DEVELOPMENT OF GREEN PAVEMENT TO REDUCE NOx EMISSIONS

By:

Abdul Moiz Sohail

Beenish Imtiaz

Muhammad Faizan Nadeem Sidiqi

Institute of Environmental Sciences and Technology

School of Civil and Environmental Engineering

National University of Sciences and Technology

2009-2013

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By

Abdul Moiz Sohail (2009-NUST-BE-ENV-01)

Beenish Imtiaz (2009-NUST-BE-ENV-4)

Faizan Nadeem Sidiqi (2009-NUST-BE-ENV-)

A Thesis Submitted In Partial
Fulfillment of the Requirement For The
Degree Of

BACHELOR

IN

ENVIRONMENTAL ENGINEERING

Institute of Environmental Sciences and Engineering (IESE)

School of Civil and Environmental Engineering (SCEE)

National University of Sciences and Technology (NUST)

Islamabad, Pakistan.

2009-2013

APPROVAL SHEET

Certified that the contents and form of thesis entitled “Development of Green Pavement to Reduce NOx Emissions” submitted by Abdul Moiz Sohail, Beenish Imtiaz and Faizan Nadeem Sidiqi, have been found satisfactory for the requirement of the degree.

Supervisor : ____________________

Dr. Ishtiaq A. Qazi,

Associate Dean, IESE, NUST.

Members:

Dr. Zaheer ud Din ____________________

Head of Department, IESE, NUST.

Dr. Ishtiaq A. Qazi ____________________

Associate Dean, IESE, NUST.

This thesis is dedicated to our parents for their undivided love and support

Abdul Moiz Sohail

Beenish Imtiaz

Faizan Nadeem Sidiqi

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Abstract

Air pollution is a rapidly growing environmental issue in Pakistan, 70% of which is caused by vehicular emissions. 50% of the NOx is due to vehicular emissions incurring an annual cost of 170 M USD in terms of health cost. This research focuses on developing a rigid pavement by using TiO_2 nanoparticles and checks its efficacy of photo catalytic air cleaning.

Anatase nanoparticles were used in preparation of three types of specimens; 5% cement substituted with TiO_2 nano-particles, 10% cement substituted with TiO_2 nano-particles and surface coated blocks. These were then compared with conventional concrete specimen and also each other for compressive strength. The concrete block containing 10% TiO_2 nano-particles showed maximum strength. Overall, as the amount of nano-particle in mix design increased, strength of concrete increased.

Preliminary testing was carried out using methyl orange solution using a 15 mg/L solution for 42 days. These tests showed that maximum photo-catalytic degradation of methyl orange took place for 10% TiO_2 block followed by surface coated block and 5% TiO_2 block.

Confirmatory testing was carried out under UV light using ambient air NOx analyzer. The surface coated sample showed maximum relative degradation, nearly equal to 10% TiO_2 block. 5% TiO_2 block showed minimum degradation.

Surface coated block proved to be efficient in degradation of NOx and showed good strength but the coating could be eroded completely over the life span of the pavement. On the contrary the design containing 10% TiO_2 will be exposed to fresh active layer of embedded nano-particles even when eroded.

Acknowledgments

Firstly, we wish to thank Allah, who made all things possible.

We would also like to express our greatest gratitude to our supervisor, ***Dr. Ishtiaq A. Qazi***, who has helped and supported us throughout our project. We are grateful to him for his continuous support for the project, from initial advice and contacts in the early stages of conceptual inception and through ongoing advice & encouragement to this day.

Special thanks goes to ***Dr. Syed Ali Rizwan (HoD Structural Engineering)*** and ***Mr. Muhammad Ismail (Lab Assistant, Structure Lab***) who helped us in completing the project & exchanging interesting ideas, thoughts & made this project easy and accurate.

We would like to thank our parents for their undivided support and interest, without whom we would be unable to complete our project. Last but not the least we wish to thank our friends who appreciated and motivated us.

Abdul Moiz Sohail

Beenish Imtiaz

Faizan Nadeem Sidiqi

List of Abbreviations

ACI - American Concrete Institute
ADB - Asian Development Bank
Ads. - Adsorbed
Ag - Silver
B - Billion
Cr - Chromium
CO - Carbon monoxide
CO_2 - Carbon dioxide
Fe - Iron
H_2O - Water molecule
HO• - Hydroxyl radical
hv - Photons in the form of light
M - Million
mg/L - Milligrams per liter
NEQs - National Environmental Quality Standards
nm - Nanometer
NOx - Oxides of nitrogen
O_3 - Ozone
OH^- - Hydroxyl ion
PKR - Pakistani Rupees
PM_{10} - Particulate matter with size less than 10 microns
$PM_{2.5}$ - Particulate matter with size less than 2.5 microns
SUPARCO - Space and Upper Atmosphere Research Commission
Sur. - Surface
TiO_2 - Titanium dioxide
USD - United States Dollar
USEPA - United States Environmental Protection Agency
UV - Ultraviolet
VOCs - Volatile Organic Compounds
WHO - World Health Organization
$\mu g/m^3$ - Micrograms per cubic meter

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1. Introduction

1.1. Background

Air pollution is a rapidly growing environmental problem in Pakistan. The major causes of deterioration of ambient air quality are (Khwaja and Khan, 2005):

- Highly inefficient energy use;
- Accelerated growth in vehicle population and vehicle kilometers travelled;
- Industrial activity without adequate air emission treatment or control;
- Open burning of solid waste; and
- Use of ozone depleting substances.

Air pollution from motor vehicles is a major issue in almost all big cities of the world. 70% percent of the air pollution in Pakistan is caused due to Motor Vehicles (Imtiaz et al., 2008). During the 20 year period i.e. 1985 to 2005, Pakistan’s road traffic grew at an alarming annual average rate of 14.1% (Asian Development Bank, 2006), consequently aggravating the air pollution issues. Figure 1 depicts the doubling of motor vehicles over a span of 15 years i.e. from 2.4 million in 1990 to 5.4 million in 2005 (Asian Development Bank, 2006). High pollution percentage is accounted by the use of poor quality of fuel and low vehicle maintenance.

