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Methods for the Application of Titanium Dioxide Coatings to Concrete Pavement

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Abstract: Photocatalytic compounds have the potential to remove harmful air pollutants from urban areas. The objective of this study is to evaluate three methods of application for TiO_2—a cement-based thin coating, a water-based titanium dioxide solution (referred to in this paper as PT), and a sprinkling of TiO_2 – to the fresh concrete surface before hardening. Prepared samples were subjected to wear and abrasion by using an accelerated loading test and rotary abrasion. The environmental efficiency of the original and worn samples to remove nitrogen oxides from the atmosphere was measured using a newly developed laboratory setup. Microscopic analysis was conducted using scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) to determine the relative concentration and distribution of TiO_2 particles on the surface before and after wear. Results of the experimental program show that in the original state the coating with 5% TiO_2 and the PT product were the most efficient in removing nitrogen oxide (NO_x) from the air stream. On the other hand, results of the rotary abrasion test indicate that the use of a thin coating would be more susceptible to abrasion than the photocatalysis compounds applied using the sprinkling method or using the PT product. The highest NO removal efficiency in the rotary abrasion state was measured for the coating with 5% TiO_2. Results of SEM and EDS analyses show that the samples treated with the PT product had a more uniform distribution and a higher concentration of TiO_2 than the samples treated with the sprinkling method. This may explain the greater NO removal efficiency observed in the samples treated with the PT product.

Key words: *Application method, Nitrogen oxides, Photocatalyst, Titanium dioxide, Sustainable concrete pavement construction.*

Introduction

The U.S. faces a significant challenge in controlling air pollution resulting from transportation activities and the growing population density. High traffic volumes cause high concentrations of nitrogen oxides (NO_x) and volatile organic compounds (VOCs) to be released into the air, which have been linked with serious health hazards to the public. These pollutants may also travel long distances to produce secondary pollutants such as acid rain or ozone. Although attempts have been made to lower vehicle emission standards, a method is needed to remove air pollutants once they are emitted to the atmosphere. This is particularly important in urban and metropolitan areas, where tall buildings prevent the dispersion of air pollutants originating at the street level from road traffic.

Photocatalytic compounds such as nano-sized titanium dioxide (TiO_2) particles can be used to trap and degrade organic and inorganic particles in the air, removing harmful pollutants such as NO_x and VOCs in the presence of UV light (sunlight). In addition, their super hydrophobic or super hydrophilic properties allow them to self-clean in the presence of rain [1]. Current applications of this technology are used in building facades and gateway elements of bridges not subjected to traffic, as in the case of the I-35W Bridge over the Mississippi River in downtown Minneapolis. Recently, researchers have applied photocatalytic technology to pavements using several application methodologies. However, the proper method of applying titanium dioxide to the concrete surface is still unclear. This critical factor demands evaluation in order to ensure acceptable durability while providing optimum environmental performance of the photocatalytic compound.

The objective of this study is to evaluate three methods of application of titanium dioxide to concrete pavement. To achieve this objective, the durability of the air-purifying layer was evaluated using an accelerated loading test and rotary abrasion. The environmental performance of the coating was measured before and after wear. In addition, the resistance to wear and the presence of the nano-particles on the surface were identified using scanning electron microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) analyses. Results of the experimental program allow comparing these application methods in terms of durability and environmental performance.

Background

The potential of TiO_2 as a photocatalyst was discovered by Fujishima and Honda in 1972 [2]. Titanium dioxide is a semiconductor, which has three crystal arrangements: anatase, rutile, and brookite. Of the three, research has shown that titanium dioxide in the anatase phase exhibits the highest photoactivity in environmental purification [3]. Being a semiconductor, when TiO_2 is exposed to energy from UV irradiation (h_v), oxidizing holes (h^+), and photogenerated electrons (e^-) create hydroxyl radicals and superoxides shown in the following reaction scheme [4]:

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Table 1. Details of the Application Methods.

Application (1)	Sample Name (2)	TiO_2 (g/m^2) (3)	Crystal Type (4)
Control	Control	0	N/A
Concrete Surface	3% TiO_2	132	Anatase
Mixture	5% TiO_2	220	Anatase
TiO_2 Sprinkled on	3% Sp	132	Anatase
Fresh Concrete	5% Sp	220	Anatase
Sprayed	PT	215	Anatase

$$TiO_2 + hv \rightarrow h^+ + e^- \quad , OH^- + h^+ \rightarrow OH^* \quad , O_2 + e^- \rightarrow O_{2^-} \qquad (1)$$

The hydroxyl radicals and superoxides have been proven to play an important role in the photodegradation reactions through oxidation or reduction processes, respectively [4]. The oxidation of nitrogen oxide by means of this photocatalyst process is described as follows [5]:

$$NO + \dot{O}H \xrightarrow{TiO_2} NO_2 + H^* \qquad (2)$$

$$NO_2 + \dot{O}H \xrightarrow{TiO_2} NO_3 + H^* \qquad (3)$$

The resulting water-soluble nitrates are washed away by rainfall. The concentrations of water-soluble nitrates produced as a result of the photocatalytic oxidation were found to reach a level 10 times inferior to the original pollution level [6].

The potential of TiO_2 as an air purifier in urban and metropolitan areas, which suffer from high concentration of air pollutants, have been widely recognized in literature [7]. Being produced in a powder form, TiO_2 is most commonly incorporated into the cement mixture that is applied to concrete pavements as a cement-based overlay. Alternative methods of applying TiO_2 to concrete pavements include spraying TiO_2 nanoparticles suspended in a binding agent or sprinkling TiO_2 nanoparticles to curing concrete as suggested in this study.

