

Durability of Epoxy-Bonded TiO₂-Modified Aggregate As A Photocatalytic Coating Material for Asphalt Pavement Under Vehicle Tire Polishing

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Abstract: Within the scope of this study, a new method to construct air-purifying asphalt pavements coated with TiO₂ modified aggregate was developed and evaluated with regard to feasibility, performance and durability. Two methods, namely the surface coating method and the pore filling method, were adopted to produce the TiO₂ modified aggregate; their durability performances in terms of both photocatalytic efficiency and mechanical performance under vehicle tire polishing applied by the Aachen Polish Machine (APM) were investigated and compared. The test results of this study indicated that it is feasible to build durable photocatalytic pavements with the developed method and spreading materials designed in this study. Both TiO₂ modification methods provided spreading aggregate with excellent and comparable NO degradation rates before polishing. However, the pore filling method exhibited a better long-term NO degradation efficiency. Both method yielded

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1 spreading aggregate with excellent long-term skid resistance and surface texture
2 properties after vehicle tire polishing.

3

4 **Keywords:** environment-friendly road surface; photocatalytic activity; nitrogen dioxide,
5 nitrogen oxides; titanium dioxide (TiO₂), wearing resistance

6 **Highlights:**

- 7 • A new approach to modify asphalt pavement with TiO₂ modified aggregate for air
8 purifying purpose was developed.
- 9 • Aachen Polishing Machine equipped with real tires was used to simulate the vehicle
10 tire polishing in the laboratory to evaluate the durability under traffic.
- 11 • Two methods, namely the surface coating and pore filling methods, were adopted to
12 produce TiO₂ modified aggregate.
- 13 • Both methods provided durable skid resistance and surface texture.
- 14 • Both methods provided satisfactory initial NO_x removal efficiency.
- 15 • The filling method provided better long-term NO_x removal efficiency.
- 16

1. Introduction

Roadside air pollution caused by automobile exhausts is a serious environmental concern, especially in urban cities with high traffic and population densities. Nitrogen oxides (NO_x), one of the most hazardous substances from vehicle emission, are harmful to both the atmospheric environment and human health. Conventionally, various measures such as using cleaner fuel and installing gas-cleaning equipment in vehicle exhaust systems have been implemented to counteract this issue by reducing the emissions from vehicle. Recently, studies have been conducted on photocatalytic pavements which have the capability of degrading NO_x into harmless substances [1, 2, 3]. The air-purifying functionality of photocatalytic pavements is achieved by incorporating or coating the pavement with catalysts, in most cases titanium dioxide (TiO_2), which are capable of degrading nitric oxide (NO) and nitrogen dioxide (NO_2) under ultraviolet (UV) irradiation [2, 3, 4, 5]. Before being applied in photocatalytic pavement, TiO_2 had been used as a self-cleansing material in many fields due to its air purifying function [6, 7]. Under UV irradiation, TiO_2 generates powerful oxidizing agents, which have very the potent capability of oxidizing NO_x to nitric acid (HNO_3), which is the final product of the degradation process. Trace amounts of HNO_3 have limited effects on the pavement performance, and can be washed away by rainwater easily and without concern [4, 6, 7]. To achieve a satisfactory air-purifying performance, TiO_2 particles should be exposed to UV light and brought in direct contact with NO_x pollutants. Correspondingly, it is important for the coating materials of photocatalytic pavements to have sufficient contact area among TiO_2 particles, pollutants and sunlight and to have sufficient resistance against vehicle tire polishing. In addition, it is necessary to ensure that the incorporation

1 of TiO₂ particles does not compromise the original mechanical performance of
2 pavements, such as skid resistance and other mechanical properties related to wear due to
3 traffic.

4 Currently, different methods have been attempted to apply TiO₂ particles onto both
5 asphalt and concrete pavements, such as: a) mixing TiO₂ with water solution or asphalt
6 emulsion and then spraying them onto road surface [3, 8], b) Using nano-TiO₂ particles
7 as asphalt modifier [9, 10, 11], c) incorporating TiO₂ to crumb rubber surface and then
8 spraying the TiO₂-crumb rubber mixture onto pavement surface during the construction
9 process [12], and d) coating asphalt pavement surface with asphalt emulsion containing
10 micro pores embedded with nano-TiO₂ particles [13]. However, so far none of
11 abovementioned methods provide a sufficient NO_x removal efficiency and durability
12 allowing for a wider practical application.