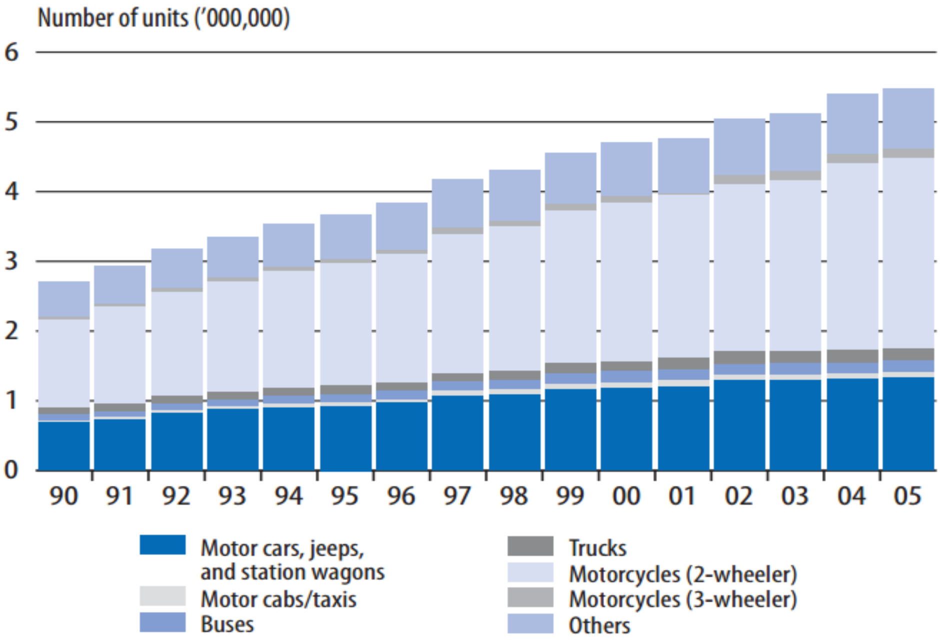


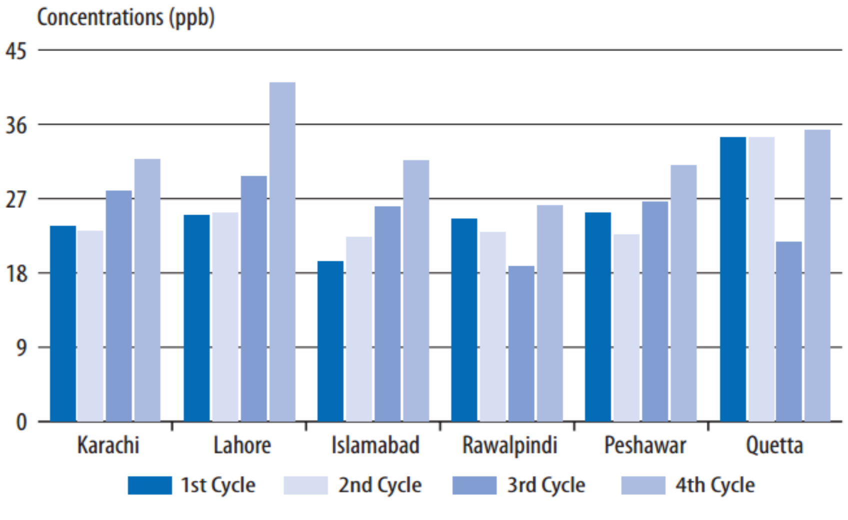
Figure 1: Number of Registered Motor Vehicles in Pakistan (ADB, 2006)

Though vehicular emissions are composed of a number of gases, NOx, VOCs, O_3, CO, CO_2, PM 10 and PM 2.5, this project focuses on NOx levels only. 50% of the NOx enters the environment through vehicular emission (USEPA, 1999). NOx is one of the many families of pollutant gasses that have been an issue for urban areas and settlements. NOx gases are also potential greenhouse gases, have a potential to deplete ozone layer that filters out harmful UV radiations in the stratosphere, and can act as a precursor for formation of photochemical smog. NOx reacts with ammonia, moisture, and other compounds to form nitric acid vapor and related particles. Small particles can penetrate deeply into sensitive lung tissue and damage it, causing premature death in extreme cases. Inhalation of such particles may cause or worsen respiratory diseases, such as emphysema or bronchitis, or may also aggravate existing heart diseases (Wikipedia).

Table 1: NOx Family (USEPA, 1999)

Formula	Name	Properties
N_2O	Nitrous oxide	Colorless gas; Water soluble.
NO	Nitric oxide	Colorless gas; Slightly water soluble.
N_2O_2	Dinitrogen dioxide	Colorless gas; Slightly water soluble.
N_2O_3	Dinitrogen trioxide	Black solid; Water soluble; Decomposes in water.
NO_2	Nitrogen dioxide	Red-brown gas; Very water soluble; Decomposes in water.
N_2O_4	Dinitrogen Tetraoxide	Red-brown gas; Very water soluble; Decomposes in water.
N_2O_5	Dinitrogen Pentoxide	White solid; Very water soluble; Decomposes in water.

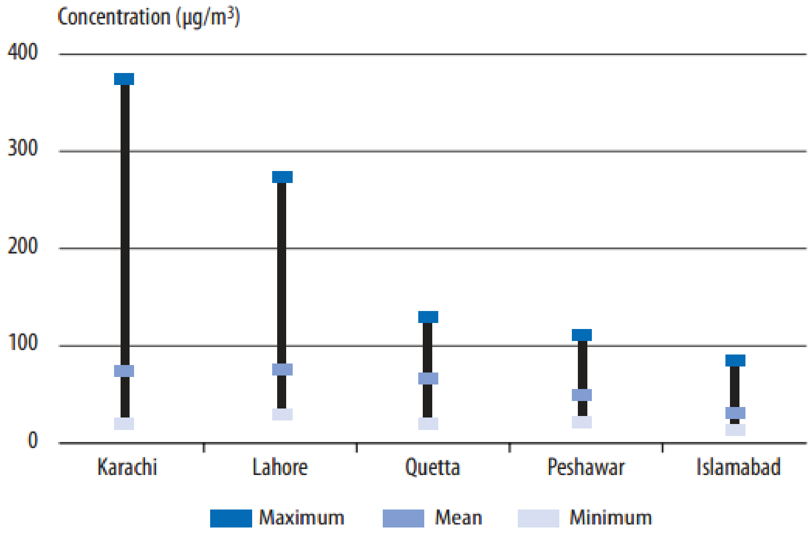
SUPARCO made observations of NOx levels in major cities of Pakistan as shown in Figure 2.



Source: SUPARCO (2005).

Figure 2: NOx Level in Major Pakistani Cities (ADB, 2006)

Internationally the standards are set for nitrogen dioxide (NO_2). The safe level of long-term exposure to NO_2 is set by WHO at 40 µg/m^3 for 1-year monitoring, while for short-term monitoring (1 hour), the threshold is set at 200 µg/m^3 (Asian Development Bank, 2006). According to NEQs ambient air concentrations of nitrogen oxides, expressed as NO_2, should not exceed 100 µg/m^3 (0.05 ppm = 50 ppb).



Source: Lodhi (2006).

Figure 3: Ambient Levels of Nitrogen Dioxide in Different Cities in Pakistan (ABD, 2006)

Karachi had the highest or maximum recorded levels followed by Lahore, Quetta, Peshawar, and Islamabad. Average concentrations showed that Karachi and Lahore have similar concentrations at 76 µg/m^3. The average concentrations of NO_2 in Quetta, Peshawar, and Islamabad were 69.50 µg/m^3, 47.28 µg/m^3, and 30.41 µg/m^3, respectively. The lowest recorded level of NO_2 (11.65 µg/m^3) was found in Islamabad in the residential area along embassy road, while the highest recorded level (399.65 µg/m3) was found at Karimabad Junction in Karachi (Asian Development Bank, 2006).

Table 2: Annual Health Cost

City	Annual Health Costs Due to Vehicles (M USD)
Karachi	69.79
Lahore	33.16
Faisalabad	13.56
Rawalpindi and Islamabad	7.74
Multan	10.37
Hyderabad	1.84
Gujranwala	6.79
Peshawar	3.11
Quetta	1.19
Total	**147.55**

Annual health cost due to vehicles is 147.55 M USD (Qamar uz Zaman). According to this study NOx contributed to 48.9% of the total cost i.e. 72.15 M USD (Wijetilleke L. et al, 1993). The vehicular population has increased from 3.4 M in 1993 to 5.4 M in 2005, thus contributing 114.59 M USD to the health cost due to vehicles.

1.2. Objective of the Research

The objective of this research is to develop a rigid pavement by using TiO_2 nanoparticles and check its efficacy of photo catalytic air cleaning.

1.3. Environmental Nanotechnology at IESE

A lot of research work has been done and is being carried out on environmental nanotechnology. Students of MS from IESE have been working on nanotubes and nanoparticles for air and water quality improvement.