Evaluation of concrete pavements treated with TiO_2 provided promising results, as recent research shows that a thin cement surface coating applied to paving blocks is able to remove a significant portion of NO_x and VOCs pollutants from the atmosphere when placed as close as possible to the source of pollution [5]. Assuming the highest removal rates, it was estimated that each square meter of titanium dioxide coating, subject to sunlight, could remove NO_x and VOCs from about 41 m^3 to 200 m^3 and 24 m^3 to 60 m^3 of air per day, respectively [8]. However, the efficiency of this technology depends on many factors, including the size of the surface exposed, the degree of purity of the photocatalytic compound, the concentration of pollutants, air humidity, and ambient temperature. As a result, the photocatalytic efficiencies of NO_x reduction have ranged from 40% to 85% [5]. Porosity of the surface and the amount of TiO_2 surface area are also important, as NO_x removal is improved as the porosity is increased. Photocatalytic activity decreased by approximately 8% with the aging of the surface but stabilized at the age of 90 days [7]. The deposition of pollutants on the surface was reported to decrease the efficiency of removal, but it can be regained through the self-cleaning mechanism [9].

Experimental Program

The objective of the experimental program is to measure and compare the environmental performance and durability of three methods proposed for the application of TiO_2 to concrete pavement. For this purpose, laboratory samples were prepared and subjected to wear and abrasion using an accelerated loading test and rotary abrasion. The environmental efficiency of the original and worn samples to remove nitrogen oxides from the atmosphere was measured using a newly developed laboratory setup. Microscopic analysis was conducted using scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) to determine the relative concentration and distribution of titanium dioxide particles on the surface before and after wear.

Laboratory Samples and Application Methods

The substrate concrete samples were prepared based on a standard concrete mix design that can achieve a compressive strength of 41 MPa according to the Louisiana Standard Specifications for Roads and Bridges. The samples were placed into molds with dimensions of 305 mm × 381 mm × 25.4 mm. The samples were cured by applying a curing compound outside seven days before de-molding. Three replicates were prepared for each testing condition. Three methods were simulated for the application of titanium dioxide to the concrete substrate. The experimental test matrix is presented in Table 1.

The first method consists of applying a cement-based 10 mm thin coating to the concrete surface. A surface mixture consisting of ultrafine titanium dioxide, cement, filler (sand with a maximum nominal size of 1.18 mm), and water was prepared. The sand was sieved to remove all grains with a particle size of 300 μm or smaller. This procedure is based on past research that show that a coating with less fine particles result in higher porosity and, therefore, improve NO_x removal efficiency [7]. The surface mixture was prepared at a water to cement ratio of 0.6 and was applied to the concrete surface as a 10 mm thick coating. A commercially available, ultrafine anatase form of 95% pure titanium dioxide with an active surface area of approximately 90 m^2/g was used at a content of 3% and 5% by weight of cement. The mix constituents (i.e. titanium dioxide, cement, filler, and water) were used with a ratio of 0.03 or 0.05 Titanium dioxide: 1 cement: 3.8 filler: 0.6 water. To apply this mixture, the concrete samples were flipped to eliminate any interference due to the curing compound. Prior to application, the surface was roughened to strengthen the bond adhesion. The photocatalytic surface coating was cured and covered at room temperature for 48 hours.

The second method consists of spraying a water-based TiO_2 surface treatment referred to in this paper as PT. The concrete samples were flipped to eliminate interference with the curing compound. Prior to application, the surface was roughened to strengthen the bond adhesion. This treatment method is applied to the hardened concrete surface in two parts. The base coat is first applied as a primer to provide a clean and durable surface. The base coat has 2% by weight of anatase titanium dioxide suspended particles. However, this was not of a photocatalytic grade. The top

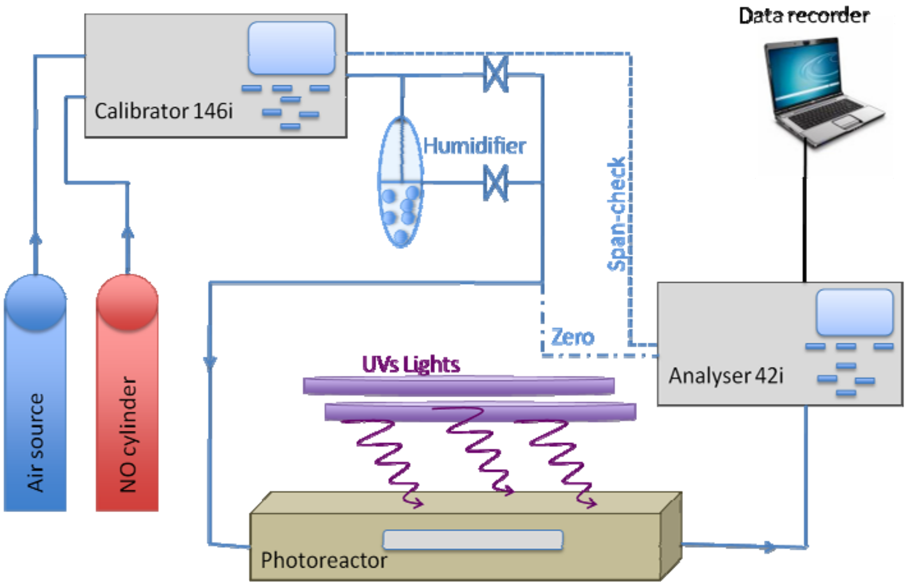


Fig. 1. Experimental Setup Flow Diagram.

coat also has 2% titanium dioxide suspended particles. However, a nano-size (about 6 nm) photocatalytic grade was used to initiate the purification reaction. Both the primer and top coat were applied by a spray gun using a cross hatch spray formation to ensure even coverage at a rate of 215 g/m^2.

