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Assessing Photocatalytic Degradation in High-Performance Concrete Mixes Using Fourier Transform Infrared Analysis and Response Surface Modelling

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Photocatalytic degradation (PD) is one of the solutions to overcome concrete degradation, which uses additives, such as titanium dioxide (TiO_2) and zinc oxide (ZnO), to break down pollutants when exposed to light. Fourier Transform Infrared (FTIR) analysis aids in the understanding of how these additives change chemical bonds, while Response Surface Modeling (RSM) predicts their effects on concrete mixes. This study aims to offer an insight into the photocatalytic behaviour of concrete under ultraviolet (UV) radiation by combining FTIR analysis and RSM. Photocatalytic properties were evaluated by assessing the degradation of Rhodamine B dye on concrete surfaces. A total of 21 compositions of TiO_2 and ZnO nanoparticles were developed using RSM. PD values, which range from 27 to 55%, indicate the self-cleaning ability of the concrete under UV light after 100 hours of exposure. FTIR analysis of high-performance concrete mixes showed similarities in peaks at around 1,000 cm^{-1}, which are associated with the presence of silica in the calcium-silicate-hydrate gel that is crucial for strength development during hydration. Differences in transmittance levels among the samples aided in gaining a clearer insight into the photocatalytic behaviour of the concrete. An analysis of variance revealed that a cubic model effectively characterizes the relationship between PD and TiO_2 and ZnO contents with a high R^2 value of 0.8353 and an F-value of 6.20, hence verifying the significance and accuracy of the model in representing the relationship between the variables.

Keywords: Photocatalytic degradation; Fourier Transform Infrared analysis; Response Surface Modeling; high-performance concrete; self-cleaning

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Concrete is commonly used in construction because it is strong and versatile. However, it can deteriorate over time due to various factors, such as pollution and microbes [1]. One way to combat this is photocatalytic degradation (PD), which uses materials like titanium dioxide (TiO_2) or zinc oxide (ZnO) to break down pollutants on concrete when exposed to light. These materials create reactive substances that break down pollutants into less harmful molecules [2]. Meanwhile, Fourier Transform Infrared (FTIR) analysis is a method to study chemical bonds in a sample by measuring how it absorbs infrared radiation [3]. By adding TiO_2 and ZnO to concrete, FTIR analysis helps understand how these additives change the chemical bonds. Response Surface Modeling (RSM) predicts how TiO_2 and ZnO affect concrete mixes. Combining FTIR and RSM can give a full picture of how concrete reacts to ultraviolet (UV) light [4].

By combining FTIR analysis and RSM, this study endeavours to offer a thorough insight into the photocatalytic behaviours displayed by concrete under UV radiation. Understanding these behaviours is crucial for progressing initiatives aimed at improving the self-cleansing properties of high-performance concrete (HPC) formulations. However, it is essential to acknowledge a limitation inherent in this study, arising from the assumption that all other components within the concrete remain consistent, thus primarily attributing observed effects to variations in TiO_2 and ZnO presence.

Literature Review

HPC is a major improvement over regular mixes, offering better strength and durability [5-7]. It is tailored for specific needs and used in critical structures where strength and longevity matter. One innovative method is adding photocatalytic materials like

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nano-structured TiO_2 and ZnO to the concrete. This enhances self-cleaning properties when exposed to sunlight, reducing the need for frequent maintenance [2, 8-9]. This self-cleaning process works naturally with sunlight, making it environmentally friendly and aligning with sustainability goals. Moreover, besides cleaning, these materials can help break down air pollutants like nitrogen oxides (NO_x) and volatile organic compounds (VOCs), potentially improving urban air quality and promoting healthier living environments [10].

Among photocatalytic materials, TiO_2 stands out for its remarkable photocatalytic capabilities, particularly when exposed to UV light. Adding TiO_2 nanoparticles to concrete greatly enhances its self-cleaning and pollutant-reducing properties, as shown by recent research. Studies conducted in 2020 and 2023 demonstrate that TiO_2 effectively cleans surfaces, separates oil and water, and maintains strong photocatalytic activity even after prolonged sunlight exposure [11-13]. Research by Guo et al. found that concrete with nano-TiO_2, applied using the spraying method, achieved a maximum PD efficiency of 73.82% with a 10mg/L concentration of sprayed nano-TiO_2 slurry [14]. Interestingly, unpolished nano-TiO_2 concrete performed better than polished versions under the same UV exposure. In another study by Wang et al., the degradation of methyl orange surpassed 98% after 40 minutes of UV exposure, and nitrate concentration measurements indirectly confirmed nitrogen oxide breakdown [15]. Additionally, research by Kim and Hong revealed that concrete containing 7.5% TiO_2 achieved an impressive 77.5% reduction in NO_x levels [16]. These findings highlight how TiO_2-incorporated concrete enhances self-cleaning and reduces pollutants, offering promising solutions for sustainable infrastructure development. In addition to the self-cleaning abilities of TiO_2, extensive research has also focused on its ability to enhance the strength of concrete, presenting an opportunity for development of sustainable concrete in diverse applications [17-18].