Hassan Younas has presented water disinfection by using metal doped titania nanoparticles. In this study Ag doped titania nanoparticles have proven to be very effective photo-catalytic agent against gram negative (*E.coli*) and gram positive (*S.aureus*) bacterial groups (MS Thesis, IESE, 2011).

Muhammad Ihsan Danish has worked on removing arsenic from water using pure and metal doped titania nanoparticles. 90% arsenic removal was seen by using Fe and Cr doped titania nanoparticles from a solution containing up to 2 mg/L of heavy metals (MS Thesis, IESE, 2012).

Saman Khan (Khan et al., 2013) has worked on self-sanitizing surface by using Ag-doped TiO_2. In this work, the photo-killing effects of 1% silver doped titanium dioxide TiO_2 were studied. The Ag-TiO_2 nanoparticle coatings were applied on glass and venetian blind surfaces and were effective in generating a loss of viability of two bacteria (Pseudomonas aeruginosa, Bacillus subtilis) after two hour of illumination under normal light in the visible spectrum.

Currently Waqas Latif, Khadija Nawaz and Naushaba Noureen are working with nanotube on removal of air borne bacteria, degradation of fluoriquinoline and degradation of phenols respectively.

2. Literature Review

2.1. Titanium dioxide

2.1.1. Characteristics and Applications

The main component for a photo catalytic product is TiO_2. It is the fourth most easily available element in the earth's crust and has powerful oxidizing properties. TiO_2 has three main forms; rutile, anatase and brookite (Venurini and Bacchi, 2009).

TiO_2 used as nanoparticles has a great specific surface and smaller diameter particles i.e. less than 100 nm. Anatase is the most suitable TiO_2 type due to its high photochemical reactivity and stability in aqueous systems. It is obtained by heating the amorphous form of TiO_2 until 300 C (Venurini and Bacchi, 2009).

The most important aspect for vehicular traffic is that TiO_2 degrades air pollutants at ordinary temperature using sunlight (Venurini and Bacchi, 2009).

2.1.2. Photo-catalytic Process

When TiO_2 is exposed to UV light below 388 nm, electrons jump from valence band (VB) to covalent band (CB) and two electrons and a positive hole is formed. Consequently the surface water is oxidized by positive hole and compounds can be rapidly oxidized by TiO_2 (Huang et al., 1999):

$$TiO_2 + hv \longrightarrow h_{vb}^{+} + e_{cb}^{-}$$

The hole on TiO_2 particle surface react with adsorbed water or surface OH- group to form HO• radicals:

$$h_{vb}^{+} + H_2O(ads.) \longrightarrow HO\bullet + H^{+}$$

or

$$h_{vb}^{+} + OH^{-}(sur.) \longrightarrow HO\bullet$$

Electrons in conduction band react with molecular oxygen to form superoxide ions:

$$e_{cb}^{-} + O_2 \longrightarrow O2\bullet^{-}$$

As a result of the reaction more HO• radicals are formed:

$$2O_2\bullet^{-} + 2H_2O \longrightarrow 2HO\bullet + 2OH^{-} + O_2$$

In the presence of NO_2, following reaction occurs (Giavarini, 2004):

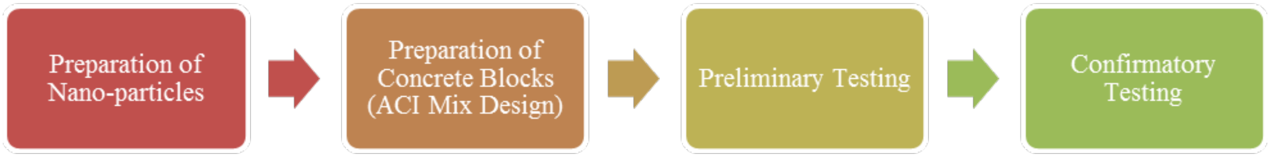
$$NO_2 + OH\bullet \longrightarrow HNO_3$$

$$2HNO_3 + Ca(OH)_2 \longrightarrow Ca(NO_3)_2 + 2H_2O$$

By the end of this reaction NO_2 has been converted into a harmless and very soluble compound i.e. $Ca(NO_3)_2$.

3. Materials and Methods

3.1. Methodology



3.2. Preparation of Nano-particles

TiO_2 was used to prepare anatase nanoparticles by following a series of steps as mentioned below:

1. 25 g of powdered TiO_2 was added to 100 ml of water in a beaker and kept for stirring on a magnetic stirring assembly for 24 hours.
2. After the period for stirring was completed, the beaker was kept on a cabinet and the contents were allowed to be settled for 24 hours.
3. Then the beaker with settled TiO_2 was kept in oven at 105 C for 12 hours to allow the water to dry.
4. After all the water had dried up, the dried TiO_2 in the beaker was scratched carefully and transferred to mortar-pestle set for crushing.
5. Once the contents were crushed, they were transferred to a china dish. The china dish was then placed in a muffle furnace for calcination at 550 C for 6 hours.

The nano-particles were characterized using X-Ray Difraction (XRD) and Scanning Electron Microscopy (SEM). The XRD image showed that the nano-particles were in the required ‘anatase’ phase. The SEM image indicated that the size of the prepared nano-particles was well within the nano range.

3.3. Preparation of Concrete Blocks (Specimens)

ACI mix design for a rigid concrete pavement having a 28th day compressive strength of 35 MPa has been used for composing test concrete cubes (specimens). The size of each cube is 64 $inch^3$.

To check the efficacy of photo catalytic oxidation potential of TiO_2 in pavements, three different application methods were evaluated in this study:

1. 5% cement in concrete substituted by TiO_2 nano-particles
2. 10% cement in concrete substituted by TiO_2 nano-particles
3. Surface coating; coating plain concrete with slurry containing 20% cement substituted by TiO_2 nano-particles.

The above mentioned three categories of specimens have been prepared according to the ACI mix design.

Figure 4: Cement, Coarse Aggregate, Fine Aggregate, TiO2 Nanoparticles, Water and Molds