The third method consists of sprinkling nano-sized titanium dioxide particles to the fresh concrete surface before hardening. Cristal Millennium PC105 was used for the titanium dioxide nanoparticles. Particles were spread on the surface of the concrete substrate at a content of 3% and 5% directly after pouring and curing.

Environmental Test Setup

An experimental setup was built to quantify the environmental efficiency of TiO_2 in removing harmful pollutants from the air, as shown in Fig. 1. The experimental setup was modified from the Japanese Industrial Standard (JIS TR Z 0018 "Photocatalytic materials – NO_x air purification test procedure") to accommodate the larger samples required for durability testing [10]. In scaling the size, the length was kept at 3 times the width to promote laminar flow as recommended by JIS. The setup simulates different environmental conditions by allowing control of light intensity and air humidity. The pollutants are introduced through an inlet jet stream to the photocatalytic testing device. A zero air generator is used to supply the air stream, which is passed through a humidifier to simulate the desired humidity level. The photocatalytic testing device creates an enclosed, controlled environment where the light and atmosphere can be simulated. Fluorescent lamps, attached to the photocatalytic device, are used to imitate natural sunlight radiation required for photocatalytic activity.

The pollutants measured from the recovered air before and after the photocatalytic device allowed for a determination of the absorbed level of pollutants. Following the JIS test procedures developed for photocatalytic materials, the efficiency of nitrogen-oxide removal was measured using the Thermo chemiluminescent NO_x analyzer [10]. The Thermo 146i Gas calibrator was used to supply a defined concentration of gas for the experimental setup at a controlled flow rate. Results presented in this paper were obtained at room temperature (23°C) and at a relative humidity of 50%. Nitrogen oxide (NO) was blown over the surface at a concentration of 410 ppb and at a flow rate of 9 L/min. The effects of the flow rate, humidity level, and pollutant concentration have been presented elsewhere [11]. Testing was conducted for a total of five and half hours. However, the photocatalytic process only started after 30 minutes from the beginning of the test to ensure that a steady concentration was reached in the environmental chamber.

Laboratory-Simulated Abrasion and Wear

Abrasion and wear resistance properties of the titanium dioxide surface layer were measured using an accelerated loading test and rotary abrasion. The Hamburg-type Loaded Wheel Tester (LWT), which employs a scaled dynamic steel wheel passing back and forth over the specimen, was used in this study to simulate loading and wear-resistance of the applied coating. The wheel applied a load of 702 N at a frequency of 56 passes per minute. Testing was conducted at room temperature under dry conditions, while the progress of surface rutting was continuously monitored. After 20,000 cycles, the test was stopped and samples were obtained to examine the surface using SEM and EDS and to measure the environmental efficiency of the worn samples. Rotary Abrasion (RA) was conducted using an in-house built device that conforms to ASTM C 944 and is conducted using a Rockwell freestanding drill press. This test method uses a cutter rotating at 200 rpm under a constant load of 98 N for 2 minutes to wear the coating surface. The abrasion wear is determined by measuring the loss of weight in grams. Since durability testing for TiO_2 photocatalytic cement mixes is limited, samples were compared to a concrete base slab with no TiO_2.

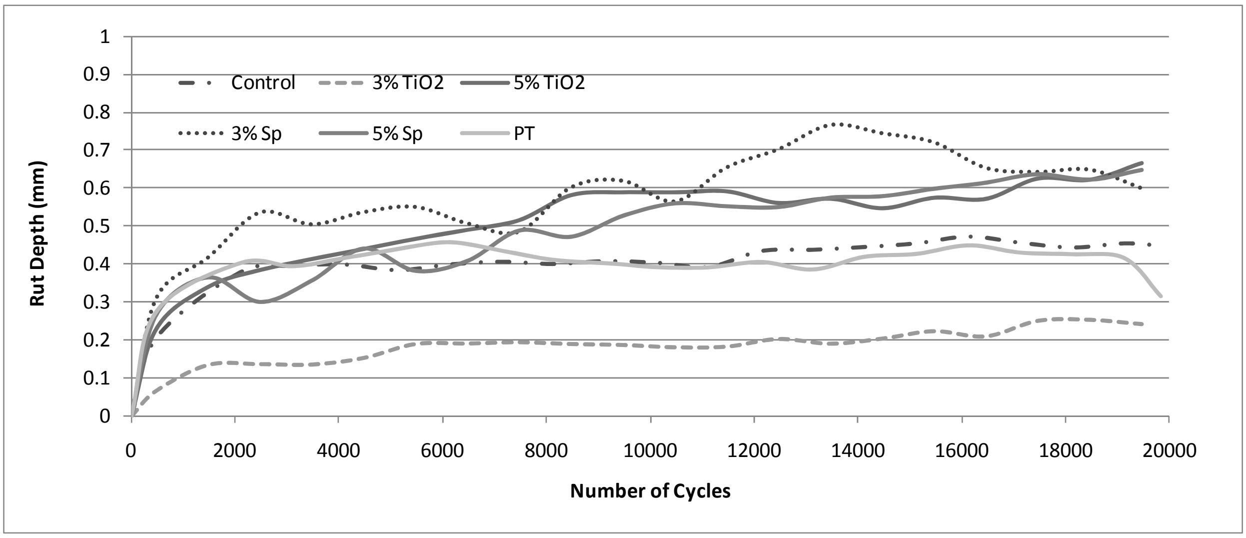


Fig. 2. Measured Rut Depth in the Loaded Wheel Tester.

