Study of the Effect of Nano- Materials on developing Properties of Concrete

By

Eng. Omar Mohmed Fathi Mohamed Mahmoud Eid

A Thesis Submitted to the

Faculty of Engineering at Cairo University

In Partial Fulfillment of the

Requirements for the Degree of

Master of Science

In

Advanced Materials and Nano-Materials

FACULTY OF ENGINEERING, CAIRO UNIVERSITY

GIZA, EGYPT

2018

Study of the Effect of Nano-Materials on developing Properties of Concrete

By

Eng. Omar Mohmed Fathi Mohamed Mahmoud Eid

A Thesis Submitted to the

Faculty of Engineering at Cairo University

In Partial Fulfillment of the

Requirements for the Degree of

Master of Science

In

Advanced Materials and Nano-Materials

Supervisors

Prof. Dr. Osama Abd El Ghafour Hodhod

Professor of Properties and Strength of Materials- Structural Department

Faculty of Engineering, Cairo University

Prof. Dr. El Saaid Ibrahim Zaki

Professor of Properties and Strength of Materials

Housing and Building National Research Center

FACULTY OF ENGINEERING, CAIRO UNIVERSITY

GIZA, EGYPT

2018

STUDY OF THE EFFECT OF NANO-MATERIALS ON DEVELOPING PROPERTIES OF CONCRETE

By

Eng. Omar Mohmed Fathi Mohamed Mahmoud Eid

A Thesis Submitted to the
Faculty of Engineering at Cairo University
in Partial Fulfillment of the
Requirements for the Degree of
MASTER OF SCIENCE
In
Advanced Materials and Nano-Materials

Approved by the
Examining Committee

Prof. Dr. Osama Abd El Ghafour Hodhod ------- ----------
(Thesis Main Advisor)

Prof. Dr. El Saaid Ibrahim Zaki ----------
(Advisor) Housing and Building National Research Center

Prof. Dr. Ahmed Mahmoud Maher ----------
(Internal Examiner)

Prof. Dr. Samir Saad Ibrahim Nassar ----------
(External Examiner) Housing and Building National Research Center

FACULTY OF ENGINEERING, CAIRO UNIVERSITY
GIZA, EGYPT
2018

Engineer: Omar Mohamed Fathi Mohamed Mahmoud Eid

Date of Birth: 29 /03 / 1985
Nationality: Egyptian
E-mail:omareid645@gmail.com
Phone: 01002020934
Address: 14 Hassan El-Emam Street, Nacr City, Cairo
Registration Date: 1 /10/2013
Awarding Date: / /2018
Degree: Master **Department:** Advanced Materials and Nano- Materials

Supervisors: Prof. Dr. Osama Abd EL Ghafour Hodhohd (Main Advisor)
Prof. Dr El Saaid Ibrahim Zaki (Advisor)
Housing and Building National Reserch Center

Examiners: Prof. Dr. Osama Abd EL Ghafour Hodhohd (Examiner)
Prof. Dr. El Saaid Ibrahim Zaki (Examiner)
Prof. Dr. Ahmed Mahmoud Maher (Internal Examiner)
Prof. Dr. Samir Saad Ibrahim Nassar (External Examiner)
Housing and Building National Reserch Center

Title of Thesis: **STUDY OF THE EFFECT OF NANO-MATERIALS ON DEVELOPMENT PROPERTIES OF CONCRETE**

Key Words: **Nano materials, Nano Calcium Carbonate, Nano Kaolin, Permeability, Scanning Electron Microscope analysis**

Summary:

This thesis aims to substitute cement mixtures using Nano particles of two different materials Nano Kaoline (NK) and Nano Calcium Carbonate $NCaCO_3$ to study its physical, mechanical and permeability performance and to provide a highly performance concrete mixture with feasibly economic cost.