Figure 5: Molds; Oiled and Ready for Casting

Figure 6: Molds; Being Filled with Concrete Mix

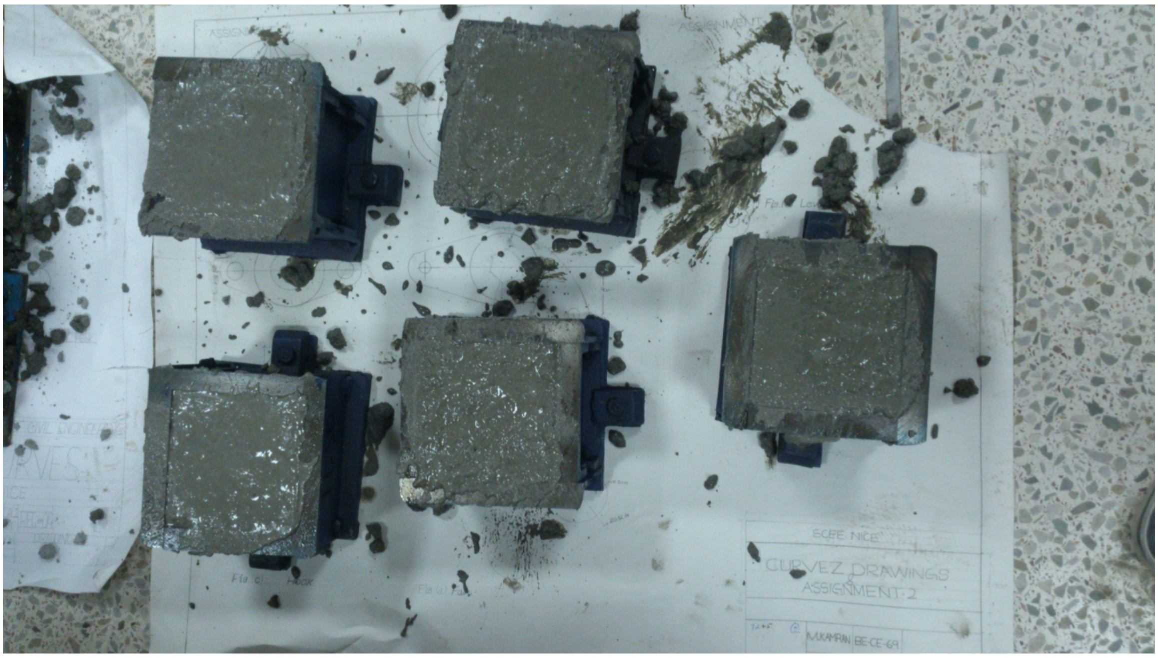


Figure 7: Molds: Completely Filled With Concrete Mix

Figure 8: Concrete Blocks to be De-molded

Figure 9: De-molding

Figure 10: Concrete Blocks

Figure 11: Curing of Concrete Blocks

3.4. Compressive Strength of Specimen

Table 3: Compressive Strength of Specimens

Specimen Type	7 Day Compressive Strength (MPa)	28 Day Compressive Strength (MPa)
Plain	19.3	29.7
Surface Coated	20.3	31.2
5% TiO2	22.0	33.9
10% TiO2	22.7	34.9

The specimens containing TiO_2 incorporated in mix design depicted more strength than conventional concrete. From Table 3 we can see that 28 day compressive strength for specimens having 10% cement substituted by nano-particles showed greatest strength of 34.9 MPa followed by specimens having 5% TiO_2 nano-particles. From here we can deduce that TiO_2 nano-particles have some sort of binding effect on concrete properties.

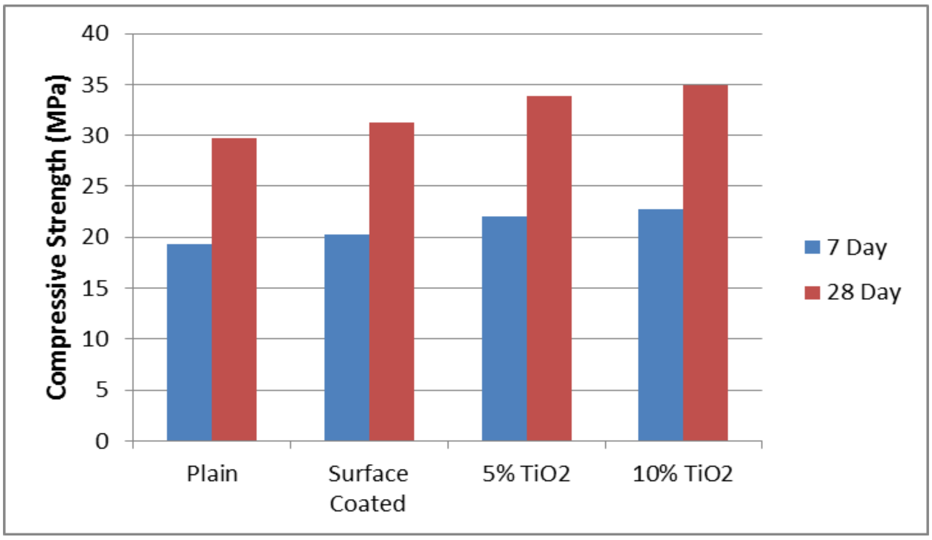


Figure 12: Compressive Strength of Specimens

4. Discussion and Analysis

4.1. Preliminary Testing

Methyl orange is an indicator that is often used in titration. It is prepared by treatment of helianthine with sodium hydroxide. Methyl orange is found in powdered form and a solution of required concentration can be prepared.

Figure 13: Powdered form of Methyl Orange

From table top research, it was seen that methyl orange showed maximum absorbance at 464 nm for any particular concentration.

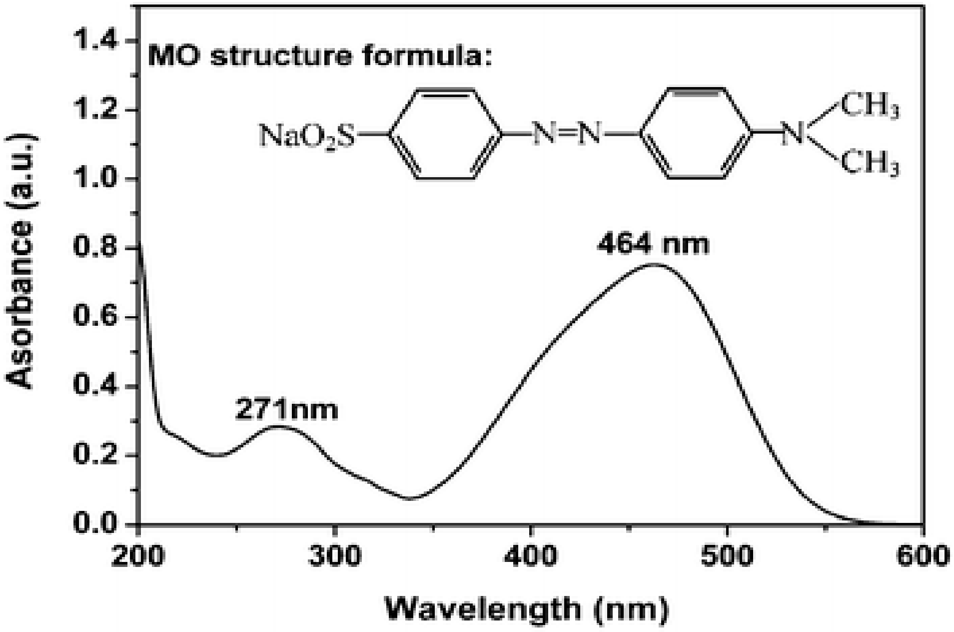


Figure 14: Absorbance of Methyl Orange

The first step was checking the validity of Beer-Lambert Law for the methyl orange. For this a stock solution of 500 mg/L was prepared. By using this stock solution, different solutions ranging from 1 to 15 mg/L were prepared and the absorbance of these solutions was measured using spectrophotometer at a wavelength of 467 nm. This wavelength of 467 nm was determined by individually analyzing each concentration in the range of 460 nm to 470 nm, by steps of 1 nm. It was observed that maximum absorbance occurred at 467 nm.