13 In Germany, the process of bonding spreading aggregate to pavement surfaces with
14 epoxy has been used as an effective treatment method to restore or improve the surface
15 characteristics of pavements (Figure 1) [14]. This surface treatment method provides
16 multiple beneficial functions, such as improving skid resistance, reducing traffic noise,
17 and enhancing smoothness.

18 Inspired by this treatment method, this study aims to develop a new method to construct
19 photocatalytic pavements by incorporating TiO₂ particles into the spreading aggregate. As
20 this form of modification consists of applying TiO₂ modified material onto the surface of
21 preexisting pavement surfaces it is of upmost importance to systematically evaluate the
22 durability of spreading materials in terms of both photocatalytic functionality and
23 mechanical properties under tire polishing. To achieve this objective, TiO₂ modified

aggregates were prepared with two different processes, namely the surface coating method and the void filling method. The TiO_2 modified aggregates were then bonded onto asphalt pavement surface to provide the photocatalytic functionality in addition to other functions such as skid resistance etc.. Since there is no direct contact between TiO_2 and asphalt in these two methods, the asphalt surface is protected from the photocatalytic processes. Moreover, a high durability of photocatalytic function is expected, because TiO_2 particles in the aggregate may keep rising even the aggregate particles are polished. To evaluate the durability and polishing resistance of the photocatalytic TiO_2 modified aggregate layer, the unique custom-designed Aachen Polishing Machine (APM), was applied to simulate the vehicle tire polishing effect in the laboratory. The APM is equipped with real vehicle tires to enable a polishing simulation which represents conditions in reality very well. Both the NO_x degradation efficiencies and the skid resistances of the photocatalytic pavements prepared with the new approach before and after APM polishing were measured to evaluate and compare the performances of two methods aggregate modification with TiO_2 .

2. Materials and Testing Program

2.1 Photocatalyst

The photocatalytic efficiency of TiO_2 modified materials is dependent on various factors, such as TiO_2 content, UV irradiation intensity, and TiO_2 type. In this study, an anatase type TiO_2 , labelled as VU7, was used, because previous studies have shown that VU7 provided the highest NO-decomposition rate among various common types of TiO_2 available in Germany [15, 16]. Some basic properties of VU7 are shown in Table 1.

2.2 Test sample preparation

2.2.1 Preparation of TiO₂ modified aggregate

In this study, the photocatalytic coating layer was prepared by spreading TiO₂-modified aggregate onto a thin layer of epoxy resin onto an asphalt pavement surface. As Figure 2 shows, two methods were adopted to prepare the TiO₂ modified aggregate, namely the surface coating method and pore filling method. In the surface coating method, the neat aggregate is mixed with cement, water and 4 M.-% TiO₂ in a rotating drum, leading to aggregate coated with TiO₂-cement film. In the pore filling method, a porous aggregate, basalt lava, which has a void content of approximately 25 Vol.%, was used. The basal lava aggregate was first submerged in a 4 M.-% TiO₂ cement suspension below atmospheric pressure for one hour to allow TiO₂ cement to penetrate into the surface pores of the aggregate. Before complete curing of the cement mortar the excessive cement was manually removed from the aggregate surfaces by means of brushes, where after the aggregate was oven dried at 105°C until constant mass.

The different methods underwent systematic investigations with regard to their mechanical strength, polishing resistance and wear/abrasion resistance, confirming that the modified aggregate complies with the respective requirements as shown in 错误!未找到引用源。 . The impact crushing tests applies a defined crushing energy onto the unbound aggregate after which the sample is passed through five sieves with defined mesh sizes. The percentage value remaining on each mesh is calculated; finally, the impact crushing value is calculated as the average value remaining on each mesh. The impact crushing value should be below the threshold value of 18. The polished stone value (PSV) represents the polishing resistance of an aggregate and is required to be higher than 51. The Chipping due to Freeze-Thaw-Cycles (FTC) represents the resistance of aggregate towards freeze-thaw-cycles. After ten

