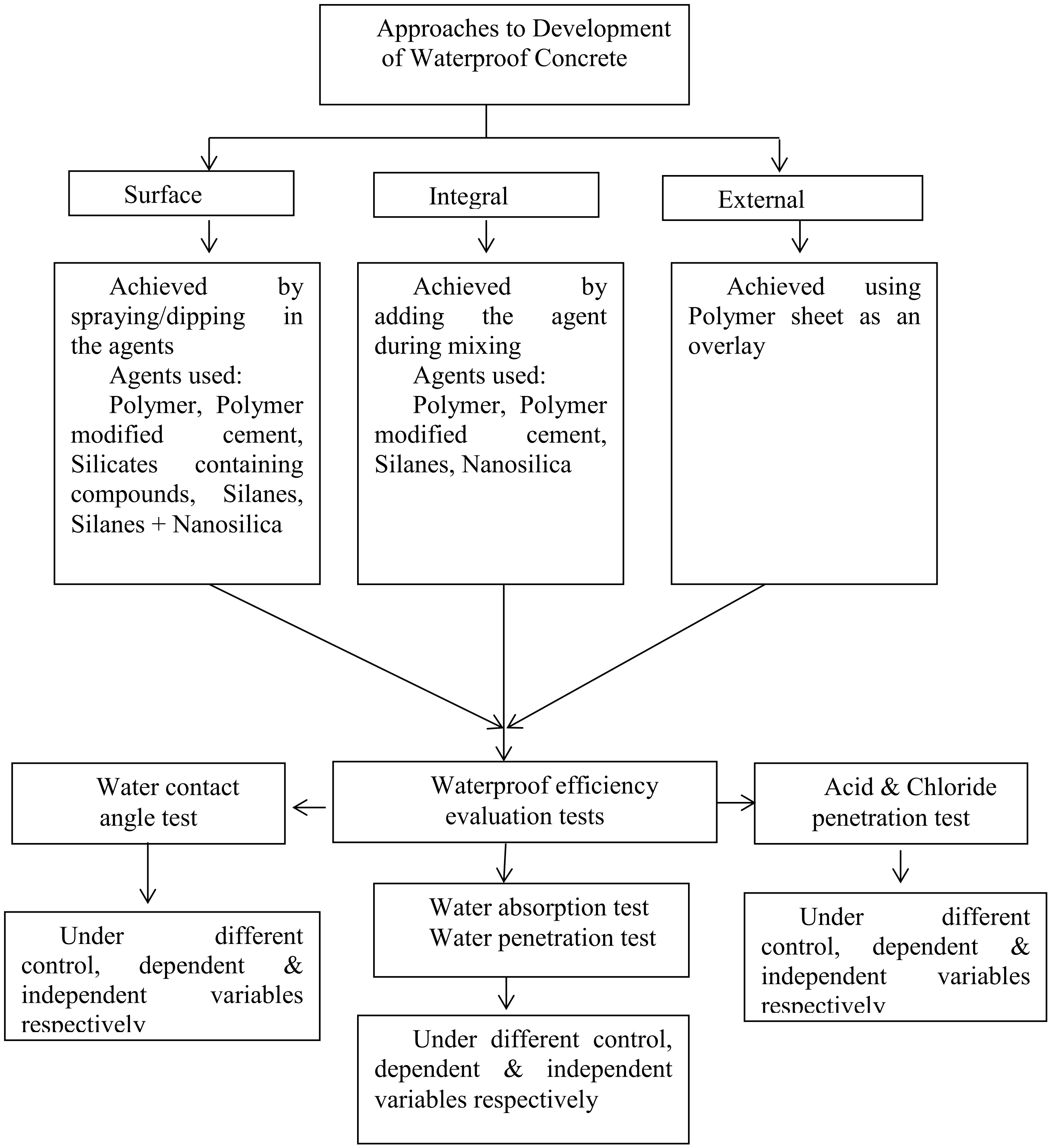


**Figure 1.1:** Taxonomy of approaches for the development of waterproof concrete (Muhammad et al., 2015)

Muhammad et al. (2015) stated that methods of external membrane and surface coating using waterproofing agents (solutions) are the common approaches for protection of concrete infrastructure. In the method of external membrane, sheets of polymers are usually overlaid on the concrete's surface. On the other hand, during the method of surface coating; waterproof solutions are sprayed on the exposed surface of concrete for rehabilitation of an old infrastructure or protection of newly cast concrete. The integral mixing method involves the addition of water repellent admixture during mixing of concrete and thus, it is exclusively for new infrastructure.

Therefore, polymer membrane, waterproof solutions and water repellent admixtures are the typical waterproofing agents. However, due to variability of mechanism of action of waterproofing agents in any of the methods, the performance of each of these agents varies. While some methods and agents are deficients, others were found to possess a remarkable attribute. Consequently, these restrict the extent to which each agent and method can be applied to develop waterproof concrete.