Figure 15: Methyl Orange Solutions (1-15 mg/L)

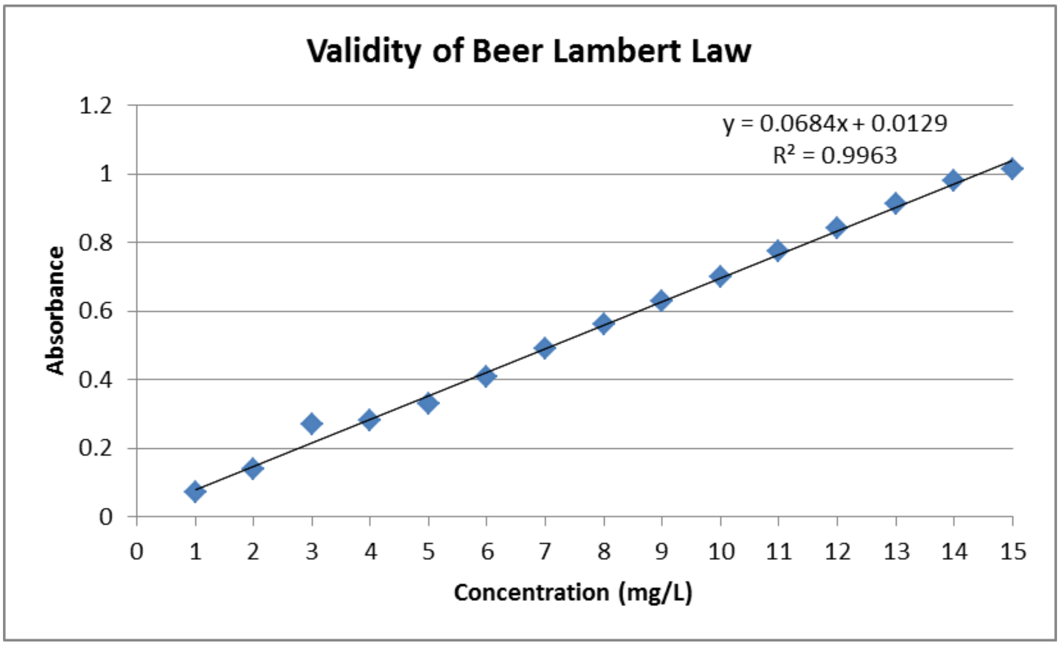


Figure 16: Checking Validity of Beer Lambert Law for Methyl Orange

The graph plotted for concentration versus absorbance, gave the value of $R^2 = 0.9963$, when linear regression line was drawn. This satisfactorily validates the Beer Lambert Law for methyl orange ranging from a concentration of 1 mg/L to 15 mg/L.

A detailed analysis was done for the concentration of 15 mg/L solution of methyl orange. It gave a maximum absorbance of 1.016 at 467 nm. Hence, 15 mg/L was selected as the solution in which the blocks were to be submerged for testing initially.

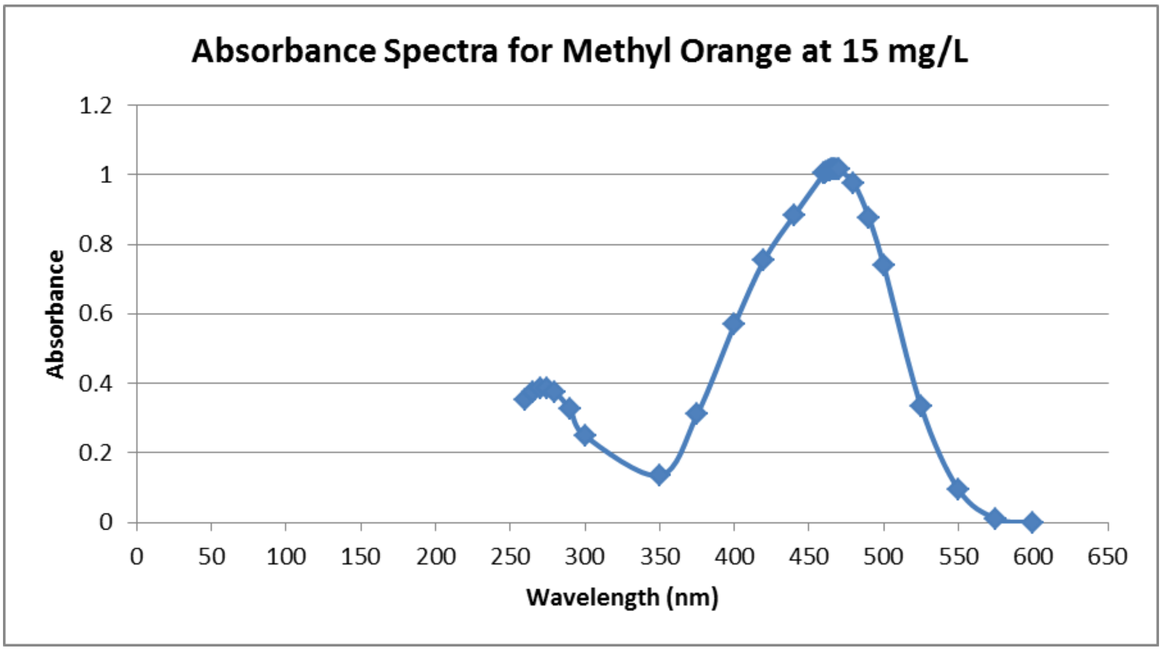


Figure 17: Absorption of Methyl Orange at 15 mg/L

The concrete blocks were submerged in tubs containing solution of 15 mg/L of methyl orange and kept in an outdoor environment under similar conditions.

Figure 18: Concrete Blocks Testing With Methyl Orange

4.1.2. Degradation Results

Table 4: Degradation Results of Methyl Orange (Initial to Day 21)

Specimen Type	Initial Absorbance	Absorbance @ Day7	Absorbance @ Day14	Absorbance @ Day21
Plain	1.061	0.855	0.675	0.459
5% TiO2	1.061	0.825	0.61	0.387
Surface coated	1.061	0.8	0.597	0.36
10% TiO2	1.061	0.798	0.587	0.344

Table 5: Degradation Results of Methyl Orange (Day 28 to Day 42)

Specimen Type	Absorbance @ Day28	Absorbance @ Day35	Absorbance @ Day42
Plain	0.284	0.185	0.134
5% TiO2	0.232	0.158	0.109
Surface coated	0.2	0.141	0.092
10% TiO2	0.193	0.119	0.08

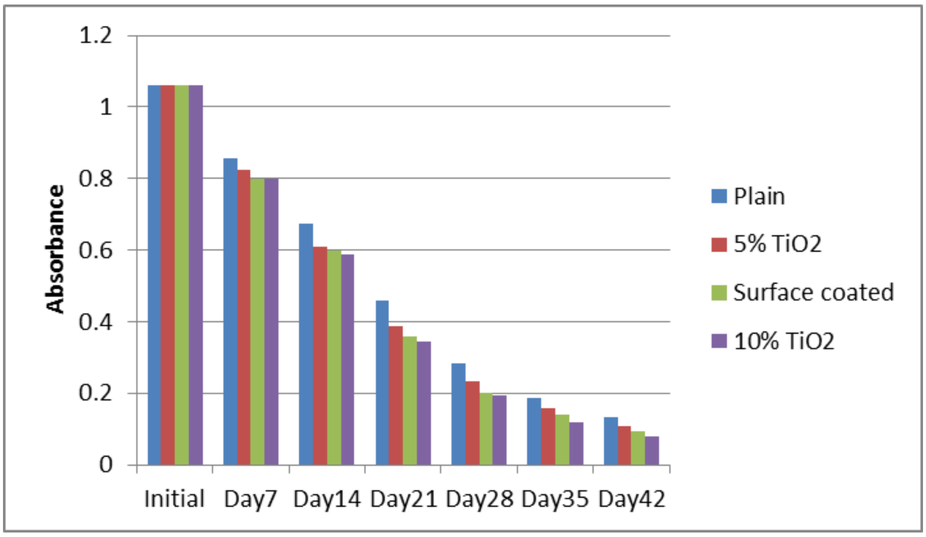


Figure 19: Change in Absorption of Methyl Orange (Upto 42 Days)

These results show that maximum degradation occurred with blocks having 10% TiO_2 nanoparticles substituted for cement. The following graph shows the relative degradation of methyl orange by different specimens standardized with the normal solar radiation degradation of methyl orange.

