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Cement Matrix Composition Impact on the Photocatalytic Performance of Immobilized TiO_2 Particles over the Fixed Bed photoreactor for Denitrification of Water

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ABSTRACT

Effective denitrification of water using photocatalytic reaction of active TiO_2 particles doped with different oxides and metals has been the subject of numerous studies. For a particular research area, the potential of silica bond and its silicate based matrices with titanium dioxide and improving the photocatalytic performance using more economic methods is still challenging, and research in this field is attractive and ongoing. In this study, the effect of cement matrix and its complex bonds with industrial grade TiO_2 particles was evaluated on the rate of water denitrification in a fixed bed circulating flow photoreactor. For this purpose, silica fume was substituted for cement in constant percent of 10 as a rich source of amorphous silica. Industrial grade TiO_2 was added to the mix as5, 10 and 15 percent weight of cementitious materials (CM). Nano TiO_2 was considered as a supplementary photocatalytic material with a constant 1% weight of CM in two mix designs. The results implied that the addition of 5% TiO_2 increased the rate of nitrate concentration reduction by up to 10 times. Also, the specimen including 10% TiO_2 increased denitrification rate by 107% compared to the previous content, which had much less impact. Also, the addition of nanoTiO_2 increased denitrification rate up 113%.

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INTRODUCTION

Titanium dioxide (TiO_2) has been used as a very effective and economic photocatalyst in combination with various dopants and materials to reduce the concentration of various pollutants (mainly organic) in water (Lee and Park, 2013). This combination with durable and inactive mirostructural properties against a variety of corrosive agents (even strong acids) is a very desirable option for photocatalytic reactions. However, the applications of this oxide are not limited to treatment industries and wide range of applications are demanded from this photocatalyst (Denis et al., 2019; Wanpei et al., 2020). One of the primal applications of TiO_2 as a purifying material into self-cleaning applications is in cement composites as construction material (Bellarditaa et al., 2010). The various oxidation mechanisms of photocatalysts, such as TiO_2, have been studied in numerous projects and the electron exchange phenomenon from

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an electrochemical perspective has been discussed thoroughly (Li *et al.,* 2006). Although many alternatives such as ZnO and CdS are introduced in photocatalytic processes, TiO_2 is still more widely used due to its high sensitivity to visible light, abundancy, and low cost (Hamilton et al., 1992). Studies reveal that the anatase TiO_2 has a higher photocatalytic activity than the other two crystallographic forms, rutile and brookite (Adebambo, 2011). A number of studies have been devoted to the removal of common microbial contaminants and the analysis of chemical contaminants in terms of sensitivity of the material and significant impact on the environment is considered as another topic in the field of photocatalytic treatment (Ceyda et al., 2018; Durán et al., 2018). As mentioned in research reports, doping has been promising to increase the sensitivity of TiO_2 to visible light (Yahui et al., 2019; Shahina and Park, 2020). In the field of wastewater treatment, TiO_2 has been introduced as a photocatalyst and has been used in various studies to remove organic and toxic pollutants (Al-Mamuna et al., 2019; Horikoshi and Serpone, 2020). One of the initial studies in the matter of utilizing nano-TiO_2 in concrete and studies on the removal of contaminants (e.g. methylene blue) was the work of Shen et al. (2014). They introduced a very smooth surface consisting sub-micron particles from the hydration of cement as the main factor in the effective performance of nano-TiO_2 as a photocatalyst (Shen et al., 2015). In another study, the effect of additives, hardness and microstructure were investigated. Removal of dyes, and decomposition of nitrogen-based oxyanions were the main prospects, and the highest efficiency results were obtained for the removal of dyes in the first 4 hours of irradiation (Jimenez-Relinque et al., 2015). Some studies focus also specified to the antifungal characteristics of TiO_2. For example, a fungus called Cladosporium sp. Cultivated in laboratory environment and the effects of photocatalyst on it have been studied. It was found that the application of TiO_2, even in micrometer particle size, reveals significant antifungal and photocatalytic effects on the surface of cement mortars (Loh et al., 2018). Zanfir et al. (2018) merged silica with TiO_2 and studied the compatibility of this composite with the cement matrix. Methylene blue was used as a reference contaminant and an increase in photocatalytic activity and improvement of mechanical properties were reported in their research (Zanfir et al., 2018). In another study, the impact of different types of TiO_2 in combination with silica on the degradation of rhodamine B were investigated. The bond of silica particles with the cement matrix was considered and the effects of this bond on photocatalytic activity were analyzed (Wang, et al., 2020). Gao et al. (2004) studied the reduction of nitrate in water by the photocatalytic performance of TiO_2 doped with dual metals such as nickel and copper, which at the time was the first photocatalytic experiment in the water treatment field (Gao et al., 2004). In another independent study by Jin et al. (2004), a mixture of oxides including TiO_2, copper and magnesium in the form of Cu/MgTiO3- TiO_2 was used. Sodium oxalate was used as a hole scavenger (Jin et al., 2004). The effect of silver along with TiO_2 as a factor in improving the photocatalytic activity to reduce nitrate in water has also been investigated. According to this study, the use of silver spikes along with TiO_2 and the use of formic acid as a hole scavenger, led to a 98% reduction of nitrate in 30 minutes (Zhang et al., 2005). In the work of Sa et al. (2009), in addition to the experimental cases, a handful of research projects utilizing formic acid, silver, copper and iron as a dopant toTiO_2 were considered. In the mentioned study, parameters such as photocatalyst particle size, reaction temperature, initial concentration, the type of hole scavenger and the doped metal were found to be effective. The use of silver as a dopant metal along with industrial TiO_2 has been shown to be promising in denitrification below the standard threshold (Sa´ et al., 2009). In another study, benzene was used as a hole scavenger and platinum and copper were used as TiO_2 metallic dopant to effectively reduce nitrate concentrations utilizing visible light spectrum. The percentage of photocatalyst was reported up to 5%. The optimal ratio of platinum to copper was 4 and the highest efficiency was reported based on this combination (Li et al., 2010). Doudrick et al. (2012) were the first researchers to study the effect of industrial TiO_2 with different mechanisms of nitrate reduction.