MATERIALS AND METHODS

Materials

This study utilized various materials to investigate concrete technology advancements comprehensively. Grade 52.5N Ordinary Portland Cement (OPC) served as the main binding agent, while titanium oxide nanoparticles (TiO_2 NPs) and zinc oxide nanoparticles (ZnO NPs) were added for self-cleaning, photocatalytic properties and potential mechanical enhancements or antibacterial effects, respectively. Coarse and fine aggregates provided bulk and stability, contributing to strength and volume. A superplasticizer improved workability without compromising strength by reducing the water-to-cement ratio [19]. Water activated the cement and facilitated necessary chemical reactions for concrete formation. The quantities, densities (specific gravities) and particle sizes of these materials were carefully measured and standardized to ensure accurate mix design and reliable experimental results. Rhodamine B dye was uniformly applied to the concrete surface for experimental purposes, such as tracing flow paths or studying surface characteristics. Through this diverse material integration, the study aimed to deepen our understanding of concrete behaviour and potentially introduce innovative applications in construction and beyond. Further details on material quantities, specific gravities, particle sizes and standard requirements are provided in Table 1.

Table 1. Material properties.

HPC components	Quantity	Specific Gravity	Range of size	Source
Cement	498 kg/m^3	3.15	< 150μm	Hume Cement
Fine Aggregates	667 kg/m^3	2.69	0.60–2.60 mm	River washed
Coarse Aggregates	1000 kg/m^3	2.76	4.75–19.0 mm	Quarry
Silica Fume	100 kg/m^3	2.25	< 0.8 mm	-
Water	144 kg/m^3	1.0	-	Potable Water
Superplasticizer	0.3 litre	1.04	-	Sika-Viscocrete 2088
TiO_2	0–2*	-	40 nm	US Research Nanomaterials, Inc.
ZnO	0–2	-	35–45 nm	US Research Nanomaterials, Inc.

******% by weight of cement*

Photocatalytic Degradation (PD)

Evaluation of the photocatalytic properties of concrete samples was conducted by assessing the degradation of Rhodamine B (RhB) dye applied to their surfaces. To initiate the degradation process, the samples were subjected to UV rays generated by a laminar flow system, with an intensity conforming to the UNI 11259 standard, set at 2 mW/cm^2. These concrete specimens, each measuring 100 mm in diameter and 50 mm in thickness, were formulated with varying combinations of TiO_2 and ZnO nanoparticles. Following exposure to UV radiation for a duration of 100 hours, the degree of colour degradation was quantified using a handheld spectrophotometer, specifically the Konica CR-400 Chroma Meter. The extent of PD was determined utilizing Equation 1, as specified in the study protocol. This systematic approach allowed for the precise evaluation of the photocatalytic efficiency of the concrete samples under investigation.

$$PD = \frac{[c^*_n - c^*_0]}{[c^*_d - c^*_0]} \times 100\% \qquad \text{Equation (1)}$$

Where, PD = Photocatalytic degradation at the nth hour of radiation, c^*_n= Intensity of specimen with RhB spray at the nth hour of radiation, c^*_d = Intensity of specimen after RhB spray but before UV radiation, c^*_0 = Intensity of the specimen without RhB.

Fourier Transform Infrared (FTIR)

FTIR spectra were acquired from the concrete samples using an FTIR spectrometer, specifically the Nicolet iS10 model, equipped with a diamond attenuated total reflectance accessory. This analytical technique enabled the identification of functional groups present in the hydration phases of the concrete, which could potentially influence the photocatalytic properties of the material when exposed to UV radiation. FTIR spectra offer valuable insights into changes occurring in silicate, sulphate, hydroxide and carbonate phases during the hydration process of the concrete.

To conduct the analysis, specimens containing various combinations of TiO_2 and ZnO were pulverized to obtain finely ground powders. These powders were then directly placed onto the diamond lens of the spectrometer for measurement. By utilizing FTIR spectroscopy, the chemical composition and structural changes within the concrete samples at a molecular level were able to be characterized. This information facilitated a deeper understanding of the interactions between the nanoparticle additives and the concrete matrix, providing crucial insights into the mechanisms underlying the observed photocatalytic properties.