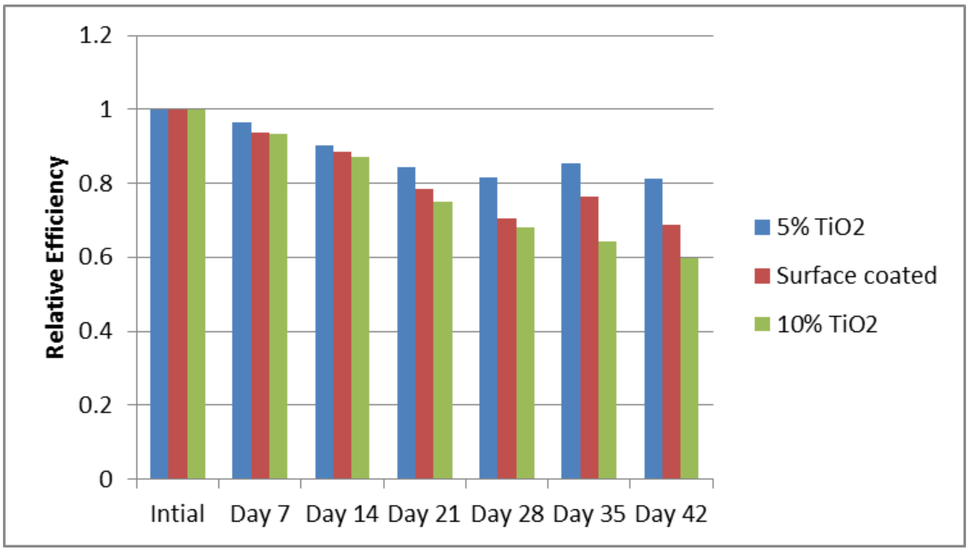


Figure 20: Relative Efficiency of Degradation of Specimens

4.2. Confirmatory Testing

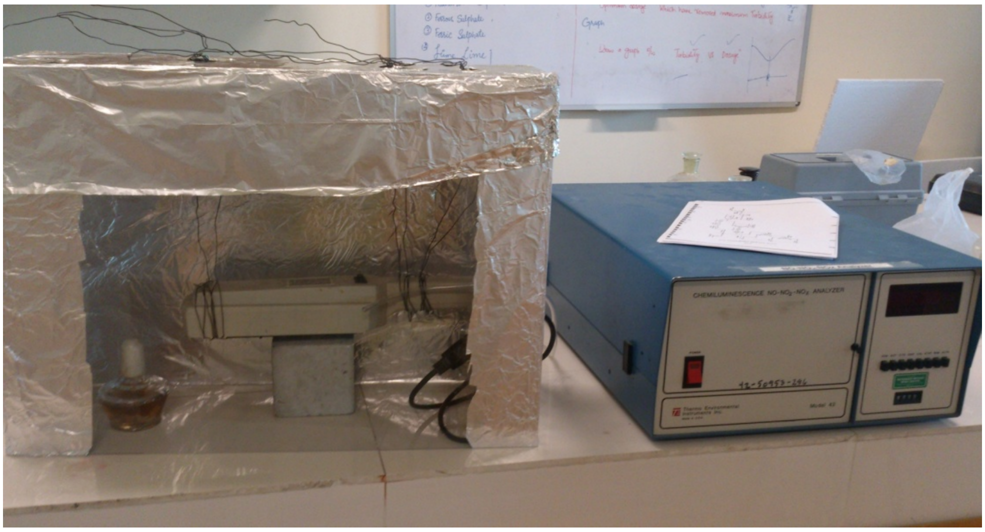


Figure 21: Experimental Setup

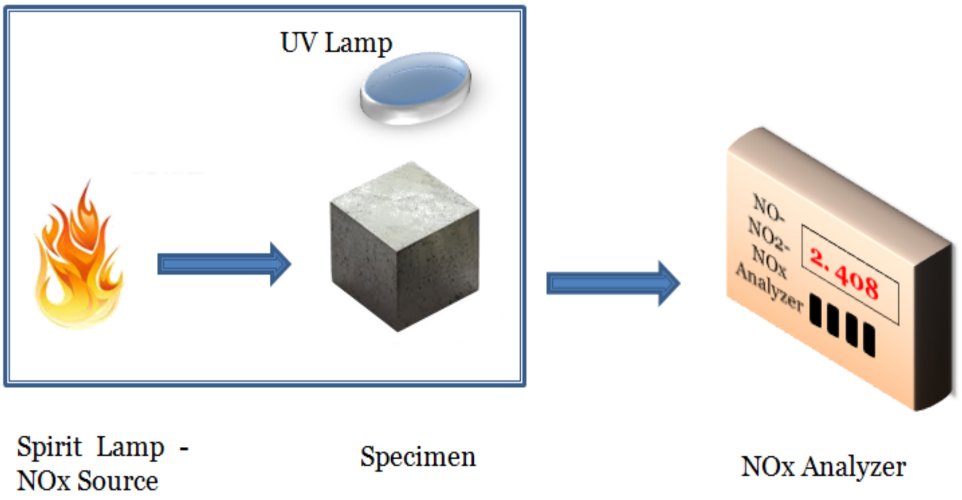


Figure 22: Basic Layout of Experimental Setup

A spirit lamp was used as a NOx source. Spirit lamp, specimen and UV lamp were enclosed in an acrylic box which had a connection to the sampling inlet of NOx analyzer. The experiment was carried out for 140 minutes. The flame was turned on for the first 25 minutes and was then turned off. NO_2 concentration was measured as ppb or µg/L after an interval of 20 minutes.

4.2.1 Results

The results were plotted as a graph as shown below as concentration of NO_2:

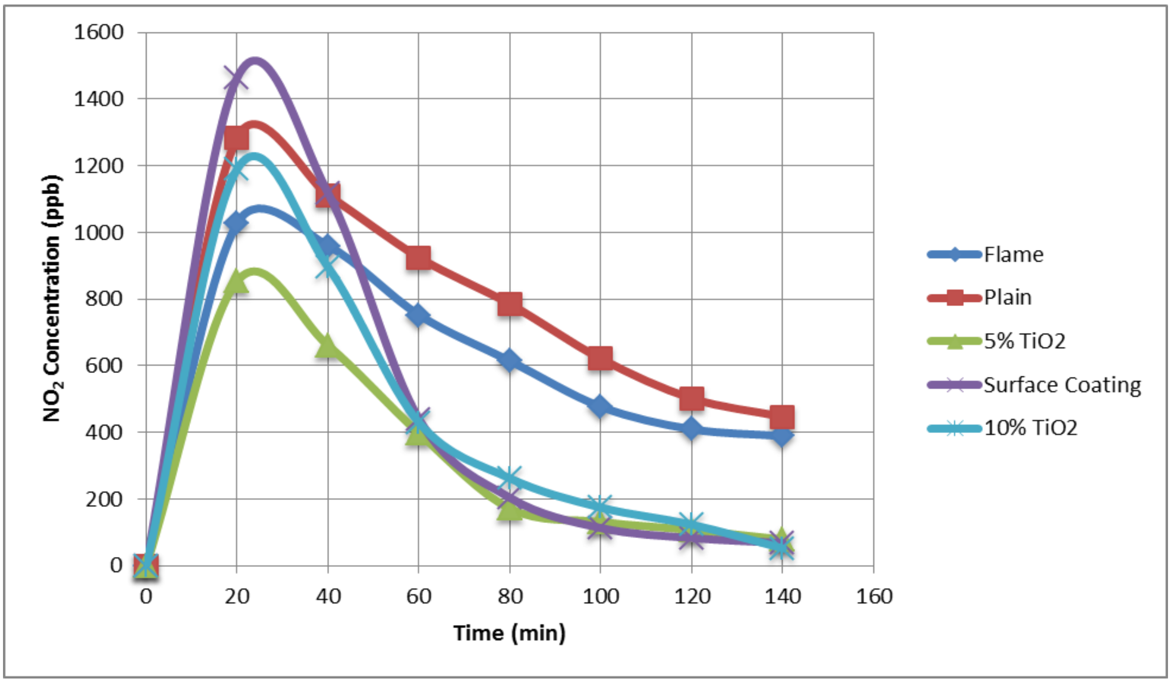


Figure 23: Degradation Pattern of NO_2

Degradation ratio was calculated for each specimen including the flame. Relative degradation was then calculated for the specimens by standardizing with the flame.

Table 6: Degradation Ratio and Relative Degradation of NO_2

Experiment Condition	Degradation Ratio	Relative Degradation (%)
Flame	0.379	-
Plain	0.348	8
5% TiO2	0.092	76
Surface coated	0.047	88
10% TiO2	0.051	87

Maximum degradation of NO_2 occurred for the surface coated specimen followed by the specimen containing 10% TiO_2 i.e. 88% and 87% respectively. 5% TiO2 containing specimen degraded 76% of NO2 while the plain specimen showed degradation of only 8% which was probably due to absorption.

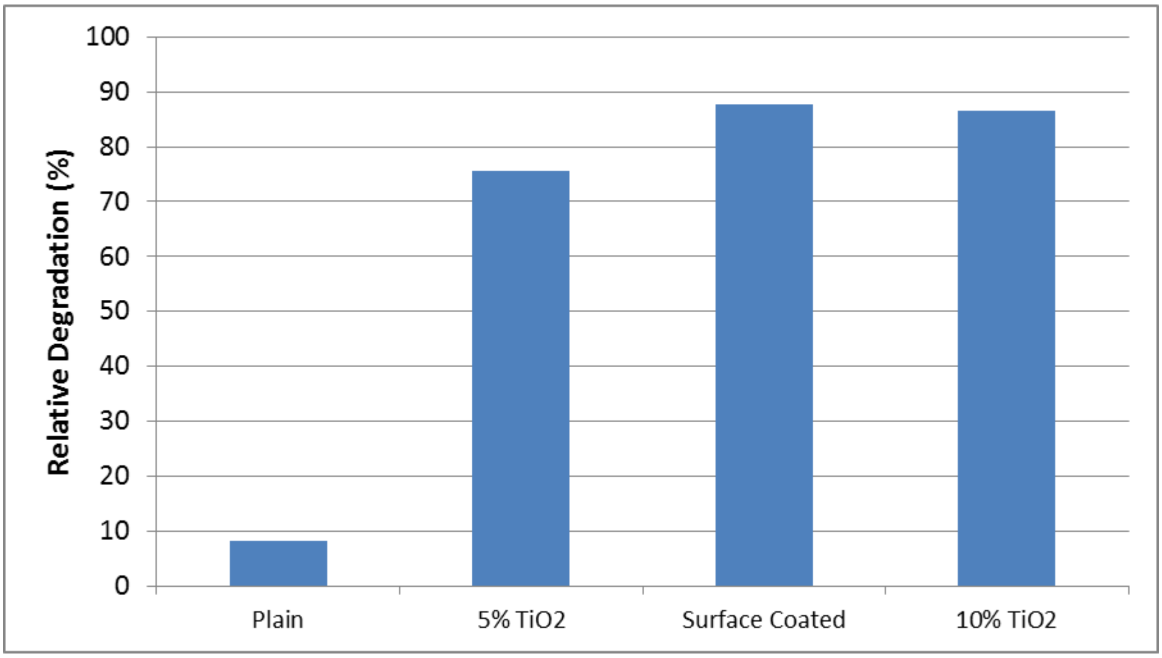


Figure 24: Relative Degradation of NO_2

5. Cost-Benefit Analysis

Annual health cost due to vehicular pollution in Pakistan, 1993 = 147.55 M USD (Qamar uz Zaman)

48.9% of this cost is due to NOx, which makes 72.15 M USD (Qamar uz Zaman)

Health cost due to NOx in 2010 = 166.58 M USD

Percentage of rigid pavements in Pakistan = 0.34% (711 km) (CIA)

Assume that 10% of the vehicles use the rigid pavements.

Health cost due to NOx from vehicles on rigid pavement will be 16.66 M USD/annum.

Over a period of 20 years, which is the lifetime of a rigid pavement, this will add up to a cost of 333.16 M USD.

Following table represents the cost of constructing nano-particles embedded pavements assuming different widths of nano-particles embedded surface:

Table 7: Additional Cost Incurred for Embedding Nano-Particles

Width of Embedded Nano-Particles (in)	Cost (M USD)
2	50.2
4	100.4
6	150.6
8	200.8
10	251
12	301.2

This shows that even if we construct an entire width of pavement with nano-particles embedded concrete we can save a considerable amount of money in terms of the amount to be spent on the health cost due to NOx.

6. Conclusion

As the nano-particles in the mix design increased, strength of the concrete increased slightly. As a result, it can be deduced that TiO_2 nano-particles can be embedded in concrete up to 10% substitution without any significant changes in strength. Also at 10% concentration of nano-particles, these can be effective in removing NO_x from the atmosphere.

The pavements that can be constructed using TiO_2 nano-particles can be cost effective as well.

7. Suggestions for Future Work

Civil engineers add a chemical mixture called 'plasticizer' in concrete to make it stronger. The plasticizers are having organic constituents. Effects of adding TiO_2 along with plasticizer on concrete’s strength and photo-catalytic degradation can be studied.

In addition to this, this research focused on rigid pavements only. Research can be done for flexible pavements too. In this research it can be seen how nano-particles interact with bitumen/coal tar, and what application method will be suitable for such pavements.

Characteristics of run-off and change in infiltration capacity of concrete by addition of TiO_2 can also be studied.

