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ABSTRACT

Photocatalytic compounds have the potential of removing harmful air pollutants from urban areas. One proposed method to expand the reach of this technology is to apply titanium dioxide to concrete pavement surface to result in air-purifying concrete pavement. However, the proper method of applying titanium dioxide to the concrete surface is still unclear. To this end, the objective of this study was to evaluate three methods of application for titanium dioxide to concrete pavement. Prepared samples were subjected to weathering and abrasion by using an accelerated loading test and rotary abrasion. The environmental efficiency of the original and weathered 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 weathering. Results of the experimental program showed that 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. On the other hand, results of the rotary abrasion test indicated 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_x removal efficiency in the rotary abrasion state was measured for the coating with 5% TiO_2. Results of SEM and EDS analysis showed 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 PT product.

Keywords: Titanium dioxide, application method, sustainable concrete pavement construction, photocatalyst, nitrogen oxides.

INTRODUCTION

The United States faces a significant challenge in controlling air pollution resulting from transportation activities and the growing population density. A number of regions in the US have been designated by the Environmental Protection Agency (EPA) as nonattainment areas, in which air pollution levels persistently exceed national air quality standards. Although attempts are made to lower vehicle emission standards, a method is needed to remove these 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 absorb organic and inorganic particles in the air removing harmful pollutants such as nitrogen oxides (NOx) and volatile organic compounds (VOC) in the presence of UV light (sunlight). This process, which is similar to plant photosynthesis, allows the decomposition of nitrogen oxide into water-soluble nitrate and sulfur dioxide (SO_2) into water-soluble sulfate in the presence of light by means of a TiO_2-anode. In addition, their super hydrophilic properties allow them to self-clean in the presence of rain given their affinity to water (*1*).

In spite of the rapid development of photocatalytic compounds, current applications of this technology are limited to 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. One proposed method to expand the reach of this technology is to apply titanium dioxide to concrete pavement surface to result in air-purifying concrete pavement. 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 for optimum environmental performance of the photocatalytic compound.

To this end, the objective of this study was to evaluate three methods of application for titanium dioxide to concrete pavement. To achieve this objective, 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 weathering. In addition, the resistance to wear and the presence of the nano-particles on the surface was identified using scanning electron microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) analysis. Results of this experimental program allowed us to compare these application methods in terms of durability and environmental performance.

BACKGROUND

Photocatalytic compounds such as nano-sized particles of titanium dioxide are able to photogenerate electron-hole pairs at their surfaces (*2*). These holes have strong oxidizing power and can remove organic and inorganic toxic materials in the presence of UV light (sunlight). Based on this heterogeneous photocatalytic oxidation process, nitrogen oxides are oxidized into water-soluble nitrates while sulfur dioxide is oxidized into water-soluble sulfates; these substances can be washed away by rainfall. While VOC's are eventually completely oxidized to carbon dioxide, one concern raised about photocatalytic compounds is the production of potentially harmful intermediate products during the decomposition process. Benoit-Marquie et al. reported that butanal and 1-butanoic acid were the intermediates of 1-butanol (*3*). Jacoby et al. identified phenol, hydroquinone and/or benzoquinone, and malonic acid as possible intermediates for the photocatalytic oxidation of benzene (*4*). In outdoor applications, it is believed that the concentration of these intermediates products will not reach a toxic level and similar substances occur naturally in the atmosphere. Nevertheless, this highlights the importance of quantifying the intermediate products formed in the photocatalytic oxidation process.

Titanium dioxide particles crystallize in three forms: anatase, rutile, and brookite. Anatase is a meta-stable phase that transforms into rutile at high temperatures *(5)*. Research has shown that titanium dioxide in the anatase phase shows the highest photoactivity in environmental purification *(6)*. However, the most abundant form is rutile and is, therefore, the predominant type used in industrial applications. This type of titanium dioxide particles is low-cost and can remove a wide range of organic contaminants *(7)*. In pavement applications, it is desirable to prepare a titanium dioxide coating with hydrophobic properties, which provide for a self-cleaning surface. Through the hydrophobic process, particles of contaminants adhere to water droplets in case of rain. These contaminants are then removed from the surface when the droplets roll of it.