In the pursuit of viable photocatalysts, a group of researchers tested three different industrial types of TiO_2 with the help of hole scavenger (formic acid) and proposed the optimal type (P90) based on controlled parameters (Li et al., 2005). They also continued their research, finding suitable industrial TiO_2, using sodium formate as a hole scavenger and nano-silver as TiO_2 dopant, and reported more promising results at lower pH. They concluded a nitrate reduction of 2.5 to 85% at low pH (Doudricka et al., 2013). In the study of Soares et al. (2014) in addition to using platinum-copper dopant along with TiO_2, various types of hole scavengers including: formic acid, oxalic acid, humic acid, ethanol and methanol were used. In addition, the role of hydrogen as a reducing agent and carbon dioxide as a buffer was investigated (Soares et al., 2014). In addition to the reduction in mineral ion concentrations by TiO_2, several cases of decomposition of organic materials such as hexachlorobenzene is accessible in research papers, one example of which has resulted in acceptable intervals of decomposition rates using a thin film of nano titanium dioxide (Shengyong et al., 2012). Also, optimizations were performed on nanocomposites including silver-doped TiO_2 chitosan and alumina by Zarei et al., Which resulted in the removal of 74% nitrate at pH 11 for 5 minutes. They used the UVA light range up to visible spectrum to activate the photocatalyst (Zarei et al., 2019).

In all of these studies, TiO_2 activity was intensified to the highest reachable level or used as a powder in Slurry photoreactors, which, of course, would raise significant economic challenges at industrial scale. In the present study, using industrial TiO_2 and making photocatalytic cement composite with this photocatalyst as a fixed bed in the reactor, the rate of denitrification under UV irradiation was investigated and the results of nitrate concentration reduction according to different mix designs for bed materials and surface acid etching method have been analyzed.

MATERIALS AND METHODS

Potassium nitrate

In the present study, Potassium nitrate from Merck Co. utilized to reach the concentration of 100 mg/L Nitrate Pollution in the water reservoir.

Cement

The white Portland cement (WPC) used in accordance with ASTM C150 (ASTM C150, 2015). The cement was provided from Urmia White Cement Co. Chemical characteristics are reported in Table 1.

Titanium dioxide

Two different types of TiO_2 used in photocatalytic bed. Sigma-Aldrich nano-TiO_2 (<25 nm particle size, 99.7% trace metals basis) used for photocatalytic activation of the composite. Industrial anatase TiO_2 (Cosmo Chemical Co., KA-100) with an average particle diameter of 300 nm, minimum purity of 98% (provided by the manufacturer) and specific gravity of 3.96 gr/cm^3 was used in mix proportions.

Table 1. Chemical compositions of cement and silica fume (provided by the manufacturer).

Compound	Cement (%)	Silica Fume (%)
CaO	66.5	1.87
SiO2	23.18	89.22
Al2O3	4.528	1.2
Fe2O3	0.392	2.12
MgO	0.77±0.1	1.61
K2O	0.12	1.056
Na2O	0.35	0.556
L.O.I.	2.39±0.5	2.6±0.5

Table 2. Mix designs for photocatalytic cement composite beds

Mix Code	Cementitious Materials	Nano TiO_2	TiO_2 P25	Water +superplasticizer	Aggregate
P0	900	0	0	342	987
P5	900	0	45	342	913
P10	900	0	90	342	839
P15	900	0	135	342	765
P5NA	900	9	45	342	898
P10NA	900	9	90	342	824

Silica fume

Silica fume used as constant 10% replacement of WPC. The silica fume was purchased from Iran Ferrosilice Co. Chemical characteristics are reported in Table 1. This silica fume is compatible with the standard specifications of ASTMC1240 (ASTM C1240, 2020).

