Zeolite-Loaded Titanium Dioxide Photocatalytic Cement-Based Materials for Efficient Degradation of Drinking Water Disinfection Byproduct Trichloroacetamide

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A two-step method was used to load TiO₂ on a cement matrix, and zeolite was chosen as intermediate support. TiO₂@Zeolite composite coated photocatalytic cement-based material (PCM) was prepared. Some advanced characterization technologies including X-ray diffraction (XRD), scanning electronic microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), and BET specific surface area (SSA) test were applied to characterize the physicochemical properties of as-prepared PCM. Photocatalytic degradation of trichloroacetamide (TCAcAm) was conducted to evaluate its photocatalytic efficiency. Results show that TiO₂@Zeolite composite can improve the adsorption ability of PCM and TiO₂ particles were dispersed on the surface of PCM homogeneously providing abundant active sites for photocatalytic reactions. Moreover, TiO₂@Zeolite composite can reduce the negative effect of cement on TiO₂. The synergetic effect of TiO₂@Zeolite composite can remarkably improve the photocatalytic degradation rate, reaching up to 97.8%. TiO₂@Zeolite composite coated PCM holds great promise to eliminate water pollution.

Keywords: TiO₂@Zeolite composite, photocatalytic cement-based material, water purification, photocatalytic degradation, disinfection byproducts

INTRODUCTION

Nowadays, water pollution is a big environmental health concern worldwide as multiple harmful substances such as antibiotics, microbes, and dyestuffs are detected in drinking water. It is urgent to eliminate these contaminants in aqueous environments to guarantee water safety (Srogi, 2007; Ahmed et al., 2010; Zangeneh et al., 2015; Liu et al., 2017). At present, chlorine disinfection is still the main method used to disinfect tap water, since it was first applied in England in 1897, due to high disinfection efficiency, low cost, and facile implementation (Hom, 1972; Winward et al., 2008). However, some carbon-disinfection by-products (C-DBPs) will be generated in the disinfection process, which can induce many diseases like cancer (Chu et al., 2012; Chang et al., 2013). Recently, some new disinfectors such as chloramine and ozone have been developed, but...
the derived nitrogen-disinfection by-products (N-DBPs) are proved to be more toxic than C-DBPs (Chu et al., 2012). Moreover, tap water is usually transported over a long distance through water supply pipes, and then stored in concrete-made water storage structures before being delivered to households, and as a result N-DBPs will concentrate and bacteria will breed fast at the end of the network (Whelton et al., 2015). Consequently, a novel strategy should be proposed to deal with these emerging challenges.

Since Fujishima and Honda found the splitting of water on a TiO$_2$ anode under illumination, TiO$_2$ has been widely studied and used in environment remediation owing to its strong photo-induced oxidation ability (Fujishima and Honda, 1972; Vinu and Madras, 2012; Wen et al., 2015; Pawar et al., 2018). In detail, active oxygen species (AOS) generated on TiO$_2$ like hydroxyl radical (-OH) can totally oxidize pollutants into water and carbon dioxide upon solar irradiation. Previous research has proved that heterogeneous photocatalysis exhibits specific superiority in eliminating aqueous pollutants in terms of low cost and harmless byproducts, when compared to conventional disinfection (Yoneyama and Torimoto, 2000; Karuppuchamy et al., 2007; Fuchs et al., 2009; Yamaguchi et al., 2010). Some pioneering works about using TiO$_2$ to purify water have been reported, which show high degradation efficiency toward pollutants. However, in most reported cases, TiO$_2$ has been employed in the form of nano-powder which is prone to agglomerate in aqueous environments due to its high surface energy, thus compromising its photocatalytic efficiency, and it is also difficult to recollect and reuse this powder TiO$_2$ (Zhang et al., 2003; Negishi et al., 2019a,b). In recent years, immobilization of TiO$_2$ on some support materials is promising to solve this problem, and among all support materials, cementitious material is a suitable one because of its strong binding property and chemical inertness (Chen and Poon, 2009; Jimenez-Relinque et al., 2015). So far, TiO$_2$-engineered photocatalytic cement-based material (PCM) has already been fabricated and applied in environment remediation such as depollution of NO$_x$ (Seo and Yun, 2017), self-cleaning of building (Wang et al., 2014), and decomposition of volatile organic compounds (VOCs) (Alsa et al., 2011), but few works about the application of TiO$_2$-incorporated PCM in water purification have been reported.

As most water storage constructions are manufactured of cement-based materials, it is attractive to coat TiO$_2$ on these cement constructions to further improve drinking water quality. Although cement paste can immobilize TiO$_2$ particles firmly, its hydration products like C-S-H gel and Ca(OH)$_2$ can cover TiO$_2$, thus leading to a decline of photocatalytic activity (Chen et al., 2011). Due to the poor adsorption ability of cement matrix, only a small number of pollutant molecules will be captured by cement-based materials, which is not favorable for photocatalytic degradation reaction (Nazari and Riahi, 2011; Lee et al., 2013). So, it is of significance to reduce the negative effect of cement on photocatalysis for facilitating the application of TiO$_2$ coated PCM in water purification. In this paper, zeolite was selected as intermediate carrier, due to its high porosity and large specific surface area (SSA), and a two-step method was proposed to load TiO$_2$ on a cement matrix. Firstly, TiO$_2$ was dispersed and anchored on the surface of zeolite, forming TiO$_2$@zeolite composite. And then TiO$_2$@zeolite composite was coated on cement paste. Degradation of trichloroacetamide (TCAcAm) was conducted to evaluate the purification effect of the as-prepared TiO$_2$@zeolite composite coated PCM.