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, 8*). Being produced in a powder form, a number of research studies have suggested to use it in a thin film form and to apply it as a coating or slurry to various types of substrates (*9*). Titanium dioxide has also been evaluated and patented as a coating to concrete paving materials, an environmentally-friendly cement (TioCem), an architectural concrete (white cement), a facade to buildings, and as concrete tiles (*10, 11, 12*). One study suggested that the use of titanium dioxide in combination with a cementitious material improves SO_2 removal efficiency through action of the alkaline substratum (*13*).

Evaluation of concrete pavements treated with titanium dioxide provided promising results as recent research shows that a thin surface coating is able to remove a significant portion of NO_x, SO_2, and VOC pollutants from the atmosphere when placed as close as possible to the source of pollution (*14*). It was reported that each square meter of titanium dioxide coating, subject to sunlight, could remove nitrogen oxides and VOC's from about $200m^3$ and $60m^3$ of air per day, respectively (*8*). The efficiency of this technology depends on many factors including the size of the surface exposed, the concentration of pollutants, the humidity, and the ambient temperature. Porosity of the surface is also important as the NO_x removal ability is improved as the porosity is increased. Photocatalytic activity decreased by approximately 8% with aging of the surface but stabilized at the age of 90 days (*9*). The deposition of pollutants on the surface was reported to decrease efficiency of removal but it can be regained through the self-cleaning mechanism (*15*).

EXPERIMENTAL PROGRAM

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

Laboratory Samples and Application Methods

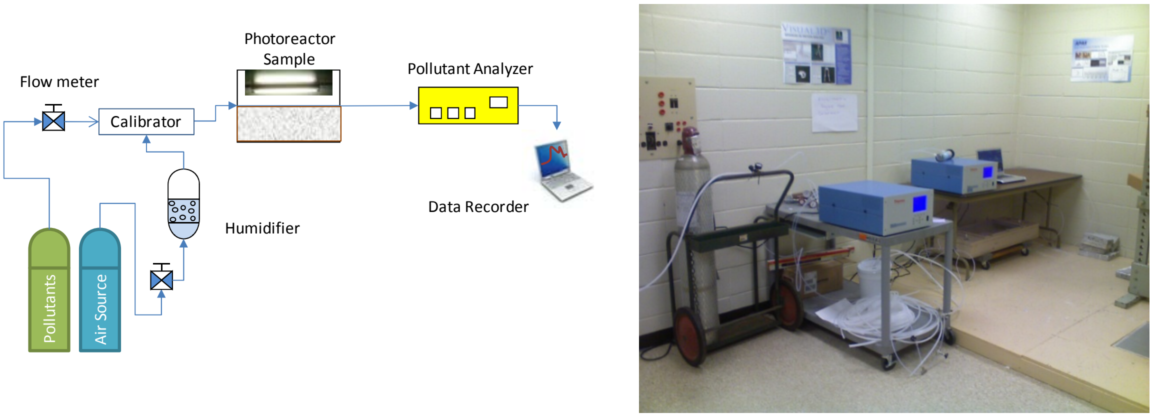
The substrate concrete samples were prepared based on a standard concrete mix design widely used in Louisiana that would achieve a compressive strength of 41 MPa. The samples were placed into molds with dimensions of 305 mm x 381 mm x 25.4 mm. The samples were cured by applying a curing compound for a period of seven days before demolding. Three replicates were prepared for each testing condition. Three methods were simulated for the application of titanium dioxide to the concrete substrate. The first method, which is the most popular technique, consisted of applying a thin coating to the concrete surface. A surface mixture consisting of ultrafine titanium dioxide, cement, filler (sand with a maximum nominal size of 1.18mm), and water was prepared. The sand aggregate was sieved to remove all fines with a particle size of 300 μm or smaller. This is based on past research that showed a coating with less fine particles would result in higher porosity, and, therefore, improve NO removal efficiency *(9)*. The surface mixture was prepared at a water-cement ratio of 0.6 and was applied to the concrete surface as a 10 mm thick coating. A commercially available titanium dioxide nanomaterial (Cristal Millennium PC105) was used at a content of 3% and 5%.