Sand

Silica sand was purchased from Parssilis Co. The grading of this sand was passing sieve No. 30 (max. particle size of 600 μm) which was used as aggregate in the mixture.

Superplasticizer

FARCOPLAST P100-3R polycarboxylate-based superplasticizer provided from Shimi Sakhteman Co. with a specific gravity of 1.1 gr/cm^3. It's used as a high-range water-reducing agent to conrol the fluidity of the fresh mixture. This superplasticizer is compatible with the standard specifications of ASTM C494 type G (ASTM C494/C494M, 2017).

Fabrication of the photocatalytic bed

To achieve the photocatalytic activity of TiO_2 in the cement matrix of the bed, TiO_2 particles and silica fume mixed separately in a solution containing 25% neutral polycarboxylate superplasticizer at a concentration of 100 g/lit. for 10 minutes. The materials were mixed with an ultrasonic homogenizer at 23°C and immediately added to the fresh mixture to reach desired distribution and avoid ineffective porosity to increase the photocatalytic efficiency of the composite surface. As implied in Table 2, both industrial and nanoTiO_2 were added as a weight percentage of CM and the amount of aggregate was adjusted based on the volume of the paste. The total content of CM material was considered constant at 900 kg/m^3. The fabricated cement composite material was freshly mixed and poured into the mold in a self-compacting rheological state and removed from the mold after 24 hours. Fabricated specimens were cured by accelerated method (hot water at 90°C) for 24 hours. Acid etching process (2M HCL) was used for 30 seconds to enhance the contact surface reaction capability (NA and SA series). The cement composite pipe (Fig. 1) containing the photocatalyst with 28 days compressive strength of 62 MPa, internal diameter of 4 cm, length of 23 cm and wall thickness of 10 mm was considered as a fixed bed photocatalytic surface inside the photoreactor.

The UV system was installed in the axis of the tube and the tube in the inner wall of the reactor acted as a photocatalytic surface. The inner wall was irradiated with a set of UV LEDs with a total power of 20 watts. Higher irradiation power up to 50 W was tested to measure photocatalytic capacity of the bed which concluded the 20 W LED lamp as an optimum irradiation capacity of the surface. A stream of nitrate-polluted water with a flow rate of 13 lit./min was chosen to avoid turbulent flow inside the reactor. Furthermore, lower flow rates (5 and 10 lit./min) led to lower denitrification rates. Initial nitrate concentration of 100 mg/lit., with a total volume of 5 liters in the reservoir, was established inside the reactor. The initial nitrate concentration was considered constant as a parameter to focus on the effect of the bed