1 FTC the mass loss is recorded and is required to be below 1 M.-% (F_1).

2 2.2.2 Coating asphalt pavement with TiO_2 modified aggregate

3 After the TiO_2 modified aggregates were prepared, they were bonded onto asphalt
4 pavement surface with an epoxy coating layer. A test section was built at the Institute of
5 Highway Engineering at Aachen, Germany. The pavement was manufactured with a
6 small paver and a rolling compactor so that the applied asphalt mixture and the given
7 circumstances represent those encountered in practice as shown in Figure 2. Before
8 spreading the TiO_2 modified aggregate, the asphalt pavement surface was coated with
9 epoxy resin, on top of which the TiO_2 -modified aggregate, 2 to 5mm in size, was spread
10 and compacted. The amount of epoxy was determined so that the spreading material
11 particles were embedded to half of the diameter of the largest grains. Finally, the
12 excessive spreading material was swept away using a broom after the epoxy resin had
13 hardened. Figure 3 illustrates the process of applying TiO_2 -containing spreading material
14 to the asphalt pavement surface.

15

16 2.3 Testing program

17 2.3.1 Measurement of NO_x degradation efficiency

18 As Figure shows, a test apparatus of the Fraunhofer Institute for Molecular Biology and
19 Applied Ecology (IME) manufactured in accordance with ISO 22197-1 was used in this
20 study to measure the NO_x degradation efficiency of the testing samples with the standard
21 dimensions of 10 cm \times 5 cm \times 1 cm [17].

22 Before each test, the surface of the testing sample was first cleaned with a brush and
23 water. Then, the sample was irradiated at an intensity of 700 W/m² for 1 hour and slowly

1 shaken for 1 hour in ultra-high quality (UHQ) water. After drying for 1 hour at 60°C, the
2 sample was stored in a dehydrator until the measurement was taken. Both the
3 microscopic and macroscopic surface attachments were removed through this pre-
4 treatment and the surface was transferred to a defined initial state.

5 A moisturized mixture of synthetic air and NO was then injected into the measurement
6 cell holding the testing sample. The amount of NO was regulated so that a constant rate
7 of 1 ppm was in the gas mixture. The volume flow was controlled at 1 L/min by a
8 Fisher+Porter precision measuring tube. The relative humidity of the gas mixture was
9 controlled between 50 and 60% during the test.

10 The measurement cell was irradiated and the NO content in the outflow gas mixture was
11 continuously monitored according to the principle of chemiluminescence. During the
12 radiation, the temperature was controlled between 25 and 30°C. As Figure 5 shows, the
13 radiant energy of the xenon-lamp was 304 W/m² within the wavelength range of 290 to
14 800 nm. Within the wavelength range of 300 to 400 nm, which is relevant to the photo
15 catalysis, the radiant energy was 46 W/m², which is comparable to the radiation intensity
16 of the sun at mid-latitudes.

17 2.3.2 Simulation of long-term tire polishing

18 Continuous exposure to traffic in reality necessitates an assessment of the spreading
19 material durability under vehicle tire polishing over the course of its lifetime. Therefore,
20 in the test section described in Section 2.2.2, after the epoxy resin was completely cured,
21 core samples with a diameter of 225 mm were extracted. These cores were then
22 embedded into concrete plates and subjected to the polishing load applied by APM for
23 300 minutes, which represents the cumulative loading exerted by traffic over 8 to 15

1 years.

2 As Figure 7 shows, the APM applies shear stresses to the test plates by providing
 3 superimposed translational and rotational motion. The translational motion is achieved by
 4 a horizontally movable sled onto which the test plates are fixed, while the rotational
 5 motion is realized by rotating two polishing wheels around the vertical axis. The
 6 polishing tires have a pressure of 0.2 MPa and an imposed load of 200 kg. The sled
 7 moves back and forth horizontally 9 times per minute, while the tires spin 41 rotations
 8 per minute. The horizontal distance between the centers of the two tires is 55 cm; the
 9 velocity of the circular motion is therefore about 1.2 m/s. Such configuration allows the
 10 entire test plate subjected to an equal polishing effect. Since dust on the road consists of
 11 about 60 to 80 M.-% SiO₂ by [18], quartz powder was applied during the tests as a
 12 polishing agent. The polishing agent and water are spread evenly over the surface at a
 13 rate of 27 ± 7 g/min. Based on the findings of the previous studies, a polishing duration of
 14 300 min was selected, because the test samples will reach equilibrium after 300 min of
 15 polishing after which little or no changes would be observed by further polishing [19,
 16 20].