Evaluating the Interactive Effects of TiO_2 and ZnO Nanoparticles on the Photocatalytic Properties

The interactive effect of TiO_2 and ZnO nanoparticles on concrete behaviour was meticulously analysed using RSM. This statistical technique enabled the systematic exploration of the combined influence of TiO_2 and ZnO nanoparticles on various properties of the concrete [9]. A total of twenty-one (21) different compositions of TiO_2 and ZnO nanoparticles were meticulously developed as outlined in Table 2. These compositions were strategically designed to replace the cement content in the HPC mix, thereby introducing variations in nanoparticle concentrations. The diverse array of TiO_2 and ZnO nanoparticle combinations employed in this study aimed to comprehensively capture all potential influences on concrete behaviour, facilitating a thorough analysis of their interactive effects. Moreover, as a reference point for comparison, HPC formulations without any cement replacement were prepared and utilized as control specimens. These control samples served as benchmarks against which the performance of the nanoparticle-modified concrete compositions was evaluated.

RESULTS AND DISCUSSION

PD of Concrete Samples

Figure 1 shows the PD of concrete samples after 100 hours of UV radiation exposure. PD values, ranging from 27 to 55%, indicate self-cleaning ability of the concrete under UV light. Understanding the relationship between PD values and self-cleansing ability is crucial for evaluating the effectiveness of photocatalytic materials in degrading contaminants [2, 20]. Higher PD values suggest lower self-cleansing ability, indicating more contaminants like RhB remain on the concrete surface after UV exposure. Conversely, lower PD values are preferred as they indicate better self-cleansing capacity. In simpler terms, lower PD values mean less RhB or other contaminants remain on the concrete surface after UV exposure, demonstrating improved self-cleaning ability [2], [20]. In this study, all concrete samples with TiO_2 and ZnO nanoparticles combinations showed lower PD values compared to the control group.

Notably, the concrete sample labelled as HPC 20, featuring the maximum combination of TiO_2 and ZnO nanoparticles, exhibited the highest reduction in PD. This observation underscores the synergistic photocatalytic effects of both nanoparticles when incorporated into the concrete matrix

Table 2. Percentage composition of nanoparticles replaced with cement in HPC.

Sample Description	Space Type	Percentage of ZnO in HPC (%)	Percentage of TiO_2 in HPC (%)
Control	Vertex	0	0
HPC 1	Third Edge	0.67	0
HPC 2	Centre Edge	1	0
HPC 3	Third Edge	1.33	0
HPC 4	Vertex	2	0
HPC 5	Axial CB	0.5	0.5
HPC 6	Axial CB	1.5	0.5
HPC 7	Third Edge	0	0.67
HPC 8	Third Edge	2	0.67
HPC 9	Centre Edge	0	1
HPC 10	Centre	1	1
HPC 11	Centre Edge	2	1
HPC 12	Third Edge	0	1.33
HPC 13	Third Edge	2	1.33
HPC 14	Axial CB	0.5	1.5
HPC 15	Axial CB	1.5	1.5
HPC 16	Vertex	0	2
HPC 17	Third Edge	0.67	2
HPC 18	Centre Edge	1	2
HPC 19	Third Edge	1.33	2
HPC 20	Vertex	2	2

Meanwhile, samples containing higher TiO_2 contents (HPC 9, HPC 12, and HPC 16) demonstrated lower PD values compared to those with equivalent amounts of ZnO (HPC 1–4). This suggests that TiO_2 nanoparticles contribute more significantly to the enhancement of photocatalytic behaviour in concrete compared to ZnO nanoparticles. Overall, the results highlight the potential of TiO_2-based nanocomposites in significantly improving the self-cleansing properties of concrete materials under UV exposure, offering promising prospects for the development of more sustainable and durable construction materials.