The second method consisted of applying a water-based TiO_2 surface treatment, commercially known as PURETI$^{®}$, and referred to in this paper as PT. This treatment method is applied to the hardened and cured concrete surface. A base coat is first applied as a primer in order to provide for a clean and durable surface. The water-based titanium dioxide solution is then applied to the prepared concrete surface to form an invisible ultra-thin coating that exposes the nano-sized titanium dioxide particles (about 6nm) to the atmosphere in order to initiate the photocatalytic reaction. The third method consisted of sprinkling nano-sized titanium dioxide particles to the fresh concrete surface before hardening. Particles were sprinkled at a content of 3% and 5% to the concrete substrate directly after pouring and before curing.

Environmental Test Setup

An experimental setup was built in order to quantify the environmental efficiency of TiO_2 in removing harmful pollutants from the air, Figure 1. The setup simulates different environmental conditions by allowing for control of light intensity and air humidity. The pollutants are introduced through an inlet jet stream to a 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 the 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 determination of the absorbed level of pollutants. In this study, nitrogen-oxide removal efficiency was measured using the Thermo chemiluminescent NO_x analyzer. 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 9 l/min. Testing was conducted for a total time of five hours; however, the photocatalytic process was only started after 30 minutes from the beginning of the test to ensure that steady concentration was reached in the environmental chamber.



(a) **(b)**

FIGURE 1 **Illustration of the Experimental Laboratory Setup**

Laboratory-Simulated Abrasion and Weathering

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 wheel passing back and forth over the specimen, was used in this study to simulate loading and wear 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 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. Rotary Abrasion (RA) was conducted using an in-house built device that conforms to ASTM C 944 and that 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.

Scanning Electron Microscopy and Energy Dispersive Spectroscopy

Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) were used in this study to investigate the distribution of TiO_2 in the coating surface before and after weathering. Sample preparation consisted of cutting a 25 mm x 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 JOEL JSM-840A Scanning Electron Microscope at an acceleration voltage of 15 kV. The images were stored as 1,290 × 968 TIFF files. Existence and distribution of TiO_2 was 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

Figure 2 presents the measured rut depth and its variation with the increase in the number of wheel cycles for 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 Figure 2, the measured rut depth for all specimens was minimal (less than 1mm) indicating that the use of the coating did not appear to affect the wear resistance of the surface. It is noted this test is usually employed for asphalt surface; failure is defined at a rut depth of 6 mm after 20,000 cycles. Therefore, the six specimen types appear to provide accepted resistance to wear. The best performer against wear was the coating with 3% TiO_2; this coating experienced only 0.3 mm rutting at the end of the experiment.