17 2.3.3 Measurement of surface characteristics of aggregate coating layer

18 To evaluate the effect of polishing on the surface characteristics of the TiO₂ modified
 19 aggregate coating layer, the following tests were conducted: 1) the pendulum test (SRT)
 20 according to EN 13036-4 to measure the skid resistance (Figure 8a); 2) the
 21 Wehner/Schulze (PWS) test according to EN12697-49 to measure the dynamic skid
 22 resistance at 60km/h according to EN 12697-49 (Figure 8b); and 3) the outflow test
 23 according to EN 13036-3 to measure the horizontal drainage capability (Figure 8c).

3. Results and Discussion

3.1 NO degradation efficiency

Figure 9 presents the decomposing rates of NO for typical samples prepared by the surface coating and pore filling methods before polishing. As it shows, the initial NO decomposition rate was zero and increased sharply after UV irradiation was applied to the test sample. After around 10 minutes, an equilibrium state was reached inside the chamber, with an NO degradation rate of approximately 41.5% for both samples. It is evident that without tire polishing, both methods were effective in degrading NO; the difference in NO degradation efficiency between the two modification methods is very minor.

3.2 Durability/Polishing resistance of photocatalytic properties

The NO decomposition test results of the samples after polishing are shown in Figure 10. As expected, regardless of the TiO₂ modification methods, the 300-min APM polishing significantly reduced the NO-decomposition rates of both samples. For the samples prepared with the surface coating method and the pore filling methods, the NO decomposition rates dropped to approximately 10% and 15% respectively after polishing. The reduction is mainly due to the fact that continuous polishing removed part of the active TiO₂-modified mortar at the top surface of the aggregate. Between the two methods, the pore filling method exhibited a NO-decomposition rate 50 % higher than that of the surface coating method. The microscopies of the TiO₂ modified aggregates after polishing (Figure 11 and Figure 12) clearly show that there were many residual TiO₂ particles embedded in the pores of the polished basalt lava, which were not affected by the tyre polishing. These embedded TiO₂ particles contribute to the increased durability

1 of the photocatalytic performance of the TiO_2 modified aggregate prepared with the pore
2 filling method.

3 It is worth noting that 4 M.-% TiO_2 content was used in this study for all cement mortars.
4 Previous studies conducted by the researchers of this study have shown that an increased
5 content of the TiO_2 to 7 or 10 M.-% may further increase the NO-decomposition rates,
6 while maintaining satisfactory mechanical performance of the aggregate [15]. This study
7 has shown that applying spreading aggregate modified with cement mortar containing 4
8 M.-% TiO_2 is capable of reducing the total NO-emissions from traffic by 10% to 15 %
9 under the most unfavourable polishing circumstances, which can already make
10 significant contribution to the roadside air quality improvement and emission control.

11 **3.3 Durability of mechanical properties**

12 In addition to the NO decomposition efficiency, it is important to ensure the durability of
13 the mechanical performance of the spreading aggregate.

14 Skid resistance tests were conducted on the modified spreading material to ensure that
15 safety in traffic is not compromised due to the inevitable polishing process. However, the
16 high abrasiveness of the spreading material prevented measurements with the SRT as
17 well as the PWS as it severely wears and even damages the rubber on the measuring
18 devices. The polishing process removes ragged edges and protrusions and consequently
19 smoothens the micro texture leading to a decreased skid resistance.

20 As Table 6 shows, after the polishing under the most unfavourable circumstances, the
21 SRT and PWS values of the samples prepared with both modification methods exceed the
22 threshold values specified in the regulation of the German Road and Transportation

Research Association [25, 26], indicating durable and satisfactory skid resistance of the testing samples. These two tests represent the skid resistances of a testing surface at relatively low vehicle speed (SRT: approximately 10 km/h) and high vehicle speed (PWS: 60 km/h). The outflow test results show that the outflow times of samples prepared with both methods are 1 s, which is far below the maximum value of 30 s [25], implying sufficient surface texture depth after polishing. In summary, the post-polishing test results indicated that the spreading aggregate modified by both the surface coating and pore filling methods have excellent mechanical durability in terms of both skid resistance and surface texture depth.