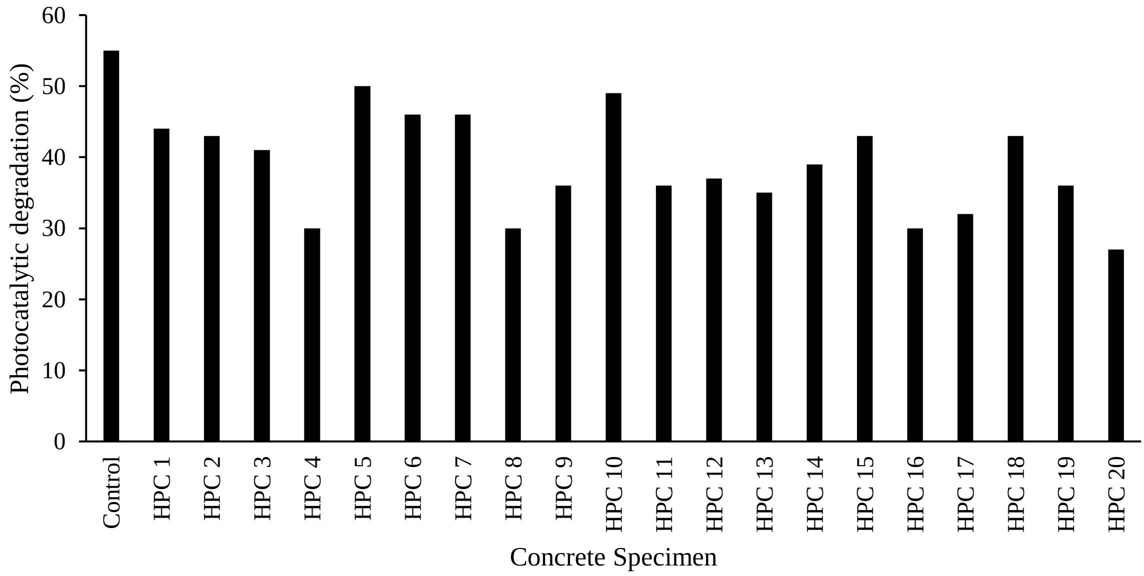

Figure 1. Photocatalytic degradation (PD) of concrete samples with TiO_2 and ZnO after 100 hrs of UV irradiation.

FTIR Evaluation of HPC Mixes

The FTIR spectra provided valuable insights into the impact of various combinations of TiO_2 and ZnO nanoparticles on the characteristics of HPC, as presented in Figures 2–5. Initially, the spectra revealed the presence of Si-O-Si stretching in the fingerprint region (500–1500 cm^{-1}), indicating the development of quasi-amorphous calcium-silicate-hydrate (C-S-H), a crucial component in concrete chemistry. Additionally, absorption peaks were observed at specific wavenumbers, such as 3483, 980 and 862 cm^{-1}, associated with the stretching vibrations of hydroxyl groups (-OH), carbon-hydrogen bonds (C-H), and carbon-oxygen-carbon bonds (C-O-C), respectively. These vibrations provided insights into the molecular structure and composition of the concrete [21].

Furthermore, infrared vibrations within the wavenumber range of 3630–3640 cm^{-1} indicated nonhydrogen-bonded stretching of the O-H bonds, attributed to the presence of calcium hydroxide, $Ca(OH)_2$. Additionally, the stretching of the H-O-H bond within the spectral range of 3240–3680 cm^{-1} was linked to adsorbed water and by-products resulting from the synthesis of tricalcium silicate (C_3S) and dicalcium silicate (C_2S) [21]. Notably, Figures 2 and 3 showed a decrease in the intensities of these bands with increasing concentrations of TiO_2 and ZnO, implying a decline in the quantity of free water due to the introduction of nanoparticles.

In contrast, for samples with different combinations of TiO_2 and ZnO (Figures 4 and 5), a broader H-O-H stretching was observed, suggesting the presence of hydrate (H_2O) and hydroxyl (-OH) in the range of 3650 and 3250 cm^{-1} of a broad absorption band [22]. Additionally, IR absorption bands corresponding to O-H bending were detected at around 1636 cm^{-1}, while peaks at approximately 1458 and 980 cm^{-1} were attributed to the carbonization process of $Ca(OH)_2$ in the presence of ambient carbon monoxide. Peaks within the range of 1000 cm^{-1} were associated with silica in the network of C-S-H gel, which is crucial for strength development during hydration.

Despite differences in nanoparticle composition, the FTIR spectra of all samples showed similarities in peak morphology and absorption bands, indicating similarities in the molecular composition of the concrete. The slight variations in transmittance levels across Figures 2–5 could be attributed to factors such as the limited amount of nanoparticle substitution within the concrete mix. The slight variation in transmittance indicates the differences in how the nanoparticle make-up of each concrete will affect its ability to break down pollutants when exposed to a uniform light source. Since each concrete sample has a unique composition, they will respond differently to light of the same wavelength thus impacting their effectiveness in breaking down pollutants. Although the concrete mixtures differed in composition, the hydration process generally follows a similar mechanism in most cases. This consistency in hydration could influence the photocatalytic properties of the concrete, resulting in resemblances in peak morphology and absorption bands observed in the FTIR spectra [22]. In summary, FTIR analysis provided detailed insights into the molecular composition and characteristics of HPC with various combinations of TiO_2 and ZnO nanoparticles.