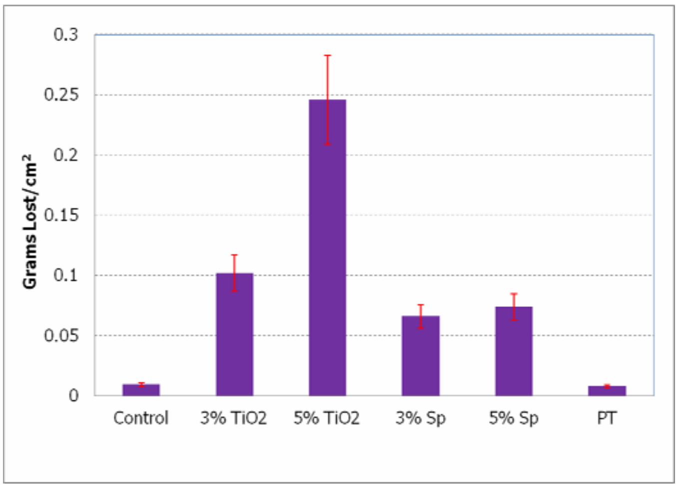


Fig. 3. Measured Abrasion Resistance in the Rotary Abrasion Test.

Scanning Electron Microscopy and Energy Dispersive Spectroscopy

SEM and EDS were used in this study to investigate the distribution of TiO_2 in the coating surface before and after wear. Sample preparation consisted of cutting a 25 mm × 25 mm specimen from the surface coating before and after abrasion testing. The samples were coated with a thin layer of carbon conducting film by evaporation. Microscopic analysis was conducted using a JEOL JSM-840A Scanning Electron Microscope at an acceleration voltage of 15 kV. The images were stored as 1,290 × 968 TIFF files. The existence and distribution of TiO_2 were determined using NIST/NIH Desktop Spectrum Analyzer (DSTA) software. The SEM images and the corresponding elemental maps were captured using the NIH imaging software to observe the microstructure and TiO_2 distribution in the coating surface.

Results and Analysis

Loaded-Wheel Tester (LWT) and Abrasion Test Results

Fig. 2 presents the average measured rut depth and the corresponding number of wheel cycles for the two replicates of each of the six specimen types (control with no TiO_2, coating with 3% TiO_2 [3% TiO_2], coating with 5% TiO_2 [5% TiO_2], sprinkled at 3% TiO_2 [3% Sp], sprinkled at 5% TiO_2 [5% Sp], and PT). As shown in Fig. 2, the measured rut depth for all specimens was minimal (less than 1 mm), indicating that the use of the coating did not appear to affect the wear resistance of the surface. However, this test is usually used for asphalt mixes, and one did not expect to observe major rutting for concrete samples.

Fig. 3 illustrates the average loss of weight observed in the RA test for three replicates of each of the six specimen types. All results on similar samples differed less than 50% from the average. Results of the RA test appear to indicate that the use of a thin coating can be more susceptible to abrasion than the photocatalytic compound applied using the sprinkling method or the PT product. A greater loss of weight was noted for the coated samples than for the other types of specimens, including the control specimen. Loss of particles in the RA test may be associated with a loss of mortar, fines, and TiO_2 nanoparticles. These results were investigated using SEM and EDS.

Environmental Test Results

Fig. 4 illustrates the variation of NO concentration during the course of the environmental experiment for the sample treated with PT. The inlet concentration is 410 ppb. The UV light is turned on 30 minutes after the start of the experiment. This results in a fast drop of NO concentration in the outlet air stream. After the initial drop, the NO concentration remained mostly constant throughout the experiment. After 5 hours of UV light exposure, the light is turned off, and the NO concentration is measured. For the test condition shown in Fig. 4, the use of TiO_2 photocatalyst coating had an NO removal efficiency of 25%. The NO removal efficiency depends on many factors, including flow rate, air humidity, application method, ambient temperature, and content of TiO_2. Table 2 presents the measured NO efficiency for the different specimen types in the original state. As shown in Table 2, the coating with 5% TiO_2 and

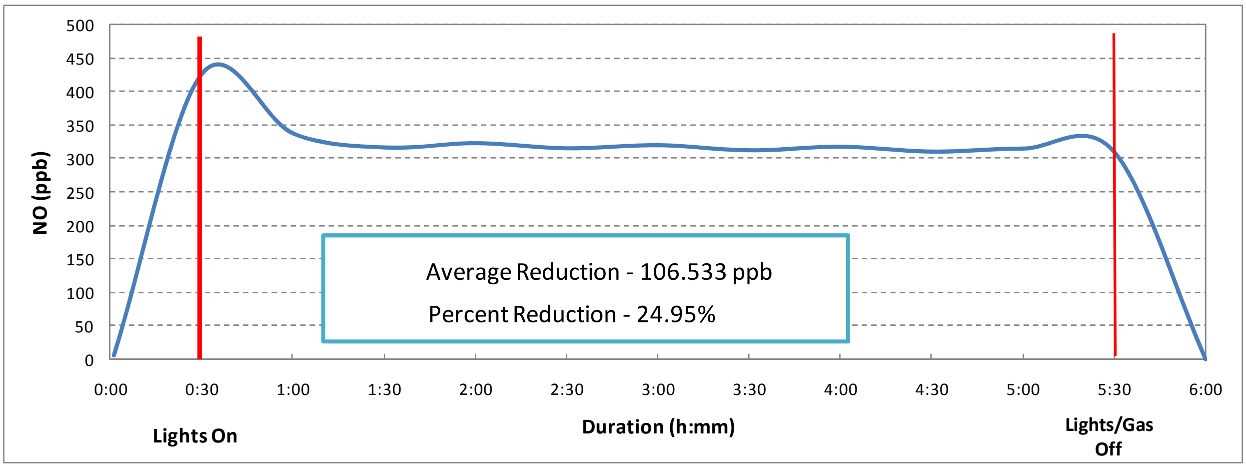


Fig. 4. Variation of NO Concentration During the Environmental Experiment.