4. Conclusions and Recommendations for Future Research

Within the scope of this study, a new method to construct air-purifying asphalt pavements with TiO₂ modified aggregate was developed and evaluated with regard to feasibility, performance and durability were evaluated. Two methods, namely the surface coating and pore filling methods, were adopted to modify aggregate, and their durability performances in terms of both photocatalytic efficiency and mechanical performance under vehicle tire polishing were investigated and compared. The following points summarize the major findings of this study:

- It is feasible to build durable photocatalytic pavement with the developed method and designed spreading material in this study.
- Both TiO₂ modification methods, i.e., the surface coating method and pore filling method, provided spreading aggregate with excellent NO degradation rates before polishing (both approximately 40%). However, the pore filling method provided a

1 better NO degradation efficiency after 300-min APM polishing (15% in comparison
2 to 10% obtained with the coating method).

3 • Aggregate modified with both methods exhibit excellent long-term skid resistance
4 and surface texture properties after vehicle tire polishing.

5 • Field testing indicated that the method allows for the surface treatment to be
6 completed within a few hours, enabling early traffic opening and thereby reducing
7 the limiting influence on traffic flow.

8 In future study, it is recommended to further optimize the TiO_2 composition so as to shift
9 the photocatalytic effect to lower and more abundant frequencies (as opposed to UV) for
10 an even higher NO decomposition rate. In addition, the feasibility of using synthetic
11 aggregate, such as high strength ceramsite and blast furnace slag, as the spreading
12 aggregate to carry TiO_2 cement mortar should also be investigated due to their high
13 mechanical strength. Finally, life cycle cost analyses and environmental impact
14 assessments should be conducted to quantify the sustainability of the method developed
15 in this study.

16 **5. Acknowledgement**

17 The work underlying this project was carried out under FE 09.0146/2010/HRB on behalf
18 of the Federal Ministry of Transport and Digital Infrastructure in Germany, represented
19 by the Federal Highway Research Institute. The funding support of the Germany/Hong
20 Kong Joint Research Scheme (G-PolyU506/15) is also highly appreciated.

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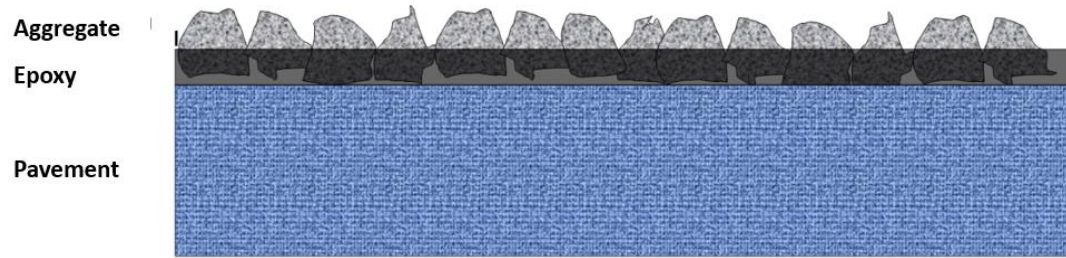


Figure 1: Surface treatment of asphalt pavement with epoxy-bound spreading material



Figure 2: TiO_2 modified aggregate prepared by means of: (a) Coating method; (b) Filling method