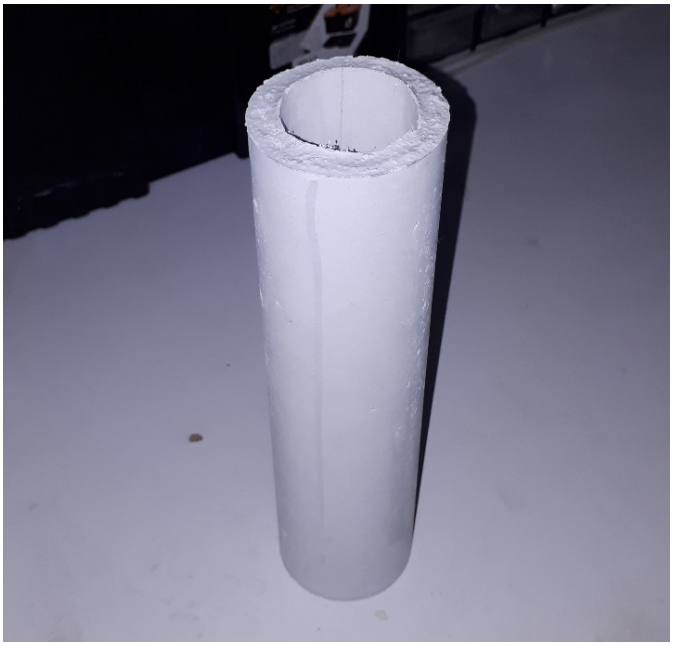

Fig. 1. photocatalytic cement composite specimen as a fixed bed

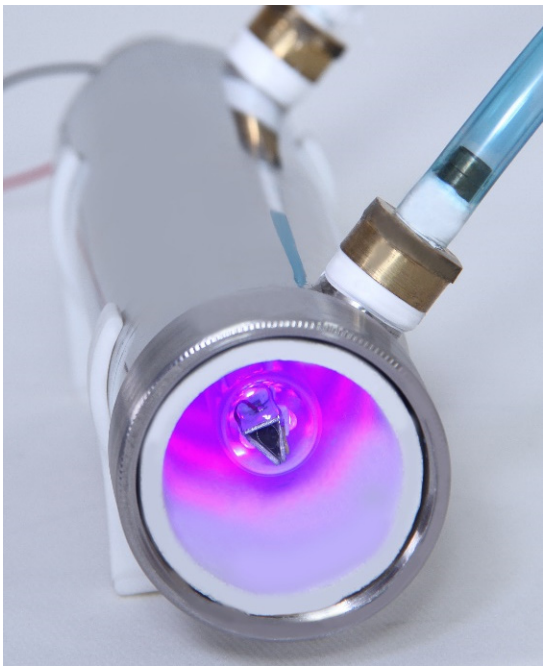

Fig. 2. an inside view of the circulating flow fixed bed photoreactor

characteristics. The water circulated inside the reactor for the total time of 4 hours. Nitrate concentration during the experiment period was measured through 5mL. sampling from the reservoir and determined by the method described in APHA (APHA/AWWA/WEF 2012). Each test according to the time periods was iterated twice. The arrangement implied in Fig. 2 shows the main body of the photoreactor and the specimen inside. The body is made of stainless steel and the screw cap on the body allows the specimen to be replaced easily. achieving samples was performed during the purification process from the reservoir at the specified time steps.

RESULTS AND DISCUSSION

The difference between TiO_2-free specimens and the pipes containing it is quite evident even at low percentages (P5), and a decreasing slope of nitrate concentration is observed for all specimens except the control (TiO_2-free) mix design. A noticeable fact that can be found in the diagrams of Figs. 3 to 5 is that they are linear with a relatively high correlation. This

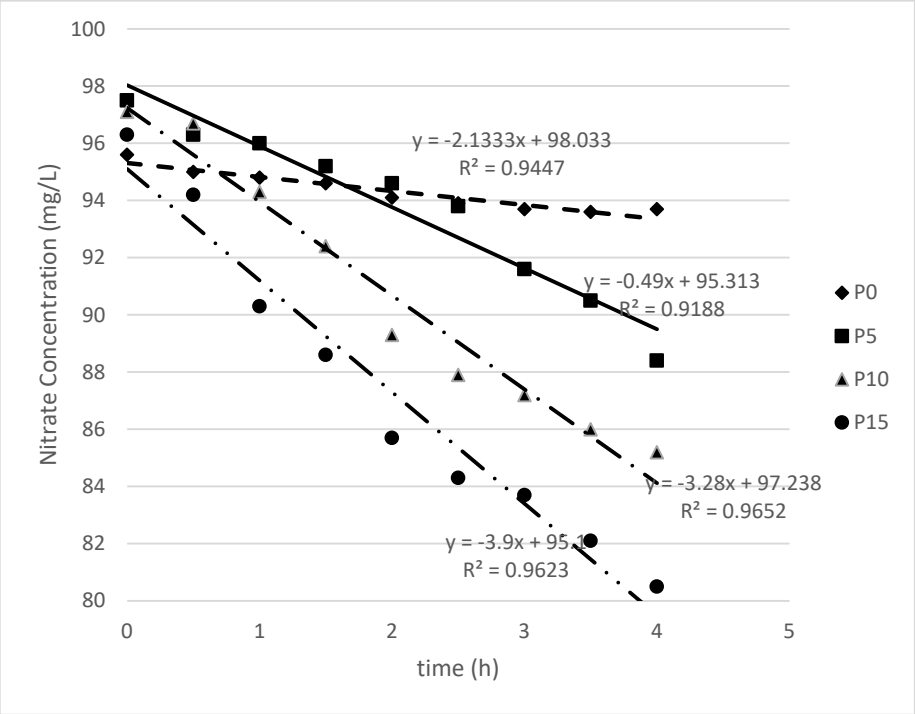

Fig. 3. Reduction of nitrate concentration over time for nano- TiO_2-free pipes and without surface polishing (Q=13 L/min, pH=7, C_0Nitrate=100 mg/L, T=25°C)