The study is concerned in the synthesizing of (NK) and ($NCaCO_{3)}$ particles in the laboratory by applying simple techniques. Both Nano Materials are obtained by the calcination of Kaolinite and Calcium Carbonate and prepared by thermal activation at 800^0 C for 2 hours. The morphology of the synthesized NK and $NCaCO_3$ are analyzed using Transmission Electron Microscopy (TEM), and the minerals are identified using X-ray diffraction Florence (XRDF).Concrete specimens were prepared in the laboratory and the results were compared against control mix. The microstructure was characterized by means of Scanning Electron Microscope analysis (SEM). According to experiments, the optimum partial replacement of NK and $NCaCO_3$ is 7% and 1% respectively which increases both compressive and splitting tensile strength and enhances Coefficient of permeability. The NK gives better results in the ratio (splitting strength/ compression tensile strength) than $NCaCO_3$, while $NCaCO_3$ achieves better results in the coefficient of Permeability. From the study, it is recommended to add 1% $NCaCO_3$ for best enhancement of concrete besides its low cost.

ACKNOWLEDGEMENT

First of all, I would like to thank God for His guidance and care throughout my life and for his endless support to overcome any obstacles and difficulties that I faced during this research.

I would like to express my deepest gratitude to my supervisor **Prof. Dr.Osama Hodhod** Professor of properties of Material, Faculty of Engineering- Cairo University for his guidance, advices, encouraging, insightful discussions, knowledge learning he gave me. Also, I would like to express my deepest gratitude to my supervisor **Prof. Dr. El-Saaid Ibrahim Zaki Eldeen** Professor of Strength and Testing of Materials in Housing and Building National Research Center for his guidance, patience, encouraging, and valuable assistance during all stages of this research.

I would like to thank **Cairo University**, Faculty of Engineering for providing Laboratories and Library facilities.

I would also to express my gratitude towards **Prof. Dr. Niveen Maousof** for encouraging me to join this study (May her soul rest in peace).

I am grateful to **Prof. Dr. Samir Saad Ibrahim**, Head of Electro Mechanical Institute, HBRC for supporting me every step of the way.

Thanks to **Eng. Alla Eldean Morsi** Director of Quality Control in Lokma Factory.

I wish to express my love and gratitude to my beloved family for their support and encouragement through the duration of my studies. Thanks to my siblings for always being there for me whenever needed. Thanks to my youngest sister **Eng. Mariam Eid** for her help during this study. A special thanks to **Prof. Dr. Fathi Eid** (may God bless his soul) **Dr. Samia Emera** my amazing Parents for their precious advice on both academic and personal level throughout my life.

DEDICATION

To my amazing Dad **Prof. Dr. Fathi Eid** (m**ay** his soul rest in peace) as he has been always encouraging me to start my master.

To my wonderful Mother Dr**. Samia Emera** for her patience, encourage and always waiting for this moment with anxiety.

Table of contents

Title	Page
Acknowledgment ……..……..	I
Dedication ……..……..	II
List of contents ……..…….…….	III
List of tables……..…….....	VII
List of figures……..…….....	IX
List of abbreviations ……..……..	XII
Abstract……. ……..…….……..….	XIV
CHAPTER (I): INTRODUCTION	1
1.1. Background…….....	1
1.2. Definition of Nanotechnology…..…….	1
1.3. Importance of Nanotechnology in Civil Engineering……...	2
1.4. Research Objectives……..…	2
1.5. Research Scope …….……	3
1.7. Research Outlines…….…….…	1
CHAPTER (2): LITERATURE REVIEW	
2.1. Introduction……..……..…	4
2.2. Nanotechnology and concrete: Definitions…….....	4
2.2.1. Concrete: a complex, nano structured material……...	4
2.2.2. Definition of Nanotechnology in concrete……...…	5
2.3. Definition of Nanomaterial…….…….….....	5
2.4. Definition of Nanoparticles……..……..….	6
2.5. Nano particles Preparation……..……..……..…….....	6
2.6. Pozzolanic Materials…….…..……...…	7
2.6.1. Pozzolanic Materials Classification……..……..……...	7
2.6.2. Pozzolanic Material in Cement concrete…….…….	8
2.7. Supplementary Cementing Materials (SCMs)…….……..…	8