Table 2. NO Removal Efficiency for Original Samples.

Sample (1)	Naming Convention (2)	Humidity (%) (3)	Flow Rate (L/min) (4)	NO Removal (%) (5)
Control	Control	50	9.0	2.4
3% TiO_2	3% TiO_2	50	9.0	18.0
5% TiO_2	5% TiO_2	50	9.0	26.9
3% TiO_2 Sprinkled	3% Sp	50	9.0	16.9
5% TiO_2 Sprinkled	5% Sp	50	9.0	18.9
Sprayed PT	PT	50	9.0	25.0

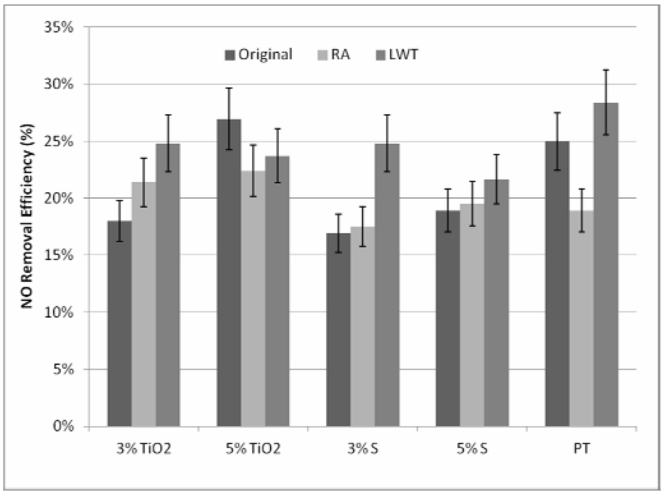


Fig. 5. NO Removal Efficiencies for Original, Worn, and Abraded Samples.

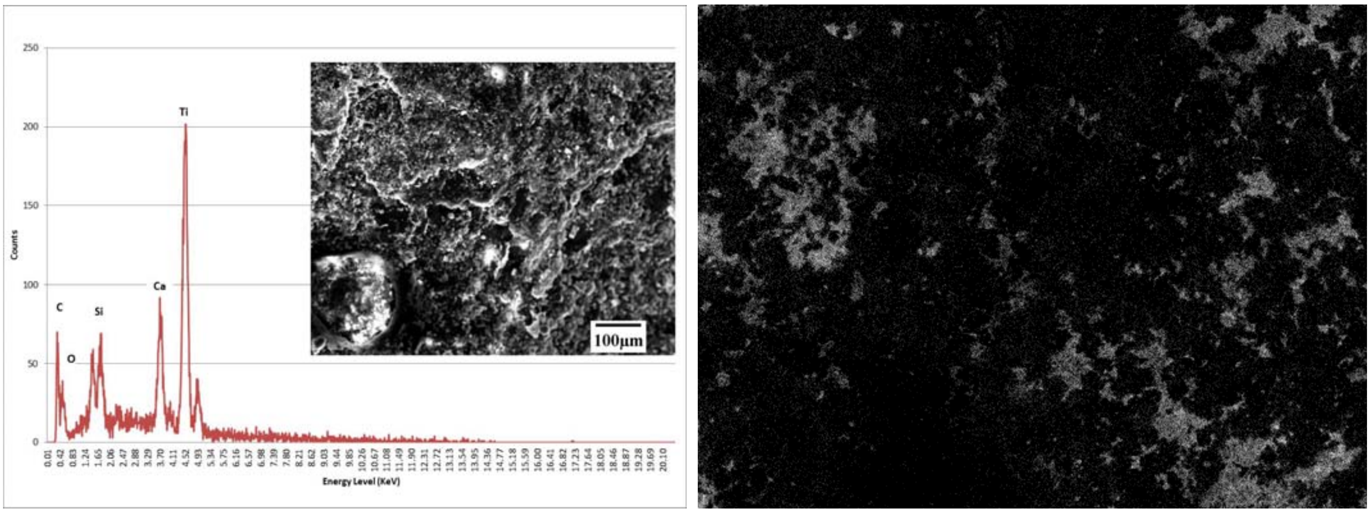
the PT product were the most efficient in removing nitrogen oxide from the air stream. However, the curing compound may have impacted the environmental effectiveness of the sprinkling technique.

Effects of Wear and Abrasion on NO Removal Efficiency

Fig. 5 presents the average NO removal efficiencies for the original, worn, and abraded samples (loaded-wheel test and rotary abrasion samples). As shown in Fig. 5, it appears that the LWT slightly improved the NO removal efficiency of the different samples with the exception of samples with 5% TiO_2, which experienced a slight decrease in efficiency. This result may be due to the wear action simulated using the LWT, which exposed part of the embedded titanium dioxide particles at the surface and therefore improved the NO removal efficiency. In contrast, rotary abrasion appears to result in a decrease in NO removal efficiency for the 5% TiO_2 coating and the PT product while the efficiency slightly improved or remained constant for the other specimen types. In general, the samples coated with 5% TiO_2 and the PT product were the most efficient in removing nitrogen oxide from the air stream. The highest NO removal efficiencies in the original and RA states were measured for the coating with 5% TiO_2. On the other hand, the highest NO removal efficiency in the LWT state was measured for the samples treated with the PT product.