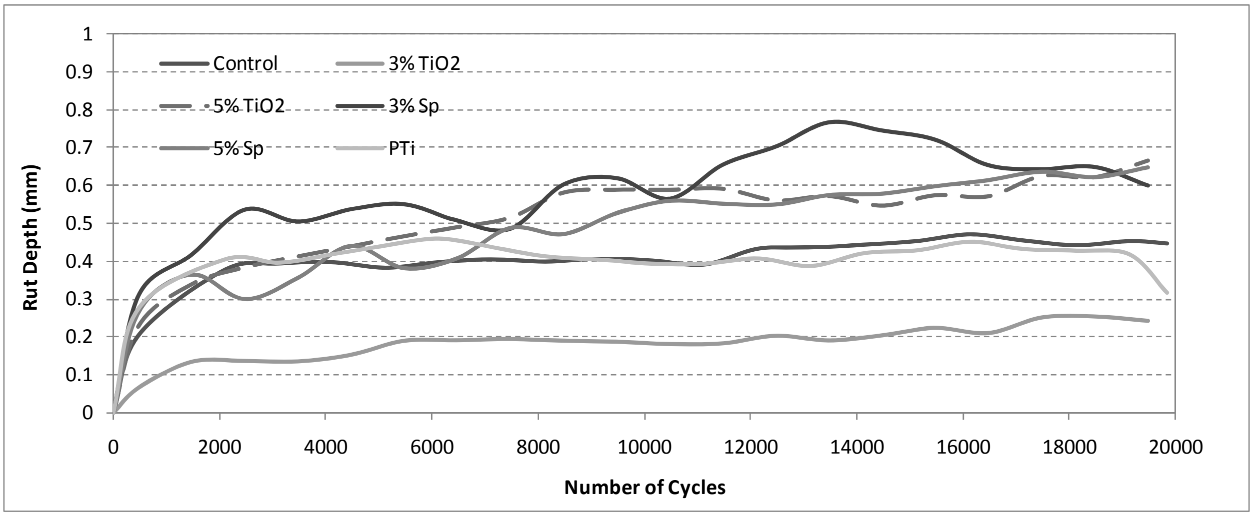


FIGURE 2 Measured Rut Depth in the Loaded Wheel Tester

Figure 3 illustrates the loss of weight observed in the rotary abrasion test for the six specimen types. Results of the rotary abrasion test seem to indicate that the use of a thin coating would be more susceptible to abrasion than the photocatalytic compound applied using the sprinkling method or using 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 rotary abrasion test may be associated with a loss of mortar, fines, and TiO_2 nanoparticles; these results were investigated using SEM.

Environmental Test Results

Figure 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 testing, the light is turned off and the NO concentration is measured. For the test condition shown in Figure 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 1 presents the measured NO efficiency for the different specimen types in the original state. As shown in this table, the coating with 5% TiO_2 and the PT product were the most efficient in removing nitrogen oxide from the air stream.