2.8. kaoline……..……...… 9

2.8.1. Introduction…… 9

2.8.2. Kaolinite mineral……...… 10

2.8.2.1. Structure and composites of kaolinite mineral……..… 10

2.8.2.2. The gensis of kaoline minerals……..… 10

2.8.2.3. The mining and processing of kaoline minerals……..… 11

2.8.2.4. Nano Metakaoline Production……..… 11

2.8.2.5. Properties of Metakaoline…… 12

2.8.2.6. kaoline Uses……..……..… 12

2.8.2.7. Advantages of kaoline……..… 12

2.9. The effect of Nano Clay on Concrete……..…..… 12

2.10. Nano Calcium Carbonate ($NCaCO_3$)……..…..… 15

2.11. The Effect of Nano $CaCO_3$ on Concrete……..…… 16

CHAPTER (3): EXPERMENTAL WORK

3.1. Introduction……..……...…….…..… 19

3.2. Materials……..……..……..… 19

3.2.1. Portland Cement…….……..……..…… 19

3.2.2. Fine Aggregate…..……..…….. 20

3.2.3. Aggregates…….…… 22

3.2.4. Water……...…… 23

3.2.5. Chemical Admixture…… 23

3.2.6. Silica Fume……..……. 24

3.2.7. Kaoline…… 25

3.2.8. Calcium Carbonate ($CaCO_3$)……..……..… 30

3.3. Specimen Preparation and Procedures…….……..…. 35

3.3.1. Mixes Proportions…….……..…. 35

3.3.2. Mixing Technique for Nano particles……. 36
3.3.3. Casting and molding of samples …….…….....….. 37
3.4. Concrete Properties Tests…….……. 38
3.4.1. Fresh cement Mortar Properties Tests……..…..… 39
3.4.2. Hard cement Mortar Properties Tests……...… 39
3.4.2.1. Water Permeability Test……..… 41
3.4.3. Mechanical Properties Tests……..… 41
3.4.3.1. Compressive Strength Tests……...… 42
3.4.3.2. Splitting Tensile Strength Tests…….…… 42
3.5. Mineralogical Characteristics Tests…….…..… 43
3.5.1. Transmission Electron Microscope (TEM)…….. 43
3.5.2. Scanning Electron Microscope (SEM)……. 45
3.6. Chemical Analysis by X-Ray Fluorescence Spectrometry (XRFS)……..... 4

CHAPTER (4): ANALYSIS AND DISCUSSION OF TEST RESULTS

4.1. Introduction…….……... 47
4.2. Properties of Fresh Concrete……...…... 47
4.2.1. Slump Flow Test……..... 47
4.2.2. The results of flow test of NK…….……..... 47
4.2.3. The results of flow test of NCaCO3…….……... 48
4.3. Properties of Hardened Concrete…….….. 49
4.3. 1.Compressive Strength……..…….. 49
4.3.1.1. Effect of using $NCaCO_3$ in Compressive Strength……...… 49
4.3.1.2. Effect of using NK on Compressive Strength……..……. 51
4.3.2. Splitting Tensile Strength……. 53
4.3.2.1. Effect of using $NCaCO_3$ on Tensile Strength……. 53
4.3.2.2. Effect of using NK on Tensile Strength …….. 54