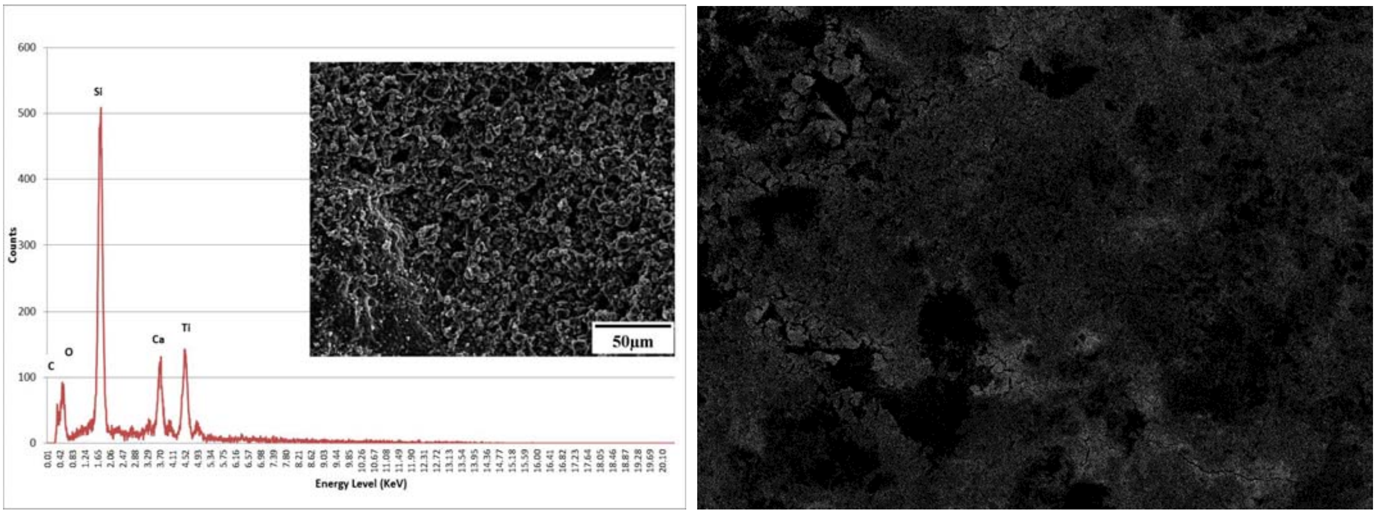
SEM and EDS Test Results

Prepared samples were magnified using SEM to reveal different microscopic features in the surface of the specimens. In this process, different locations in the sample were captured at a low magnification rate and were then repeatedly enlarged to higher magnification rates. Features were selected and zoomed in at high magnification to determine their elemental composition. Figs. 6, 7, and 8 present microscopic images of the sprinkled surface at 5% TiO_2 content, the surface treated with the PT product, and the cement coating with 5% TiO_2, all in the original state. Along with the SEM image, results of the EDS spectrum analysis and elemental mapping are presented. Results of the EDS spectrum analysis provide an elemental analysis of the sample in which the intensity of the peaks can infer the relative concentration of each element. Therefore, the relative concentrations of titanium particles on the surface before and after wear were assessed. From the SEM image coupled with an elemental map of titanium, one may notice the



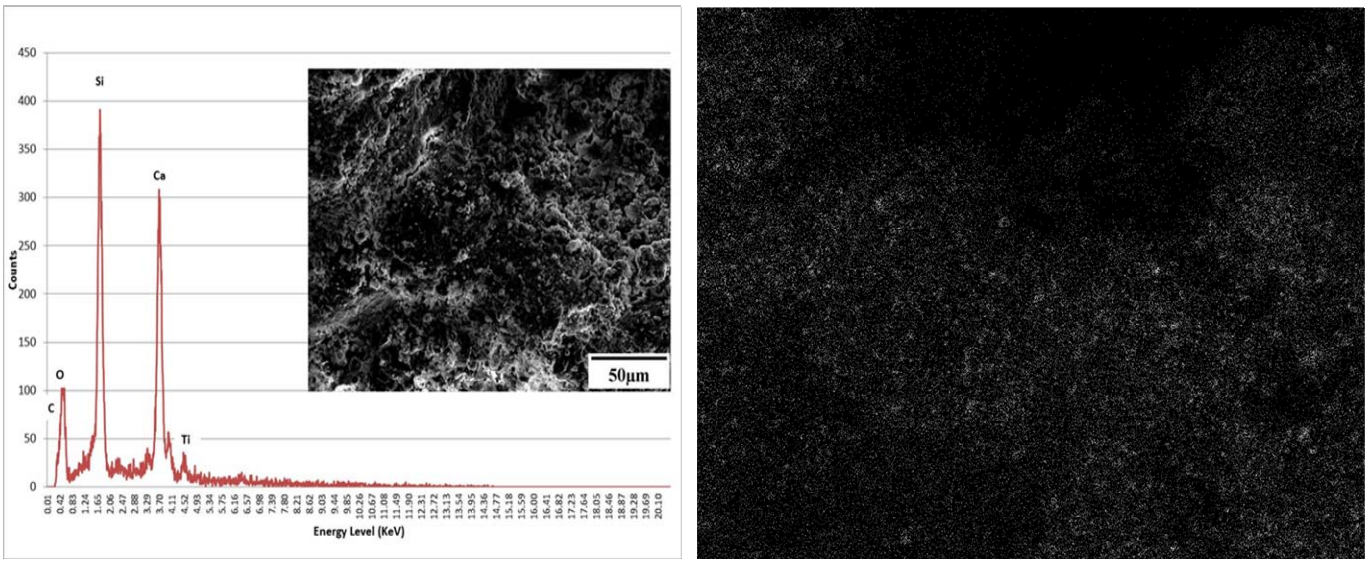
(a) SEM and EDS Test Results (b) Titanium Elemental Mapping

Fig. 6. SEM and EDS Test Results for the Original Sample Sprinkled with 5% TiO_2.