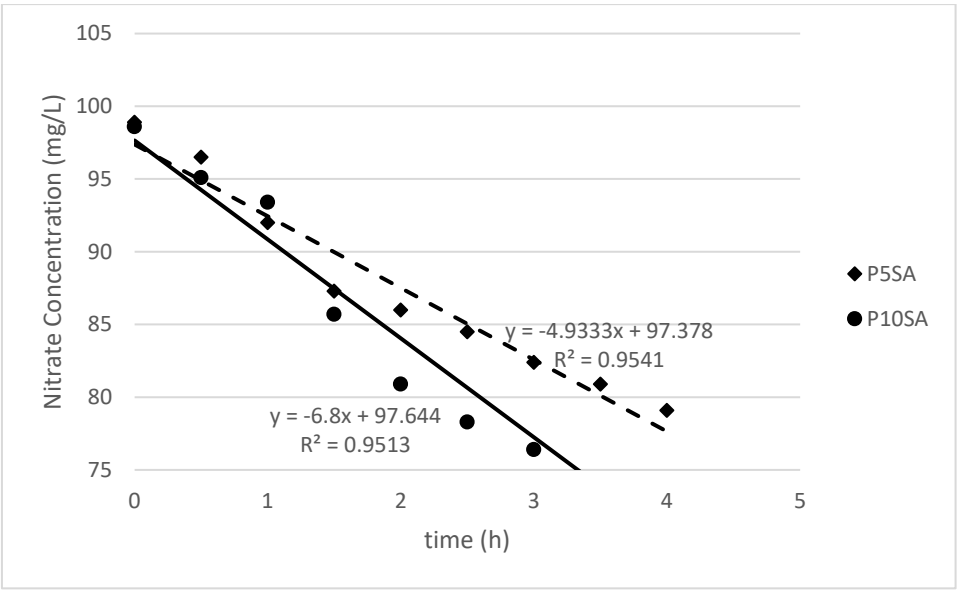

Fig. 4. Nitrate concentration reduction over time for nano- TiO_2-free pipes with inner wall surface finishing (acid etching) (Q=13 L/min, pH=7, C_0Nitrate=100 mg/L, T=25°C)

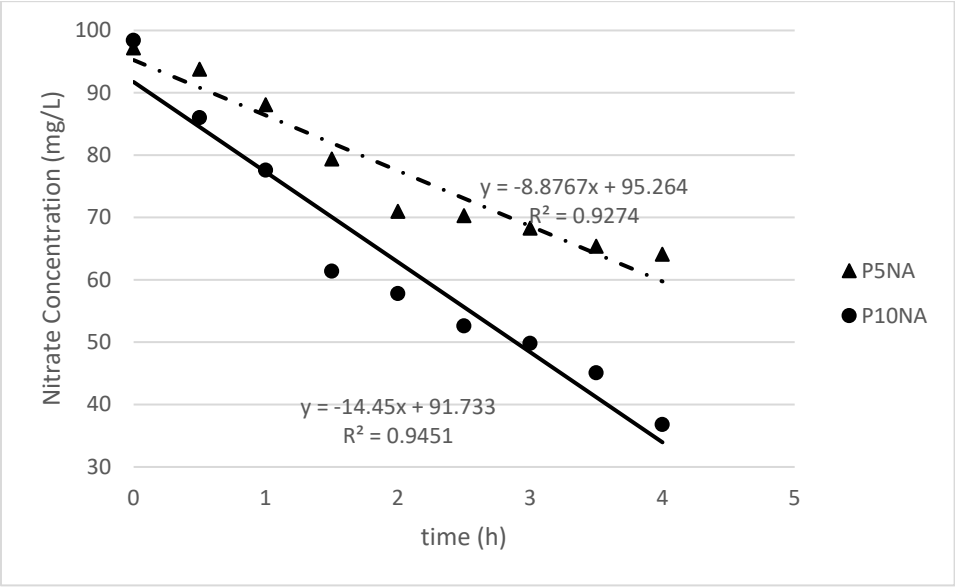

Fig. 5. Reduction of nitrate concentration over time for pipes containing nano- TiO_2 with inner wall surface finishing (acid etching) (Q=13 L/min, pH=7, C_0Nitrate=100 mg/L, T=25°C)

linear fit indicates a significant decrease in the first 4 hours of irradiation and no decrease in photocatalytic activity even with a decrease in nitrate concentration which gradually happens during the circulation. One logical debate on the linear slope is the relatively low efficiency of the reactor in the period of experiment which candidates the data for lower denitrification rates and thus the common exponential fit of degradation curves found in the literature doesn't rule in this study. The other hypothesis is the closed loop circulation with a considerable flowrate of water stabilizes the reactor bed activity with a roughly constant rate of denitrification which possibly arises from a continuously absorption-desorption of the nitrate ions on the surface of the photocatalytic bed. Also, based on the diagram in Fig. 3, it is obvious that the difference between 5 and 10 percent TiO_2 in the denitrification is greater than the difference between 10 and 15% TiO_2. Also, the effect of surface etching of cured specimens containing 5 and 10% TiO_2 (Fig. 4) shows a significant increase in the rate of concentration reduction, which at 5%, this rate increases up to 10 times. At 10% of TiO_2, there is a 107% increase in denitrification rate, which is much less for 5% mix. The addition of nano-TiO_2(Fig. 5) along with the surface finish again creates a significant increase in denitrification rate. This increase in 5% is around 80% and in 10% of TiO_2 reaches to 113%. It can be implied that the synergy of nano- TiO_2 with industrial TiO_2 increases by 10% and intensifies its effect. The diagram in Fig. 6 clearly shows the best result for the 10% nanoparticles containing surface coatings. But the difference between the results in 2- and 4-hours periods is also a significant point of this figure. At 5% TiO_2 as well as 10% including nano, the greatest difference is seen between 2- and 4-hour results. Also, the significant difference between the composite mix design containing 10% TiO_2 and other mixtures, clearly indicates the effectiveness of the nano-included composite and the surface finish at higher percentages of industrial TiO_2 consumption.