4.3.3. Relation between Compressive and Tensile Strength……. 55

4.3.4 Water Permeability test results……. 57

4.3.5. Microstructure Analysis……. 60

4.3.5.1. SEM Results for specimens with 1% $NCaCO_3$ ……. 61

4.3.5.2. SEM Results for specimens with 3% $NCaCO_3$…… 63

4.3.5.3. SEM Results for specimens with 7% NK…… 65

4.3.5.4. SEM Results for specimens with 8% NK…… 68

4.3.5.5. SEM Results for specimens with 10% NK…… 70

4.3.5.6. SEM Results for specimens with 4% $NCaCO_3$..... 73

4.6. Economic Feasibility Study of using nano materials in concrete construction 76

CHAPTER (5): CONCLUSIONS, RECOMMENDATIONS AND FUTURE WORK

5.1. Introduction..…..……..…….. 80

5.2. Conclusion…….…… 80

5.3. Recommendations…….. 81

5.4. Future Work……... 81

REFRENCES……..... 83

Appendices…….**.....** 91

Appendix (1)…… 92

Appendix (2)…… 93

Appendix (3)…… 95

Appendix (4) .…….. 97

Appendix (5)……. 99

Appendix (6) ……. 101

List of Tables

CHAPTER (3) **Page**

Table 3.1: Physical and Chemical Properties of cement used……..…..... 29

Table 3.2: Chemical composition of materials used by weight……..….. 29

Table 3.3: Physical Properties of fine aggregates…….……..…..... 31

Table 3.4: Physical Properties of Coarse aggregates……... 32

Table 3.5: Technical Data and Physical properties of GLENIUM C315……..… 33

Table 3.6: Physical Properties of Silica Fumes……..…….**.....** 34

Table 3.7: Physical Properties of NK……..……..… 35

Table 3.8: Grading of Kaoline %**.....** 35

Table3.9: Mineral Composition of Kaoline…….**.....** 36

Table 3.10: Physical Properties of CaCO3…… ……..… 40

Table 3.11: Chemical Composition of CaCO3 by weight……..……..…… 41

Table 3.12: Proportions of concrete samples on weight basis ……..… 47

Table3.13: Specifications of water vane motor …….…….…. 48

Table3.14: Comparison in Specification between light microscope and TEM…..**..** 55

CHAPTER (4)

Table 4.1: Slump Flow Test results for NK mixes and Control mix……..…. 63

Table 4.2: Slump Flow Test results for Nano CaCO3and Control mix…… 64

Table 4.3: Compressive strength results for for mixtures containing NCaCO3…..**..** 65

Table 4.4: Percentage of increase of Compressive Strength for NCaCO3……**...** 65

Table 4.5: Compressive strength results for mixtures containing NK.……..…….. 67

Table 4.6: Percentage of increase of Compressive Strength for NK ……**.....** 67

Table 4.7: Splitting Tensile Strength of $NCaCO_3$ mixtures ……...……. 69

Table 4.8: Percentage of increase of Splitting Tensile Strength of $NCaCO_3$ ……**..** 69

Table 4.9: Splitting Tensile Strength of NK mixtures…..…… 70

Table 4.10: Percentage of increase of Splitting Tensile Strength of NK……..…… 71

Table 4.11: Comparison between (F_{tst}/F_C) using different NK mixtures…….……. 72

Table 4.12: Comparison between (F_{tst}/F_C) using different $NCaCO_3$ mixtures…… 73

Table 4.13: Water Permeability Result tests after 28 Days……..… 74

Table 4.14: Materials Price list…….……..……..…… 94

Table 4.15: Total Prices of Mixtures with and without $NCaCO_3$……..… 94

Table 4.16: Total Prices of Mixtures with and without NK…….…… 95

Table 4.17: Excess Compressive Strength unit Price of Mixture ……..….. 96

List of Figures

CHAPTER (3) **Page**

Figure 2.1: Nano particle size [38]……. 10

Figure 2.2: Illustration of the 'top down' and 'bottom up' approaches [39]…… 11

Figure 2.3: Location map of Kaoline of Gebel Gunna Egypt…….…… 14

Figure 2.4: Diagrammatic sketch of the structure of kaolinite mineral……. 15

CHAPTER (3)