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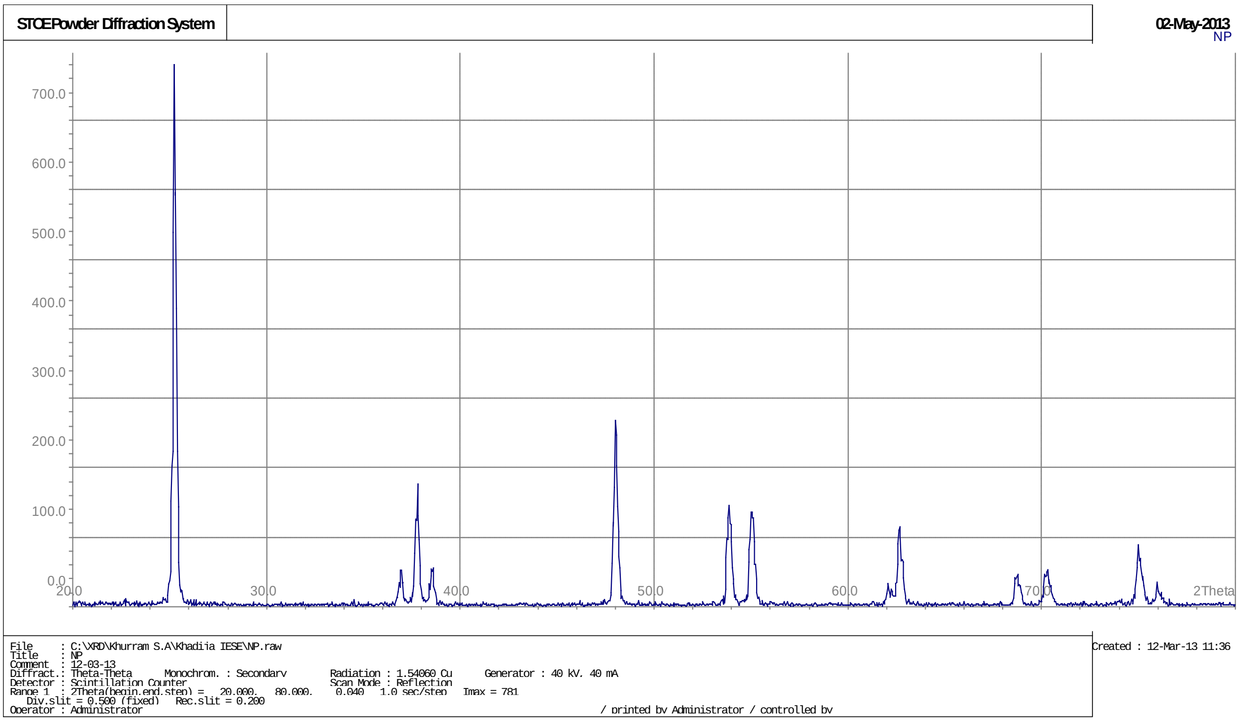
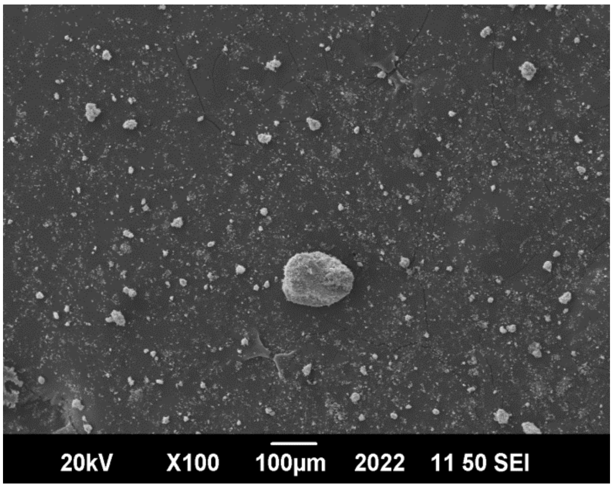
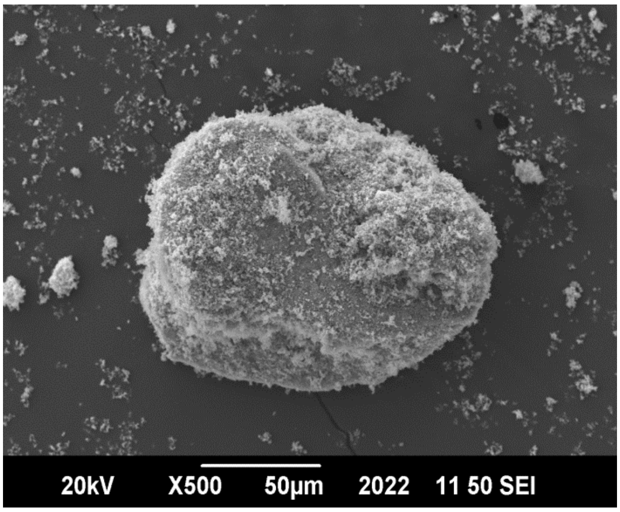
9. Appendix A – X-Ray Diffraction

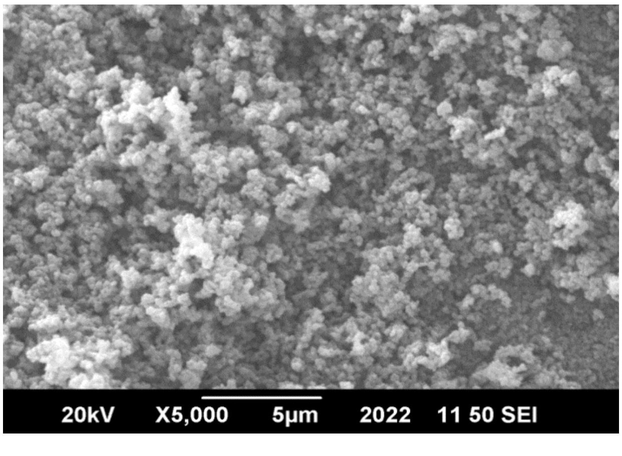


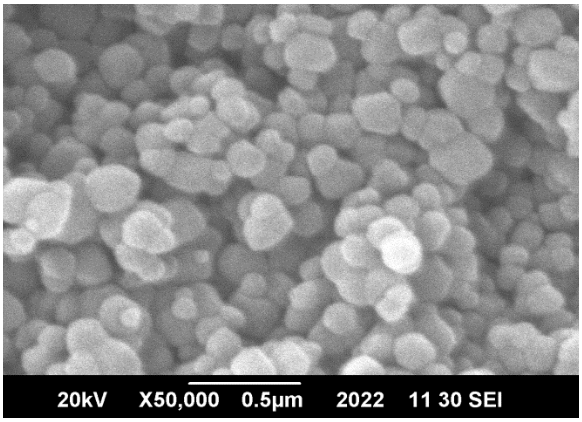
```
File     : C:\XRD\Khurram S.A\Khadija IESE\NP.raw                                                                      Created : 12-Mar-13 11:36
Title    : NP
Comment  : 12-03-13
Diffract.: Theta-Theta      Monochrom. : Secondary        Radiation : 1.54060 Cu      Generator : 40 kV, 40 mA
Detector : Scintillation Counter                          Scan Mode : Reflection
Range 1  : 2Theta(begin,end,step) =   20.000,   80.000,    0.040   1.0 sec/step   Imax = 781
   Div.slit = 0.500 (fixed)   Rec.slit = 0.200
Operator : Administrator                                                              / printed by Administrator / controlled by
```

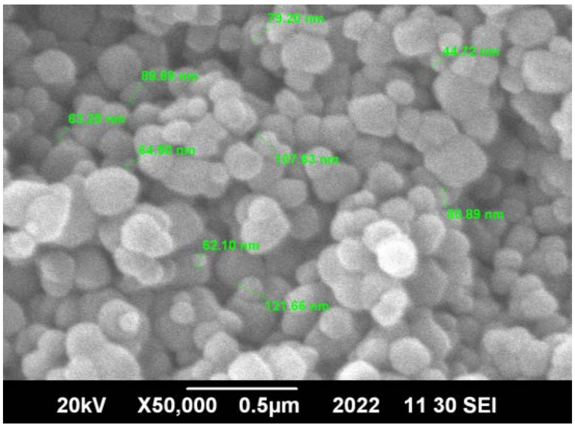
10. Appendix B – SEM Images








20kV
X50,000
0.5µm
2022
11 30 SEI


79.20 nm
44.72 nm
89.89 nm
63.25 nm
64.50 nm
107.63 nm
80.89 nm
62.10 nm
121.66 nm
20kV
X50,000
0.5µm
2022
11 30 SEI