Table 3 demonstrates a comparison between the results of 3 studies and the current research in denitrification kinetics. According to the mentioned studies, extremely high removal efficiencies up to 98% were achieved. However, a key and very influential parameters that is not taken into account in the calculation of the removal rate is the initial polluted water volume and the performance of the reactor. The removal efficiency based on the bed surface area and the amount

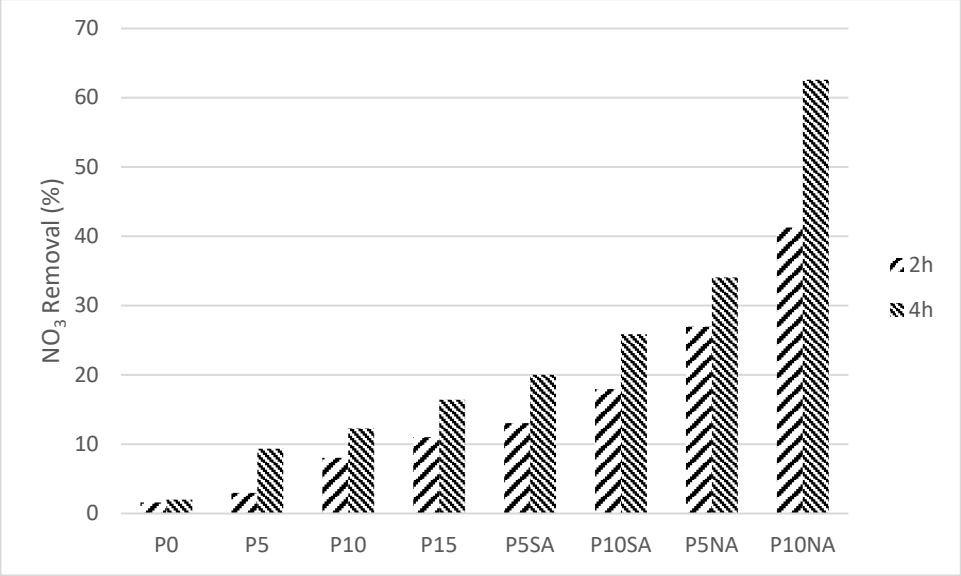

Fig. 6. Amount of nitrate degraded in percentage for different types of pipes at intervals of 2 and 4 hours

of irradiation energy spent per unit area of this photocatalytic bed are vital characteristics of the reactor which are enlightened in equations 3 and 4. The results of these calculations are reported in the table 3 for this study along with other 3 reports. Including the beforementioned parameters to compare the energy demand and denitrification efficiency of the reactor reveals that the utilization of nanomaterials or high removal efficiencies as common comparison tools may not identify the true performance of the operated reactors and the photocatalyst performance. Based on the new calculations, the difference is substantial enough so that the energy demand of the reactor in other studies is at least 10 times of the photoreactor used in this study. Furthermore, the photocatalytic efficiency of the present reactor's fixed-bed is at least 1182 times higher than the best result observed in other reports (Ramirez et al. 2012). There is a significant difference in this comparison group. The mentioned studies used slurry reactors and TiO2 directly introduced into the polluted water in the form of powder. Nevertheless, the complexities of this method and the challenges of powder separation and filtration, in this group the studied reactor has consumed less energy for denitrification at a certain rate (neglecting the energy required to separate the photocatalyst from water). In the comparison of the contact surface of the photocatalyst, considering that the nature of the photocatalyst used in other research reactors is powder, the contact surface was calculated based on the specific surface area (BET) of the photocatalyst and the amount weight used in water.

$$KT = ln\frac{C_0}{C_T} \tag{1}$$

$$RE = \left(C_0 - C_T\right) \times 100 / C_0 \tag{2}$$

$$MRE = PT / \left(VC_0 \times RE\right) \tag{3}$$

$$SRE = \left(VC_0 \times RE\right) / \left(T \times A\right) \tag{4}$$

K is the photocatalytic reaction constant, T is the total treatment period, C_0 is the initial molar

Table 3. Comparison of the denitrification rates obtained by several studies and the current study in terms of removal efficiency, reaction rate constant, molar removal energy, and surface removal rate.

Photocatalyst/Support	Volume of nitrate Sol. (mL)	$[NO_3^-]_0$ ($mol.L^{-1}$)	Power (W)	Contact Surface (cm^2)	Irradiance ($mW.cm^{-2}$)	Time (min)	K (min^{-1})	Removal efficiency (%)	Molar Removal Energy (kW.h/mol)	Surface Removal Efficiency ($*10^{-10}$ $mol/min.cm^2$)	Ref.
Ag/TiO2	250	7.0×10^{-3}	125	267000	0.47	30	0.13	96	0.036	21.4	(Lozovskii et al, 2009) [32]
Pd-Cu/TiO2	180	7.0×10^{-3}	150	107000	1.40	240	0.008	84	0.567	4.12	(Vergili et al, 2012) [33]
Pd-Cu/TiO3	500	3.5×10^{-3}	75	1070000	0.07	60	0.065	98	0.044	2.67	(Wang et al, 2021) [34]
TiO_2-CC (P10NA)	5000	7.0×10^{-3}	20	361	55.4	240	0.0041	62.6	0.0036	25289	Current Study