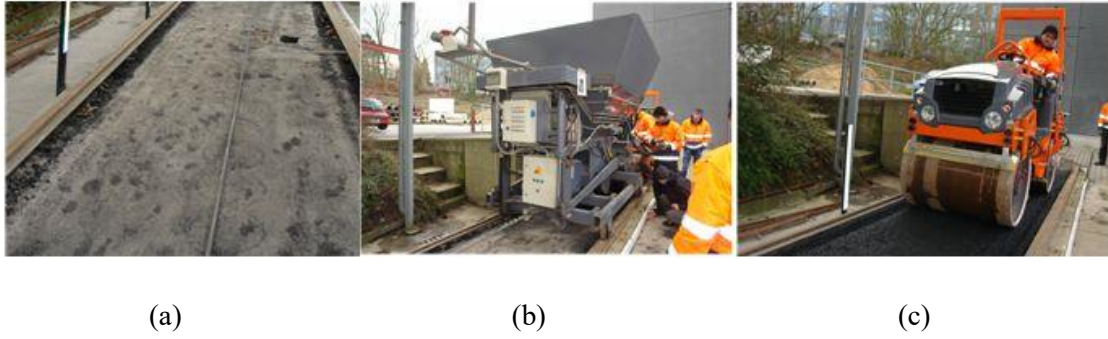


Figure 2: Asphalt mixture paving of test section: (a) test section before asphalt paving; (b) paving of asphalt mixture; (c) compaction of asphalt mixture



Figure 3: Application of the epoxy resin and spreading material: (a) application of epoxy resin; (b) application of spreading materials and removal of excess aggregate; (c) final asphalt pavement surface treated with spreading material



(a)

(b)

Figure 5: Measurement of NO degradation efficiency: (a) Sunset radiation apparatus and Horibo NO analyzer; (b) test specimen (10 cm × 5 cm × 1 cm)

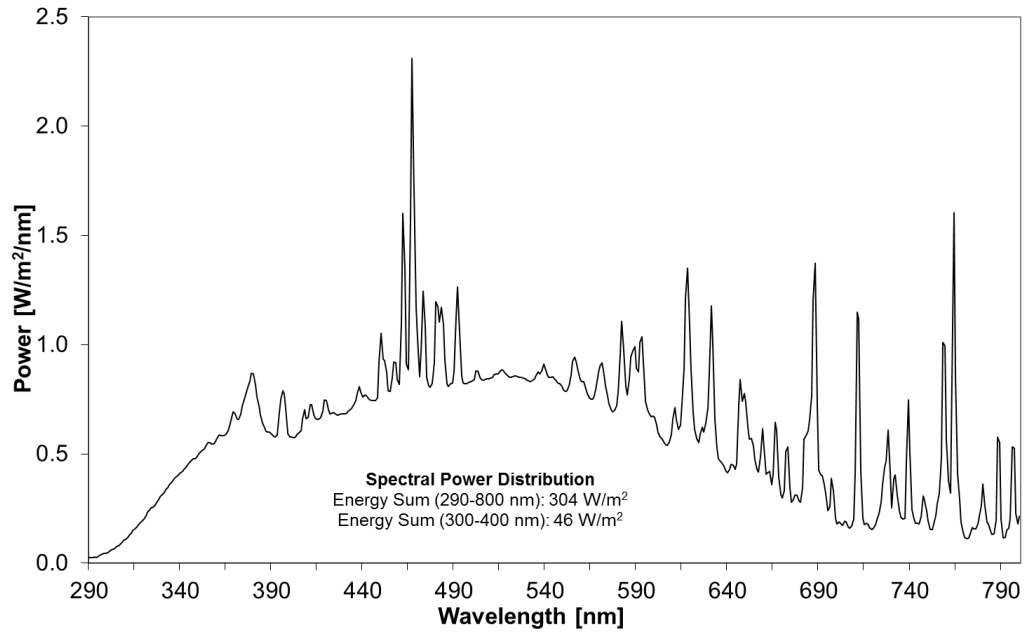


Figure 6: Spectral power distribution of the xenon-lamp light



Figure 7: Aachen polishing machine (APM)



(a)

(b)

(c)

Figure 8: Tests to measure surface characteristics of asphalt pavements with epoxy-bound spreading material: (a) Pendulum test; (b) W/S test; (c) Outflow test