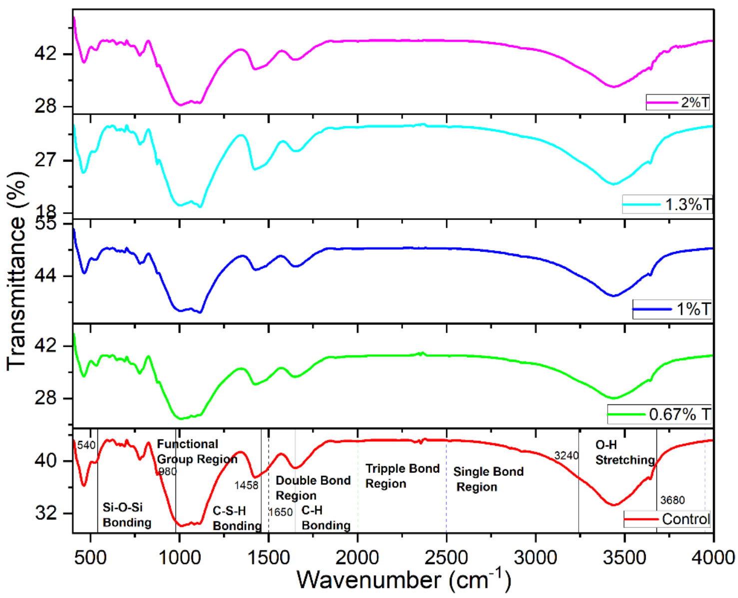

Figure 2. FTIR spectra of HPC with varying content of TiO_2.

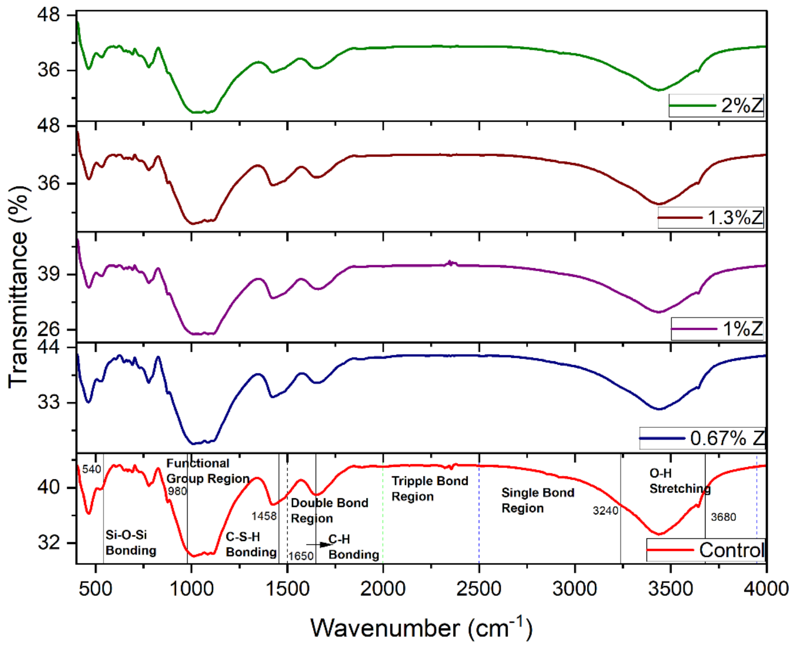

Figure 3. FTIR spectra of HPC with varying content of ZnO.