To increase water resistance of concrete, the use of waterproof membrane on concrete deck was investigated. In this regard, Zhou and Xu (2009) and Liu et al. (2014) studied the influence of surface roughness, material quantity, compaction temperature as well as environmental temperature on adhesive strength between the concrete deck and overlaid waterproof membrane. The results showed that adhesion between the membrane and the concrete progressively deteriorates due to the fluctuation of environmental conditions. According to Suffian (2013), long term protective effect of this membrane cannot be guaranteed

Also, Blight (1991) investigated and established the performance of silanes-based waterproof solutions as a surface coating for concrete infrastructure. The result indicated that the performance of silane as a waterproofing agent decreased over time. This decrease was attributed to the low viscosity of the waterproof solution, which makes its apparently difficult to sufficiently penetrates the concrete due to evaporation during application (Dai et al., 2010). In addition, Suffian (2013) asserted that long efficiency of surface coating agents is compromised and thus, need to be re-applied in the future.

To offset these limitations, the use of silane-based integral water repellent admixtures was acknowledged (Zhang et al., 2011). In the integral method , conventional approach using one-variable-at-a-time was the common practice by the previous studies in the optimization of water repellent admixtures. However, resistance to water and other transport properties was improved at an optimum amount of admixture compared to reference specimen. On the other hand, compressive strength was found to be significantly reduced. This finding was supported by Vejmelkova et al. (2012) and Zhu et al. (2013) where they reported significant resistance to water absorption at an optimum amount of zinc stearate and silane respectively. However, compressive strength was substantially reduced. In another study, Nunes and Slizkova (2014) investigated the performance of linseed oil as a water repellent admixture in lime mortar. Though the result indicated a significant resistance to water ingress, but compressive strength was also drastically reduced.

In view of the need to increase the level of alternatives to the existing waterproofing admixtures and perhaps, to improve their performance, the use of nano materials is currently found. To date, some studies were conducted to reduce transport properties in cement based materials and thus, to increase water resistance using different nano based materials (Hou et al., 2015; Hou et al., 2014; Zhang et al., 2012; Woo et al., 2008). In the recent years, while, Hallmann et al. (2010) have investigated the characteristics of nano silicon, on the other hand, there has not been a study on the use of hydrophobic nano crystalline silicon as cement based water repellent admixture.

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## 1.2 Problem Statement

Intrusion of water into concrete structures causes reinforcement corrosion, poor aesthetic of building façade, cracks and other forms of degradation. Likewise, due to water intrusion, other common defects of concrete structures such as fungal growth, salt crystallization, peeling of paint and dampness. A lots of funds have been employed in the rehabilitation of concrete structures Kenai and Bahar (2003) reported that construction of Algiers Airport was interrupted and abandoned without waterproofing for three years. Before the continuation of the project, three million dollars USD 3 Million was spent to affect the repair works. Likewise, Bhaskaran et al., (2013) stated that cost of building repair due to moisture related issues in United Kingdom (UK) was estimated at GBP 250 million. On the other hand, the cost of repair of building façade due to water damage accounted for 55.6% of some building value in United States (Liu & Scott, 2006). This was corroborated by Jumaat et al. (2006) where they reported the repair cost, in Italy to be about 50% of the total expenditure in some construction.

To protect concrete infrastructure, most researchers focused on the use of surface coating. However, due to variability of weather, waterproofing performance of the coating agents degrades over time. Consequently, future reapplicaton of coating agent becomes necessary. Alternatively, integral methods for waterproofing of concrete were adopted by few studies. In this approach, waterproofing performance of some few nano based materials was investigated and established However, use nano silicon is not yet reported to this effect. In addition, major setback of the integral method was the drastic fall in compressive strength of the concrete. Also, a well designed step for waterproofing experiment control test approach lacks in this subject area. Likewise, one variable optimization technique was the common approach adopted by previous studies to establish optimum mix ratio. Moreoever, this type of optimization technique has a major limitation since the complete influence of all variables affecting the experimental response cannot be illustrated, Likewise, the interactive effect of the variables is not possible. Furthermore, previous studies failed to establish macro and micro-structural waterproofing mechanism of water repellent admixtures.