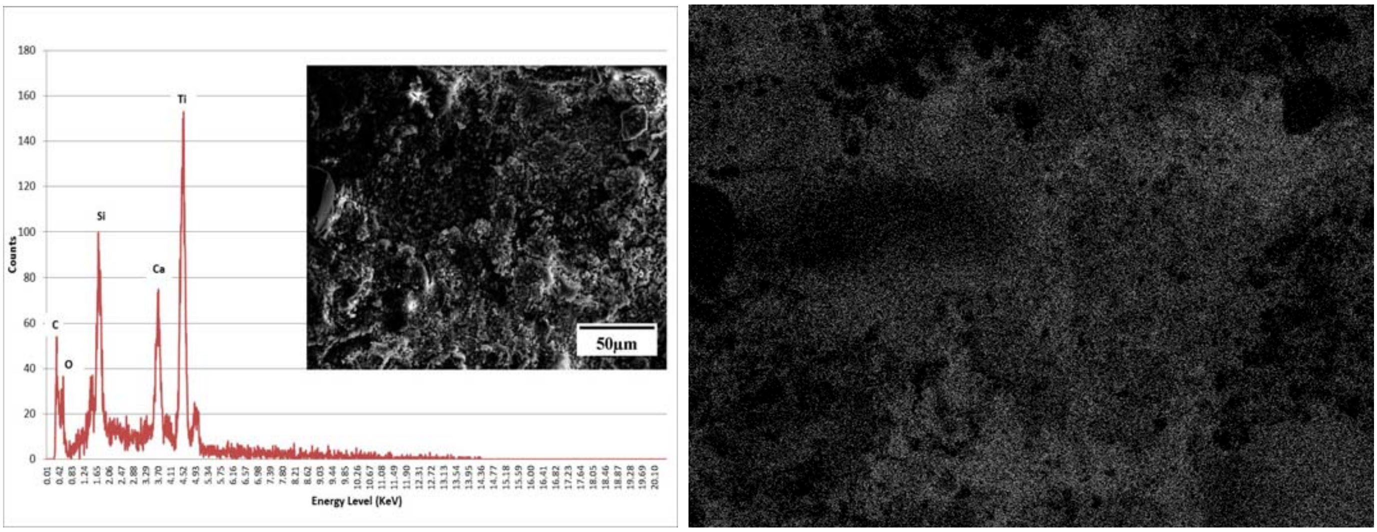
(a) SEM and EDS Test Results (b) Titanium Elemental Mapping

Fig. 7. SEM and EDS Test Results for the Original Sample Treated with PT.



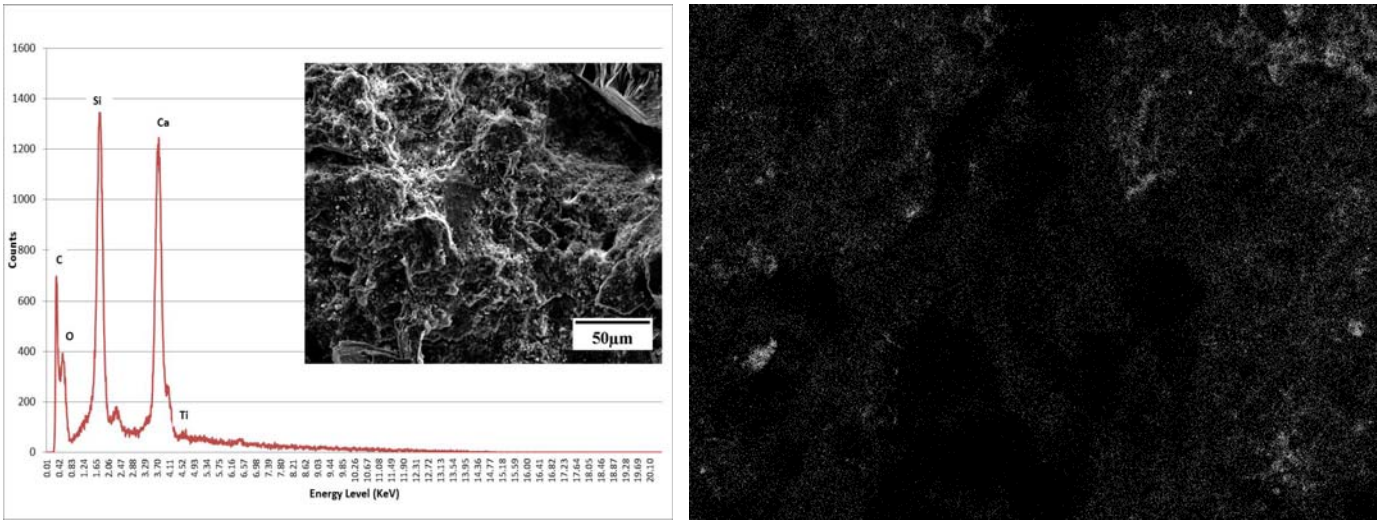
(a) SEM and EDS Test Results (b) Titanium Elemental Mapping

Fig. 8. SEM and EDS Test Results for the Original Sample with the Cement Coating with 5% TiO_2.



(a) SEM and EDS Test Results (b) Titanium Elemental Mapping

Fig. 9. SEM and EDS Test Results for the Abraded Sample Sprinkled with 5% TiO_2.