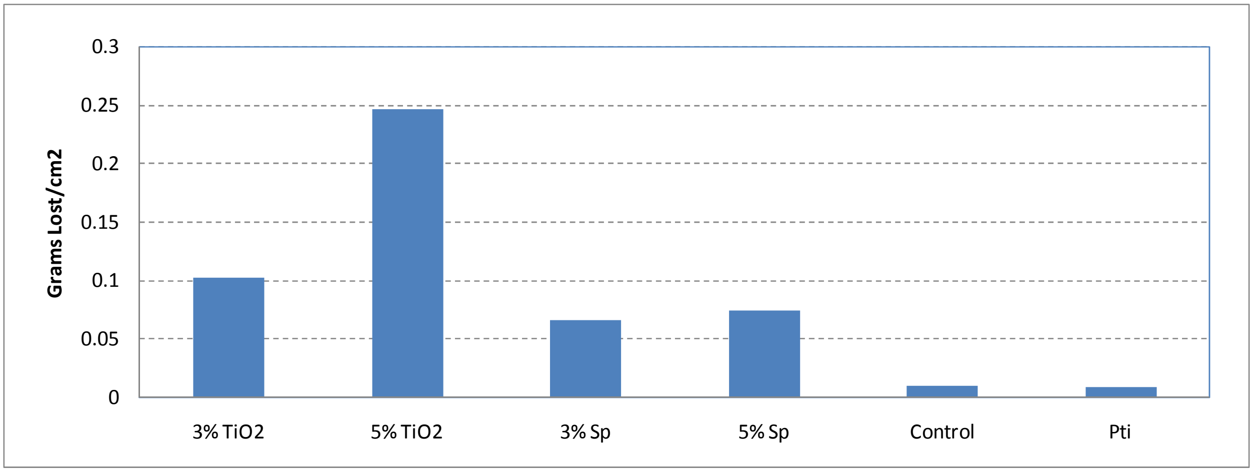


FIGURE 3 Measured Abrasion Resistance in the Rotary Abrasion Test

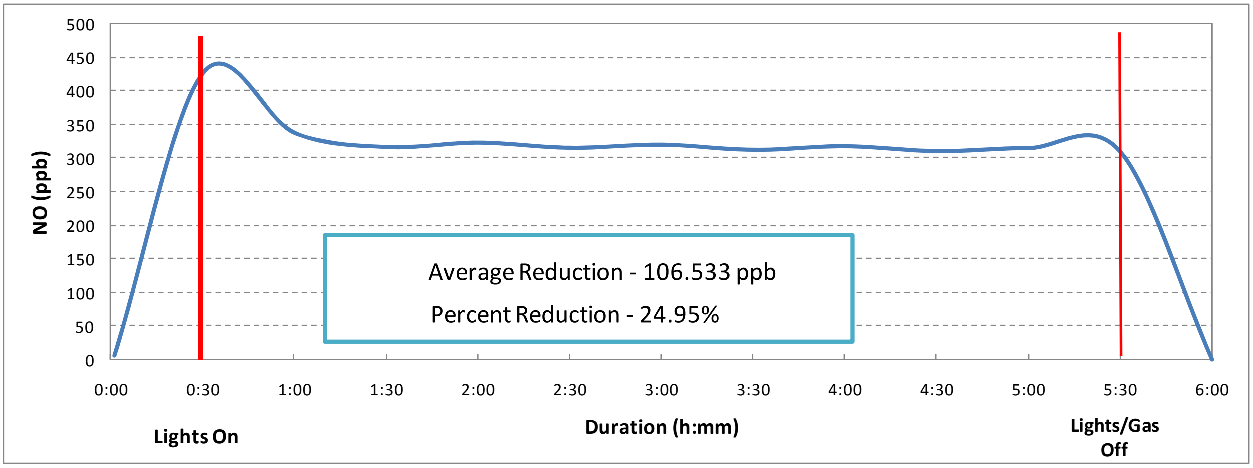


FIGURE 4 Variation of NO Concentration During the Experiment (PT)

TABLE 1 NO Removal Efficiency for Original Samples

Sample	Humidity (%)	Flow Rate (l/min)	NO Removal (%)
Control	50	9.0	2.4
3% TiO_2	50	9.0	18.0
5% TiO_2	50	9.0	26.9
3% TiO_2 Sprinkled	50	9.0	16.9
5% TiO_2 Sprinkled	50	9.0	18.9
PT	50	9.0	25.0

Effects of Weathering and Abrasion on NO Removal Efficiency

Figure 5 presents the average NO removal efficiencies for the original, weathered, and abraded samples (loaded-wheel test and rotary abrasion samples). As shown in this figure, it seems that the LWT slightly improved the NO removal efficiency of the different samples with the exception of the samples with 5% TiO_2, which experienced a small decrease in efficiency. This may be due to the weathering action simulated using the LWT, which exposed part of the embedded titanium dioxide particles at the surface, and therefore, improved its NO removal efficiency. In contrast, rotary abrasion seems to result in a decrease in NO removal efficiency for the 5% TiO2 coating and the PT product while the efficiency slightly improved or remained constant for the other specimen types. In general, 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.