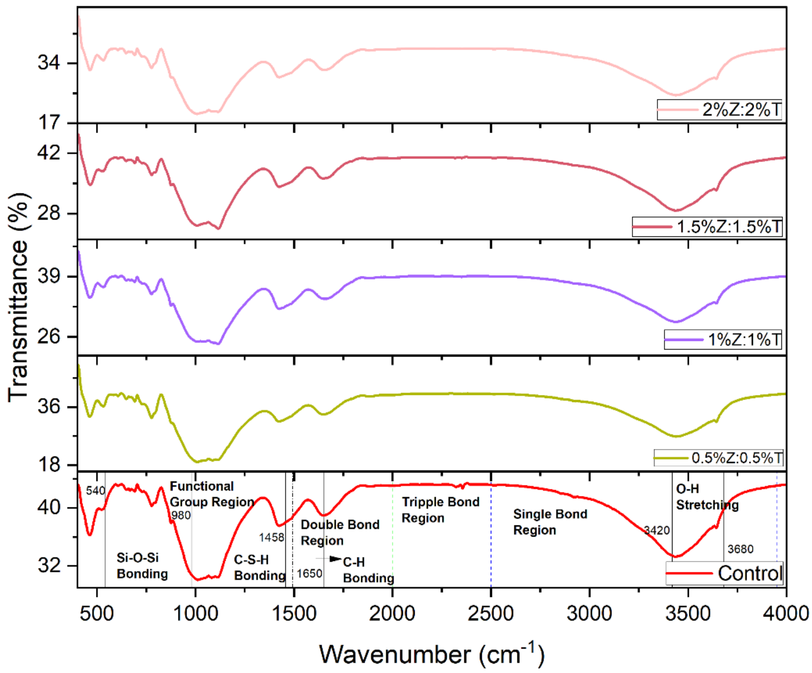

Figure 4. FTIR spectra of HPC with equal combinations of TiO_2 and ZnO contents.

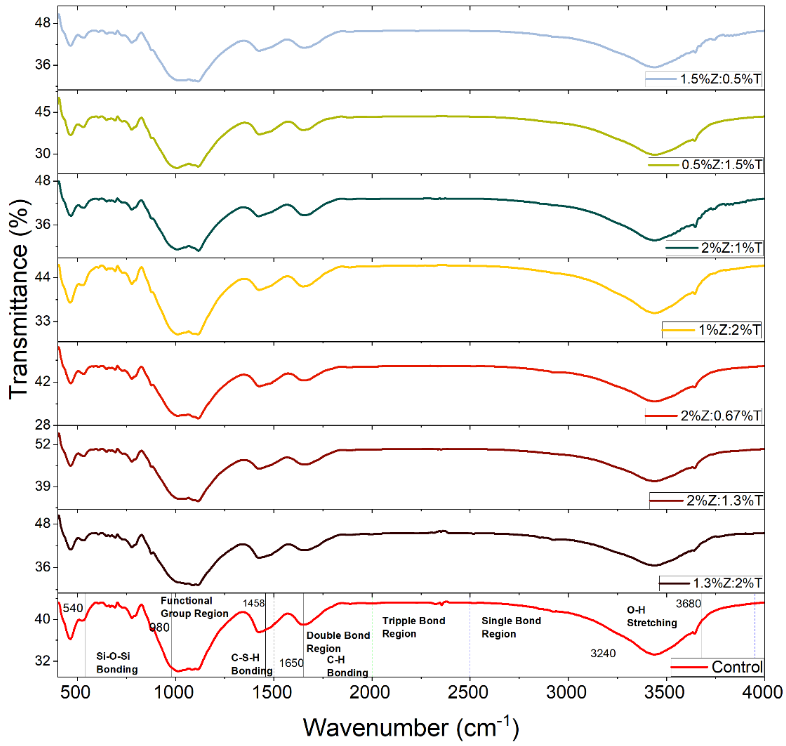

Figure 5. FTIR spectra of HPC with varied combinations of TiO_2 and ZnO contents.

Interactive Effect of TiO_2 and ZnO on PD

When studying the combined effects of TiO_2 and ZnO on the PD of HPC samples, interesting findings emerged. Initially, with low TiO_2 concentration (0%) and increasing ZnO in the samples, there was a gradual decrease in concrete degradation efficiency. This suggested that ZnO influenced the photocatalytic properties, as shown in the declining degradation efficiency shown by the black line in Figure 6. Conversely, with TiO_2 at its highest level (2%) and minimal ZnO in the concrete (as shown by the red line in Figure 6), there was a significant and sudden drop in PD. This indicated that TiO_2 had a more substantial effect on the photocatalytic properties of the concrete samples compared to ZnO.

Meanwhile, with maximum TiO_2 content and increasing levels of ZnO, there was an observed increase in the PD coefficient of the concrete samples. This rise suggested a reduction in the efficiency of the concrete under UV radiation, highlighting the complex interplay between TiO_2 and ZnO in influencing the photocatalytic behaviour of the material [11-16]. This intricate relationship is depicted in the 3D surface plot in Figure 7, which illustrates the dynamic interaction between TiO_2 and ZnO concentrations and their effects on PD efficiency. These findings underscore the importance of carefully balancing the concentrations of TiO_2 and ZnO in HPC formulations to optimize photocatalytic performance.