## 1.3 Aim and Objectives of the Research

The aim of the study is to investigate and establish waterproofing performance of nano Silicon based cement mortar**.** The aim is to be achieved through the following objectives:

i) To characterise nano Silicon for application in cement mortar as waterproofing agent;

ii) To optimise mix ratio of nano Silicon and water cement ratio in cement mortar based on capillary absorption and compressive strength tests

iii) To design the steps for waterproofing experiment control tests on cement mortar with nano silicon at the optimum mix ratio

iv) To establish macro and micro-structural waterproofing mechanism of nano silicon based mortar

## 1.4 Scopes of the Research

The scope of this study covers only a fresh mix and thus, for new concrete Likewise, the range of strength of the mortar was kept between 15 $N/mm^2$ and 30 $N/mm^2$. Also, w/c between 0.38 and 0.5 are used.

**Scope on admixure dosage:** Furthermore, the range of nano silicon dosage between 0% and 12% by weight of cement was adopted. These choices comply with what other studies have commonly adopted in the literature.

**Scope on type of investigation:** Moreover, the study will include both analytical and experimental investigations.

**Scope on duration of waterproofing performance**: This study focuses on short term waterproofing performance of nano silicon. Consequently, performance of nano silicon cement mortar under aggressive environment is not covered.

**Scope on characterization of nano silicon**: The characteristics of nano silicon identified are both physical and chemical .Likewise, all the tests conducted in this study are under laboratory condition.

**Scope on optimization technique:** The optimization was based on two independent variables which are crtical to water absorption characteristics/transport properties of concrete. To this effect, the relatinship betwenn experiemntal variables was modeled. However, the model was not based on mahematical assumption rather emperical in nature.

The set of tests conducted during this study are based on American Society for Testing and Materials (ASTM), British Standards (BS) as well as International Union of Laboratories and Experts in Construction Materials, Systems, and Structures (RILEM). Some tests were conducted in accordance with other methods developed in the previous literature. Soon after these are established, comparison with the related studies was made with information on their precision nearly known

## 1.5 Significance of the Research

The study investigate and establish waterproofing performance of nano Silicon based cement mortar. This study designed waterproofing experimental control tests, which previous studies failed to incorporate..Therefore, concrete infrastructure produced using this approach can resist water ingress in both unsatutated and saturated condition. Consequently, this is useful for both submerged and unsubmerged infrastructure. Furtheremore, the approach can be used to produce material that will resist both gas (carbondioxide) permeability and water vapor transmission. Hence, the deterioration effect of acid rain on concrete infrastructure in tropical climate can be minimized. On the aggregate, this new approach will aid in avoiding lots of repair and thus, maintenance costs of infrastructures. Furthermore, this will be more beneficial to tropical climates countries

Likewise, the study introduces a nano silicon as a new construction material which has not been previousely used in the oconstruction industry. The nano silicon increases the water resistance of cement base material without impairing the compressive strength, which is common deficiency to the existing waterproofing admixtures. Also, while othe nano based materials reduce workability of conccrete, on the other hand, nano silicon increases the workability of cement mortar. In this regard, it can be used with minimum or no super plasticizer in concrete during mixing. To this effect, additional cost of super plasticizer can be avoided.

Moreover, previous studies have adopted traditional approach in the optimization of water repellent admixtures. On the other hand, nonlinear multivariate technique is employed in this study to establish optimum mix ratio. In this regard, interactive effect of experimental variables on the experimental response can be presented. .

The prospect of this study can also serve as a basis for further research. In this regard, a better understanding of the characteristics of nano silicon will be established. Ultimately, this will add value to the existing information in this subject area, and thus, aids to the advancement of the frontier of knowledge to this effect.

## 1.6 Thesis Organisation

A brief description for each chapter is presented as follows:

**Chapter 1: Introduction:** In this chapter, overall evaluation and logic behind conducting this research are provided. Also, clear and short descriptions of problem background, aim, and objectives, scope and limitations and the significance of the research are presented in this chapter.

**Chapter 2: Literature Review:** In this chapter, characteristics of various waterproofing materials are discussed. Likewise, the techniques adopted by previous studies for the optimization of mix ratio. deficiencies of these optimization techniques are also discussed.. Also, performance of various waterproofing agents is reviewed. Moreover, tests methods adopted by various studies to explain the mechanism of waterproofing function/action due to these agents are reviewed

**Chapter 3: Methodology:** In this chapter, detailed report of the analytical approach, materials, specimen preparation as well as the various test methods adopted during the experimental work are presented. In addition, results of these tests are presented in the subsequent chapters

**Chapter 4: Results and Discussion:** In this chapter, the examined physical and chemical characteristics of nano silicon are discussed. Likewise, the results and discussions on the modeling and optimization of experimental variables are presented and discussed. Furthermore, results of the the entire approach to producing waterproof cement mortar using nano silicon is submitted and discussed. Moreover, results and discussion on the validation of the output of the approach are presented.

**Chapter 5: Conclusions and Recommendations:** In this chapter, overall conclusions from the study and thus, recommendations for further research are presented. Likewise, contributions, as well as limitations of the study, are highlighted.

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