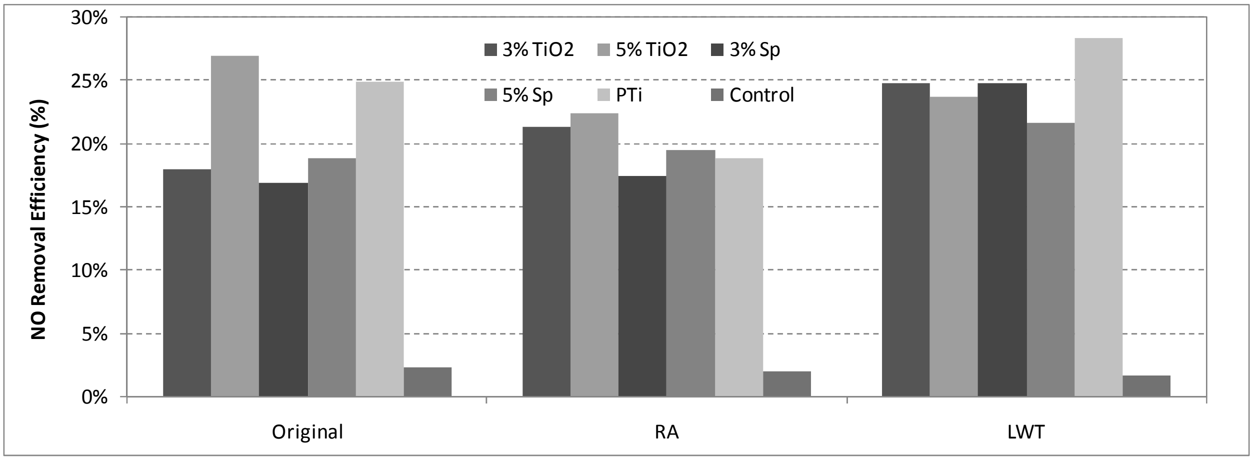
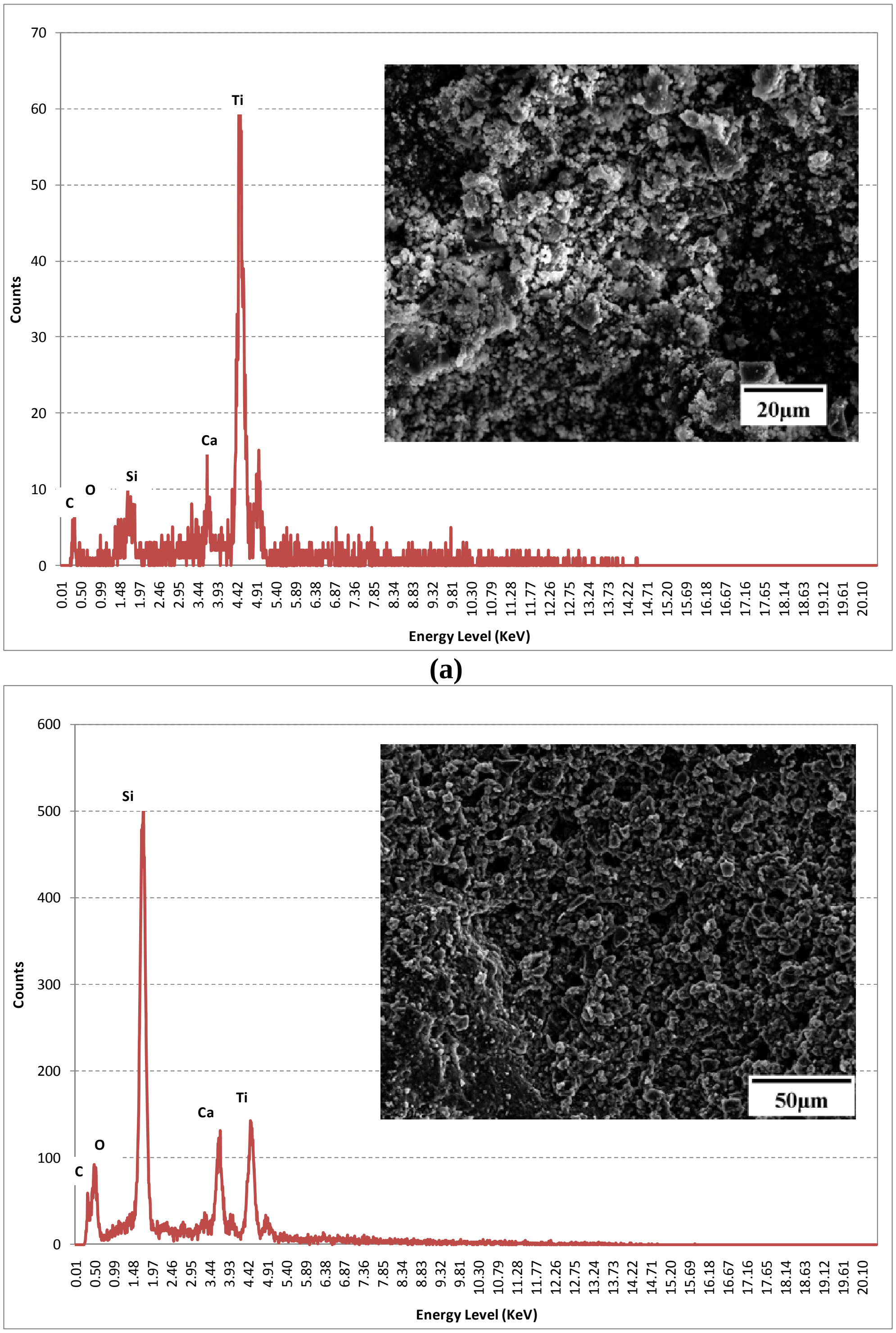