Analysis of Variance (ANOVA) findings revealed that a cubic model effectively characterizes the relationship between PD and the corresponding contents of TiO_2 and ZnO in the concrete, with a high coefficient of determination (R^2) value of 0.8353. The model is F-value of 6.20, as shown in Table 3, indicates its significance and ability to accurately represent the relationship between the variables. This suggests that the cubic model provides a reliable framework for predicting the influence of both nanoparticles on the photocatalytic properties of concrete within the range of combinations studied in this research. The cubic model, expressed as Equation 2, can be employed to predict PD based on the concentrations of TiO_2 and ZnO.

The predictive efficacy of the model is illustrated in Figure 8, which demonstrates how well the model fits the experimental data points. Additionally, the probability of obtaining an F-value of this magnitude solely due to noise is calculated to be a mere 0.32%, further supporting the validity and reliability of the model. In conclusion, the cubic model provides a robust framework for understanding and predicting the relationship between TiO_2 and ZnO concentrations and PD in concrete. This information can be valuable for optimizing concrete formulations and designing materials with enhanced photocatalytic properties for various applications.

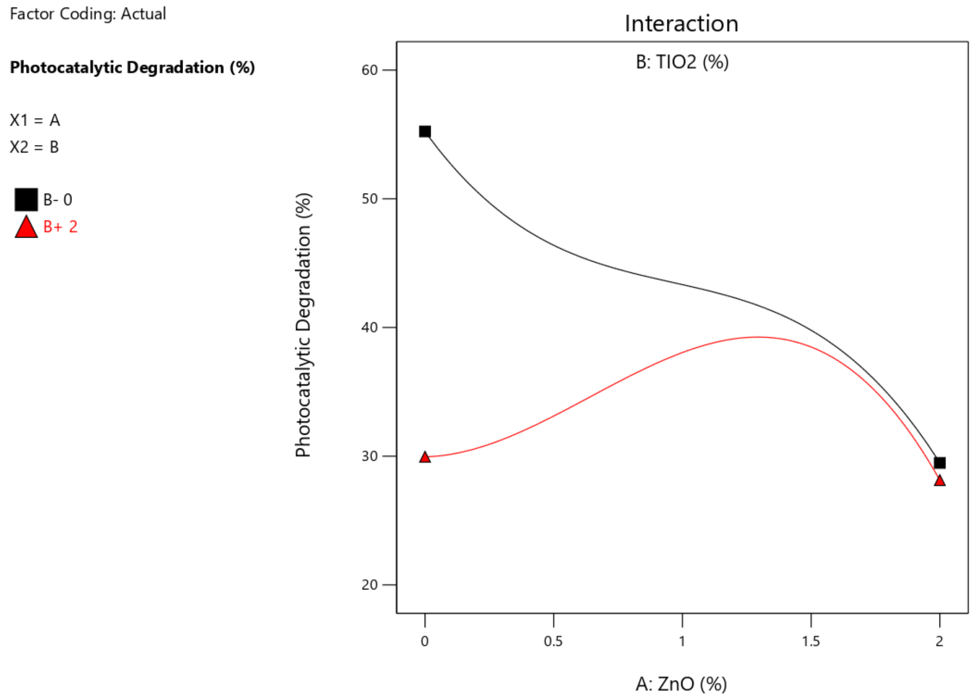

Figure 6. Interactive effect of TiO_2 and ZnO on PD.