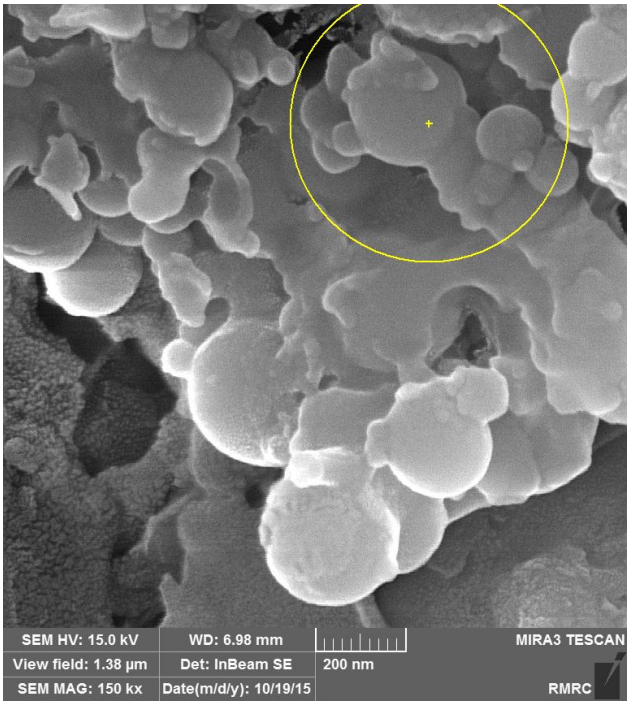

Fig. 7. Cement matrix structure optimized with silica fume particles

concentration of the pollutant, C_T is the molar pollutant concentration at the end of the treatment period, RE is the removal efficiency of the contaminant during the reaction period, MRE is the required energy for degradation of one mole of the pollutant, P is the power consumption of the UV lamp in kW, V is the initial volume of contaminated water in liters, SRE is the pollutant removal rate per unit photocatalytic bed, and A is the total photocatalytic area exposed to UV radiation.

Microstructure

Cement matrices, which are actually obtained from the quasi-polymer-like development of calcium silicate crystals due to their hydration, form a relatively uniform texture and, if the properties are controlled and the mixture is sufficiently compacted, very fine particles, even in nanometer dimensions, are well merged and hydrated in a quasi-form of polymerization. Silica fume, which is an amorphous type of silica and a considerable percentage of it is in the nanometer range, performs two different types of operations with a fully spherical-shaped structure. In the amorphous type, it is chemically reacted to become part of hydrated calcium silicate gel (CSH), improving the structural integrity of the matrix, and in its second function helps to improve the density by filling in voids. In composite systems where $TiO_2$is used in the mixture, silica fume plays the role as an accelerator of TiO_2 photocatalytic performance. Fig. 7 shows the association of spherical-shaped particles with the cement matrix. The relatively uniform size distribution of the silica fume results in more compact microstructural system and better electron exchange with TiO_2 particles.

In Fig. 8, the chemical bond of the cement matrix with the wall of the silica particles and the association of smaller particles with larger ones is well illustrated. This cohesion formed after completion of the hydration reaction and completion of the pozzolanic reaction (chemical reaction of excess lime with silica particles) leads to increased contact surface due to near perfect particle packing (due to lower abrasion resistance of CSH gel compared to silica) and