FIGURE 5 NO Removal Efficiencies for Original, Weathered, and Abraded Samples

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. Figure 6a and 6b present microscopic images of the sprinkled surface at 5% TiO_2 content and the surface treated with the PT product in the original state. White colored positions on these images are indicative of TiO_2 particles present on the surface. Along with the SEM image, results of the EDS analysis are presented. Results of the EDS analysis provide an elemental analysis of the sample, which present the distribution of titanium particles on the surface before and after weathering. From the SEM image, one may notice the uniform distribution of titanium dioxide particles across the section of the surface treated with the PT product, Figure 6(b). In contrast, the sample with the sprinkled TiO_2 particles demonstrates less uniformity with regions that do not show titanium dioxide on the surface. Uniform distribution is a desirable characteristic to ensure maximum exposure of the nano-particles on the surface, which would provide for maximum NO removal efficiency. It is also noted that the content of TiO_2 on the surface treated with PT was significantly greater than that observed on the sprinkled surface. This may explain the greater NO removal efficiency observed in the samples treated with PT product, see Figure 5.



(a)

(b)

FIGURE 6 **SEM and EDS Test Results for the Original Samples (a) Sprinkled with 5% TiO_2 and (b) treated with PT**

Similarly, Figure 7 presents the SEM image and results of the EDS analysis for the abraded sample (RA) for the concrete surface treated with PT. As shown in this figure, the concentration of Ti on the sample sprinkled did not substantially change as compared to the original sample presented in Figure 6b. However, one may notice from the SEM image shown in Figure 7 that part of the surface did not show any TiO_2 particles possibly due to abrasion. These inactive locations may explain the reduction observed in NO removal efficiency for the abraded samples.