(a) SEM and EDS Test Results (b) Titanium Elemental Mapping

Fig. 10. SEM and EDS Test Results for the Abraded Sample Treated with PT.

uniform distribution of titanium dioxide particles across the section of the surface treated with the PT product, as shown in Fig. 7. In contrast, the sample with the sprinkled TiO_2 particles demonstrates less uniformity with regions that show agglomerates of titanium dioxide on the surface. Uniform distribution is a desirable characteristic to ensure maximum UV exposure of the nano-particles on the surface, which would provide maximum NO removal efficiency. The content of TiO_2 observed on the surface of the sample treated with PT and on the sprinkled surface was significantly greater than on the sample with the cement coating of 5% TiO_2. This may explain the greater NO removal efficiency observed in the samples treated with PT product, as seen in Fig. 5.

Figs. 9 and 10 present the SEM image and the corresponding results of the EDS analysis for the sprinkled surface at 5% TiO_2 content and for the surface treated with the PT product in the abraded state. As shown in Fig. 9, the concentration of Ti on the sprinkled sample did not substantially change as compared to the original sample presented in Fig. 6. However, one may notice from the SEM elemental map of Ti (shown in Fig. 9) that the surface shows a reduction of agglomerates of TiO_2 particles, which is possibly due to abrasion. As for the PT sample, the relative concentration of Ti compared to the cement components was significantly lower than the original state. The corresponding element map even exhibits areas void of Ti. As a result, these are inactive locations, which may explain the reduction observed in NO removal efficiency for the abraded PT samples. The low initial photocatalytic efficiency shown in Fig. 5 for the sprinkled samples is likely to be caused by the interference with the organic curing compound, which may have inhibited the TiO_2 degradation of NO_x. After abrasion, some of the curing compound was abraded off exposing more TiO_2 particles and decreasing the curing compounds negative impact, thus leading to a slight increase in efficiency.

Summary

As indicated by the results of this study, the coating with 5% TiO_2

and the PT product were the most efficient in removing nitrogen oxide from the air stream. The highest NO removal efficiencies in the original and RA states were measured for the coating with 5% TiO_2. On the other hand, the highest NO removal efficiency in the LWT state was measured for the samples treated with the PT product. Results of the SEM and EDS test results indicate that the PT product provides the most evenly concentrated coverage. Although the sprinkling method shows the greatest surface exposure to TiO_2, evidenced by the higher counts in the EDS analysis, the sprinkled coverage was not evenly distributed, as seen in the elemental maps, and was negatively impacted by the curing compound. In fact, the patches of agglomerates are visible in the Ti mapping, as seen in Figs. 6 and 9.

Cost of the Photocatalytic Coating

In a competitive industry like the construction industry, it is important to quantify the economic performance of the proposed titanium dioxide coating. Therefore, ultrafine, nano titanium dioxide producers were contacted. Contacts with the producers showed that the average market price of this technology is $20/kg. Based on the selected surface mix design, this translates to an added cost of $2.20 per m^2 of surface mix. Assuming a concrete cost of $51 per m^2 for a layer thickness of 254 mm, the use of titanium dioxide coating will result in an added cost of approximately 4% per m^2 of installed concrete. As interest in this technology increases, especially in the neighborhood of large metropolitan areas, it is expected that the unit cost of titanium dioxide coating will significantly decrease as this technology has the potential of achieving economy of scale.

Conclusions and Recommendations

The use of titanium dioxide coating for pavements to improve air quality near large metropolitan areas has received considerable attention in recent years. However, the proper method of applying titanium dioxide to the concrete surface is still unclear. In this regard, the objective of this study is to evaluate three methods of application of titanium dioxide to concrete pavement. The first method consists of applying a thin coating to the concrete surface at a titanium dioxide content of 3% and 5%. The second method consists of applying a water-based TiO_2 surface treatment (PT) to the hardened and cured concrete surface. The third method consists of sprinkling nano-sized titanium dioxide particles to the fresh concrete surface before hardening at a titanium dioxide content of 3% and 5%. Based on the analysis conducted in this study, the following conclusions may be drawn:

- In the original state, the coating with 5% TiO_2 and the PT product were the most efficient in removing nitrogen oxide from the air stream.
- The measured rut depth in the LWT state for the three specimen types was minimal (less than 1 mm). The highest NO removal efficiency in the LWT state was measured for the samples treated with the PT product.
- Results of the rotary abrasion test indicate that the use of a thin coating would be more susceptible to abrasion than the photocatalytic compounds applied using the sprinkling method or using the PT product. Rotary abrasion seems to result in a decrease in NO removal efficiency for the 5% TiO_2 coating and the PT product. The highest NO removal efficiency in the RA state was measured for the coating with 5% TiO_2.
- Results of SEM and EDS analysis show that the sample treated with the PT product had a more uniform and concentrated distribution than the samples treated with the sprinkling method. This may explain the greater NO removal efficiency observed in the samples treated with PT.

Based on these results, one concludes that photocatalytic compounds can be successfully applied to concrete pavements using a surface coating or the spraying method with adequate durability and environmental efficiency. This study represents the first step towards evaluating and supporting the implementation of titanium dioxide coating to concrete pavements as a feasible solution in improving air quality near large metropolitan areas. Based on the results presented in this study, further research is recommended to evaluate the effects of the coating on the pavement surface microtexture and the impacts of oil, debris, and deicing salts on the effectiveness of the photocatalytic process. Research is also needed to quantify the effects of byproducts resulting from purification and the long-term effectiveness of the photocatalytic process.

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