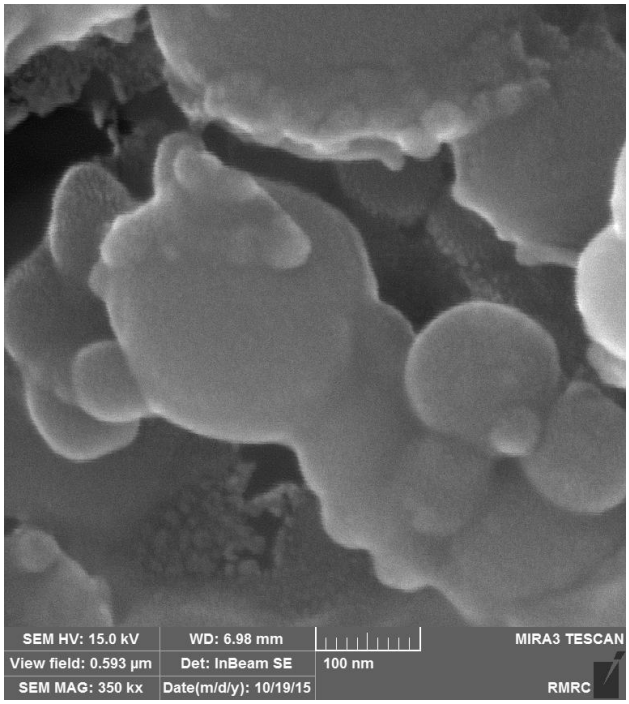

Fig. 8. Development of CSH gel around amorphous silica particles

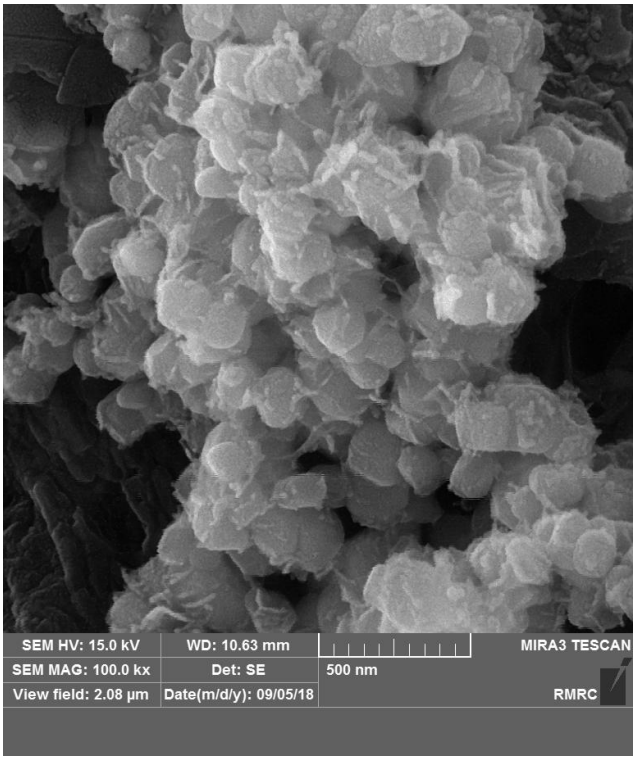

Fig. 9. Stabilization of TiO_2 particles in the matrix substrate by CSH gel development

increased surface photocatalytic efficiency. The charge asymmetry is between the silica and TiO_2 particles. Fig. 9 shows the trapped particles of TiO_2 in the cement matrix. From the magnitude of the image, we can understand the scales of particles the process of immobilization in a lattice state with significant free surfaces. The noticeable point of the image is the free surface part of

the particles and the possibility of photocatalytic reaction while they are bound to the cement matrix. This leads to the permanent operation of the particles while maintaining them in the matrix, the composite surface, which greatly saves from the heavy costs of separation and processing for reuse in slurry reactors.

Economic Perspective

So far, all studied reactors have been designed and tested based on relatively expensive fixed beds with very high energy consumption. In addition, the photocatalysts used are of the nano type or commercial types with relatively high cost for production and have poor performance according to the equations presented in this study. The reactor studied in this research, while using abundant materials (cement composites) as a substrate, uses the industrial type of titanium dioxide, and the technology used in the reactor leads to the denitrification with very low energy consumption rates and high efficiency yields. Therefore, future research based on the present study can lead to the industrialization of photocatalytic water treatment if the optimization process of reactor surface and specifications of the reactor improves.

CONCLUSION

According to the results of nitrate removal obtained from the performance of different specimens in photoreactor with different percentages of photocatalyst and evaluation of internal wall acid etching, the following results are inferred:

- The cement substrate with immobilized photocatalyst can be a cost-effective option for nitrate removal industrial systems compared to the results obtained by other researchers due to its much lower manufacturing and maintenance costs.
- Given the performance of TiO_2 alongside the amorphous silica particles trapped in the cement matrix, the chain between particles inside the CSH gel is provided and the independence from doping can be considered as a vantage point.
- Increasing the contact surface of photocatalyst has a significant effect on the efficiency of nitrate removal rate.
- Creating an active surface led to an increase in efficiency and results in higher denitrification rates compared to similar processes.
- The addition of nano-TiO_2 leads to a significant improvement in the efficiency of nitrate removal rate, and due to the low percentage of its consumption compared to industrial TiO_2, an economic justification can be considered for large scale applications.
- Two new equations introduced in this paper considering important parameters which have been neglected during the performance evaluation of reactors in order to measure the viability of these reactors (not the process itself) in large-scale applications such as initial volume of water and energy demand for treatment.
- Lower removal rate doesn't necessarily mean lower reactor performance due to shedding light on the impact of important parameters.
- The comparison of the results of this study with others reports indicated an important analysis on the performance of different types of reactors and the requirements for successful industrial applications.
- Microstructural images show the bond of TiO_2 particles in the cement matrix, which reveals the role of silica particles and the CSH matrix in stabilizing the position of TiO_2 crystals and keeping their activity in the cement substrate.

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The present research did not receive any financial support.

CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/or falsification, double publication and/or submission, and redundancy has been completely observed by the authors.

LIFE SCIENCE REPORTING

In addition, no life science threat was practiced in this research.

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