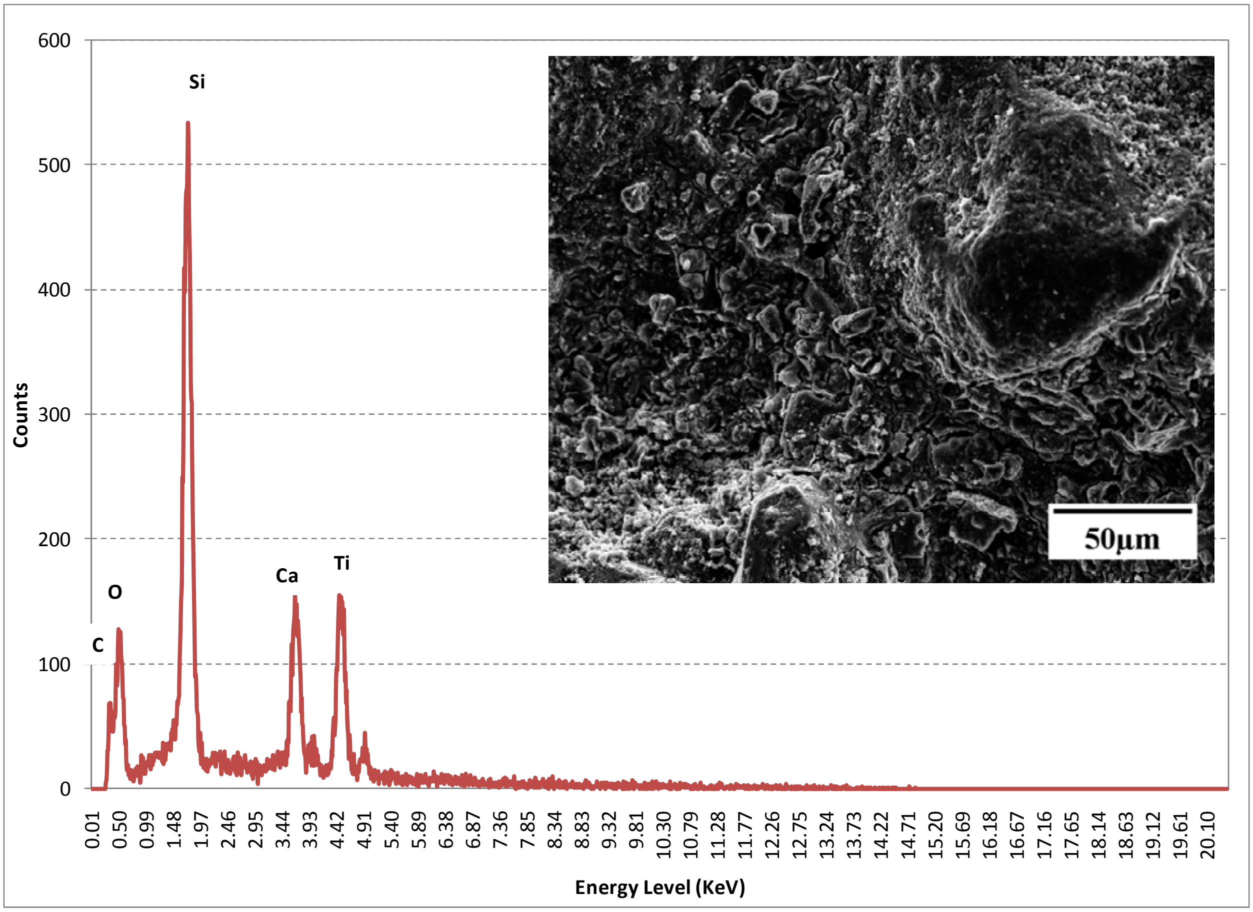


FIGURE 7 SEM and EDS Test Results for the Abraded Sample Treated with PT

CONCLUSIONS

The use of titanium dioxide coating for pavements has received considerable attention in recent years to improve air quality near large metropolitan areas. However, the proper method of applying titanium dioxide to the concrete surface is still unclear. To this end, the objective of this study was to evaluate three methods of application for titanium dioxide to concrete pavement. The first method consisted of applying a thin coating to the concrete surface at a titanium dioxide content of 3% and 5%. The second method consisted of applying a water-based TiO_2 surface treatment, PT, to the hardened and cured concrete surface. The third method consisted 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 for the three specimen types was minimal (less than 1mm). 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 indicated 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 seemed to result in a decrease in NO removal efficiency for the 5% TiO_2 coating and the PT. The highest NO removal efficiency in the RA state was measured for the coating with 5% TiO_2.
- Results of SEM and EDS analysis showed that the sample 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 PT.

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