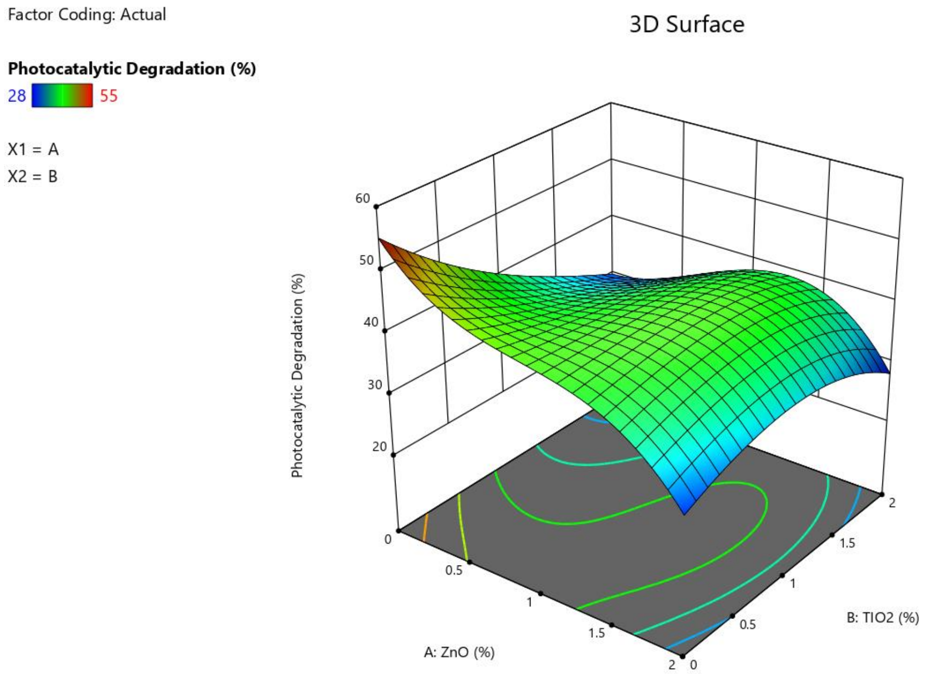

Figure 7. 3D surface plot of the effect of TiO_2 and ZnO on PD.

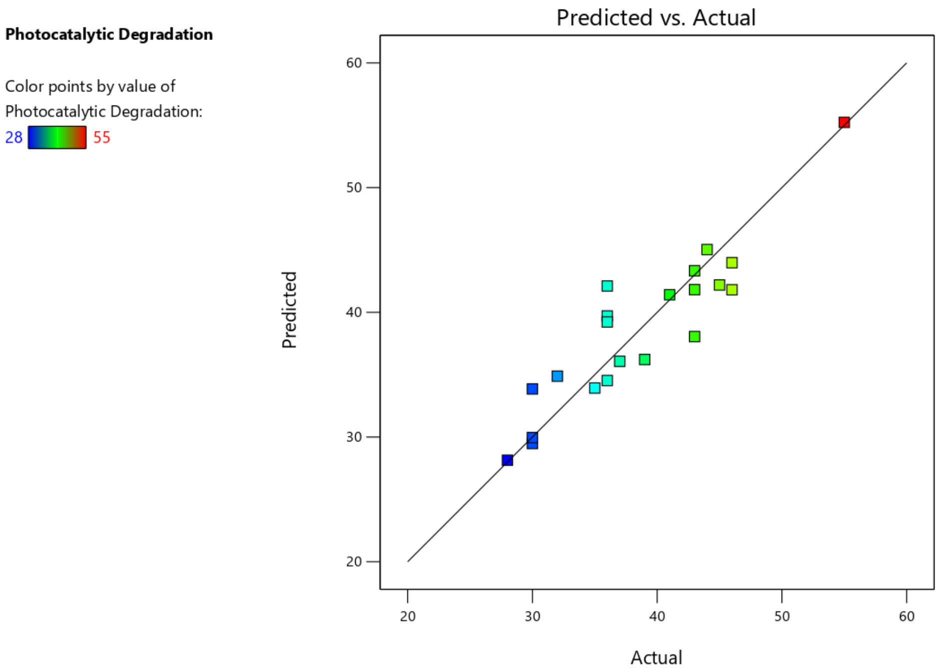

Figure 8. Plot of predicted PD against actual degradation.

$$PD = 42.11 + 5.78\,A - 1.76\,B + 5.98AB - 4.99A^2 - 1.42B^2 - 4.02A^2B - 4.30AB^2 - 8.37A^3 0.8766B^3 \quad \text{Equation (2)}$$

Where, PD = Photocatalytic degradation, A = ZnO content, B = TiO_2 content

CONCLUSION

This study offers a thorough understanding of how concrete reacts under UV radiation regarding photocatalytic activity. PD values ranging from 27 to 55% reflect the concrete's self-cleaning ability when exposed to UV light. Additionally, FTIR spectra analysis provides valuable insights into how different combinations of TiO_2 and ZnO nanoparticles affect HPC characteristics. Similarities in peak morphology and absorption bands across all samples suggest resemblances in the molecular composition of the concrete. However, the slight differences in transmittance levels helps to better understand the photocatalytic behaviour of the concrete samples under a uniform light source. The evaluation of the interactive effect of TiO_2 and ZnO on HPC PD yields intriguing insights, emphasizing the importance of adjusting their concentrations for optimal photocatalytic performance. ANOVA demonstrates that a cubic model effectively captures the relationship between PD and TiO_2/ZnO contents, with a high R^2 value of 0.8353 indicating a strong fit between experimental data and the model. Thus, further investigation and refinement of nanoparticle-concrete interactions will drive innovation in developing high-performance, environmentally friendly building materials.

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