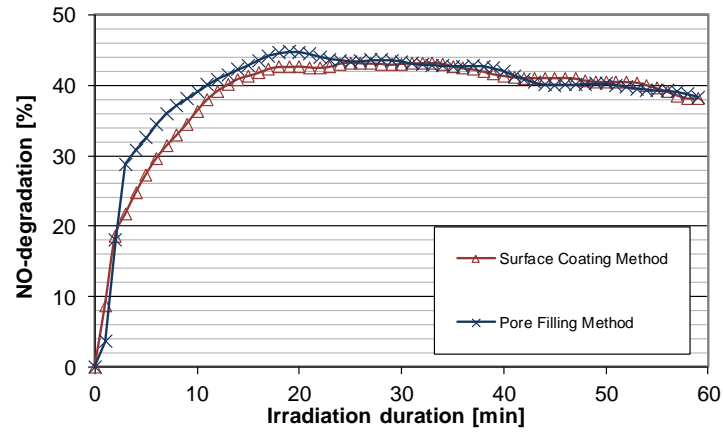


Figure 9 Reduction of nitric oxide before simulated polishing

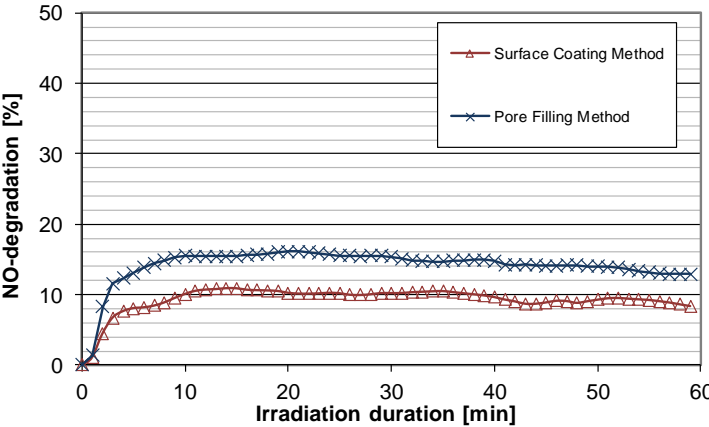


Figure 10: Reduction of nitric oxide after polishing



Figure 11: Polishing effect on TiO_2 modified aggregate (surface coating method): before (left) and after (right) polishing

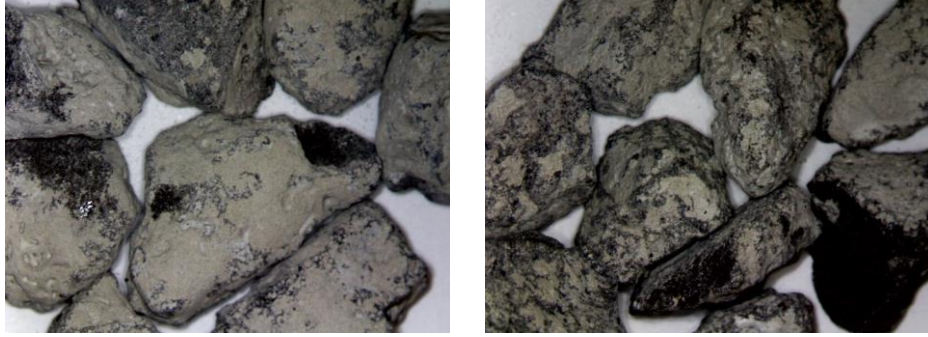


Figure 12: Polishing effect on TiO₂ modified aggregate (pore filling method): before (left) and after (right) polishing

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Table 4: Basic properties of the selected TiO₂: Antanase

Label	Mineralogical Type	Grain Size (nm)	Surface Area (BET) (m2/g)	pH	TiO ₂ content (M.-%)
VU7	Anatase	15	90	1.5	99

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Table 5: Mechanical properties of spreading material and requirements

Requirements	Method 1	Method 2
Impact crushing according to DIN EN 1097-2 (reference value: SZ_{18})	11.5	17.8
Polished stone value (PSV) according to DIN EN 1097-8 (reference value: 51)	58	55
Chipping after freeze-thaw-cycling according to DIN EN 1367-1 (Reference value: F_1)	F_1	F_1

1 **Table 6 Results of pendulum test, W/S test, and outflow test after 300 min polishing**

Requirements	Method 1	Method 2
Pendulum Test (SRT) according to EN 13036-4 (>65)	65.4 (± 0.3)	68.8 (± 0.7)
Wehner/Schulze (PWS) according to EN 12697-49 (>0.45)	0.491	0.493
Outflow time according to EN 13036-3 (<30s)	1	1

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