Figure 3.1: Ordinary Portland cement ……..…… 28

Figure 3.2: Testing sand used in Lab…… 30

Figure 3.3: Fine aggregate used grading curve……..….. 30

Figure 3.4: Coarse aggregate used grading curve…….…… 31

Figure 3.5: Silica Fume Powder…….…….. 34

Figure 3.6: SEM micrograph of Silica Fume particles……..…… 34

Figure 3.7: Kaoline Powder……..……. 35

Figure 3.8: Automatic electrical furnace…… 36

Figure 3.9a: TEM micrograph of NK…… 37

Figure 3.9b: TEM micrograph of NK mean size particles 24nmm……..…. 37

Figure 3.10: TEM micrograph of NK mean size particles 72nmm…..……..…… 39

Figure 3.11.a: TEM micrograph of NK means size particles 173nmm..... 39

Figure 3.11.b: TEM micrograph of NMK means size particles 92nmm…….. 40

Figure 3.12: Calcium Carbonate Powder……..…… 41

Figure 3.13: Morphology of Calcium Carbonate Nanoparticles……....…… 42

Figure 3.14: TEM micrograph of nano-$CaCO_3$ size 40nm…… 42

Figure 3.15: TEM micrograph of nano-$CaCO_3$ size 24 nm……..…… 43

Figure 3.16:: TEM micrograph of nano-$CaCO_3$ (rejected) ……..…… 43

Figure 3.17: TEM micrograph of nano-$CaCO_3$ size (rejected)…..... 44

Figure 3.18: TEM micrograph of nano-$CaCO_3$ (rejected)…… 44

Figure 3.19: TEM micrograph of nano-$CaCO_3$ **(rejecte)…….…** 45

Figure 3.20: TEM micrograph of nano-$CaCO_3$ **(rejecte)…….……** 45

Figure **3.21:** Van Motor **……..……...…** 48

Figure 3.22: The Rotary Mixer used**……..……** 49

Figure 3.23: Mold for Concrete samples (15x15x15) cm and cylinder (15x30) cm**..** 4

Figure 3.24: Slump Cone…….…..… 9

Figure 3.25: Slump flow is measured.…….……...… 51

Figure 3.26: Water Permeability Equipment……..…… 51

Figure 3.27: Adjusting the specimen**…….…...…..** 52

Figure 3.28: Specimen extracted from Permeability Equipment**……..…….** 52

Figure 3.29: Specimen after splitting Test**……..……** 52

Figure 3.30: Specimen water penetration depth…….…...….…… 53

Figure 3.31: Universal testing machine SHIMADZU 500 KN**…….……** 54

Figure 3.32: Universal testing machine……..…… 55

Figure 3.33: Comparison in Configuration between light microscope and TEM… 56

Figure 3.34: The Transmission electron microscope (TEM)……..….……. 57

Figure 3.35: Diagram of TEM…… 57

Figure 3.36: Scanning Electron Microscope (SEM)……..…… 58

Figure 3.37: (Axios, WD-XRF, PANalytical 2005, Netherlands)……..… 60

CHAPTER (4)

Figure 4.1: Slump Flow diameter for mixes containing NK…..…… 63

Figure 4.2: Slump Flow diameter for mixes containing Nano CaCO3…….……. 64

Figure 4.3: Compressive strength results for mixtures containing NCaCO3 ……. 66

Figure 4.4: Compressive strength results NCaCO3 …… 66

Figure 4.5: Compressive strength results for mixtures containing NK …… 68

Figure 4.6: Compressive strength results NK……..… 68

Figure 4.7: Splitting Tensile strength results for mixtures NCaCO3……..….. 70

Figure 4.8: Splitting Tensile strength results for mixtures NK……..…..…. 71

Figure 4.9: Comparison between spitting and compressive strength for NK……... 70

Figure 4.10 Comparison between spitting and compressive strength for $NCaCO_3$…. 73

Figure 4.11: Water penetration depths for mixtures NCaCO3……. 75

Figure 4.12: Coefficient of permeability for mixtures NCaCO3…… 75

Figure 4.13: Water penetration depths for mixtures NK…… 76

Figure 4.14: Coefficient of permeability for mixtures NK…….. 76

Figure 4.15: SEM for Control Sample……..…… 78

Figure 4.16a: SEM for formation of the carboaluminates crystals in concrete… 79

Figure 4.16b: SEM for formation CSH in concrete…… 80

Figure 4.16c: SEM for formation of pores in concrete…….. 80

Figure 4.17a: SEM for Control Sample…….. 81

Figure 4.17b: SEM for agglomeration of nano-$CaCO_3$…….. 83

Figure 4.18a: SEM for Control Sample…….. 84

Figure 4.18b: SEM for NK 7%..... 84

Figure 4.18c: SEM for NK 7%..... 85

Figure 4.18d: SEM for NK 7%..... 85

Figure 4.19a: SEM for Control Sample……... 86

Figure 4.19b: SEM for NK 8%..... 87

Figure 4.19c: SEM for NK 8%..... 87

Figure 4.20a: SEM for Control Sample…….…..… 88

Figure 4.20b: SEM for NK 10%..... 89

Figure 4.20c: SEM for NK 10%..... 89

Figure 4.20d: SEM for NK 10%..... 90