**EXPERIMENTAL**

**Raw Materials**

Conch brand P•O 42.5 cement was used in this study. Natural zeolite was produced in Henan province, China, and its chemical composition is shown in Table 1. All reagents including Tetrabutyl orthotitanate (TBOT), nitric acid (HNO$_3$), and absolute ethyl alcohol (C$_2$H$_5$OH) were provided by Sinopharm Chemical Reagent Co., Ltd., China.

**Sample Preparation**

**TiO$_2$@Zeolite Composite Fabrication**

Natural zeolite pre-treatment process: natural zeolite was washed by deionized water to eliminate impurities and heated in a 100°C drying oven for 1 h. Finally, treated zeolite was sieved by a standard sieve with a mesh number of 100. TiO$_2$ sol preparation process: 10 g TBOT was added into 80 g deionized water dropwise with continuous stirring, and 0.8 g HNO$_3$ was also incorporated to inhibit the fast hydrolysis of TBOT. After finishing adding TBOT, milky white suspension was obtained, and then thermally treated in a 40°C water bath for 24 h, thus gaining light blue TiO$_2$ sol. TiO$_2$ loading process: 1 g zeolite was added into a certain amount of TiO$_2$ sol, where TiO$_2$ accounts for 30% weight of zeolite. Then, the mixture was stirred for 1 h and followed with a 45-min ultrasonic treatment. Finally, the mixture was placed in a vacuum reactor with a pressure of 0.07 MPa for 5 h, and then heated in a muffle furnace for 4 h at different temperatures (200, 300, 400, and 500°C), obtaining TiO$_2$@zeolite composite. The specimens were labeled as n-TZ, where n stands for the final thermal treatment temperature and TZ represents TiO$_2$@zeolite composite, for instance, 200-TZ stands for the composite treated under 200°C. For comparison, pure TiO$_2$ sol was dried and heated at 200°C to obtain pure TiO$_2$ powder.

**TiO$_2$@Zeolite Composite Coated PCM Fabrication**

22 g cement and 8.8 g water were mixed for 5 min by a mixer, and the fresh cement paste was casted in a round mold with a diameter of 50 mm and a height of 8 mm. Before the hardening of the cement paste, a certain amount of TiO$_2$@zeolite composite was sprayed onto the surface of the cement paste, and demolded 1 day later. All specimens were cured in a standard curing chamber (20 ± 2°C, RH ≥ 95%) for 28 d. The samples were labeled as n-TZ-m-PCM, where n stands for the final thermal treatment temperature, m% stands for the mass ratio of

| SiO$_2$ | Al$_2$O$_3$ | Na$_2$O | CaO | MgO | Fe$_2$O$_3$ | FeO | TiO$_2$ | K$_2$O | P$_2$O$_5$ |
|--------|------------|--------|-----|-----|-------------|-----|---------|-------|---------|
| 60~70  | 17.8       | 4.2    | 2.6 | 0.8 | 1.6         | 1.2 | 0.6     | 3.2   | 0.26    |
TiO$_2$@zeolite composite to cement, TZ represents TiO$_2$@zeolite composite, and PCM represents PCM, for instance, 200-TZ-25-PCM represents that composite treated under 200°C was used and the composite to cement ratio was 25%. The schematic diagram of the TiO$_2$@zeolite composite coated PCM is shown in Figure 1.

**Physicochemical Properties**

**Characterization**

X-ray diffraction (XRD) was used to analyze the crystal phase of as-prepared specimens, and the analysis was conducted on an X-ray diffractometer equipped with a Cu Ka ray source. The diffraction angle ($2\theta$) ranged from 10° to 80° with the interval of 0.02° at the speed of 4° min$^{-1}$. Scanning electronic microscope (SEM) was applied to observe the micro-morphology of samples and energy dispersive X-ray spectroscopy (EDS) was adapted to investigate element distribution at a micro-area level. BET SSA test was used to characterize the pore structure of samples.

**Photocatalytic Degradation Efficiency**

**Characterization**

TCAcAm solution preparation process: 0.5 g TCAcAm solid was added into 1 L deionized water and mixed evenly, and then the solution was diluted 100 times and the final concentration of TCAcAm solution preparation process: 0.5 g TCAcAm solid was transferred into a 30 mL volumetric flask by using a large-capacity pipette. And then 20 mL TCAcAm solution and 4 mL of methyl tert-butyl ether (MTBE) extractant were added to a tube, and the mixture was vibrated for 2 min by a tube shaker and placed without stirring for 5 min. Gas chromatography-mass spectrometry (GC/MS) was used to measure the TCACAm in the upper extractant solution.

**RESULTS AND DISCUSSION**

**Microstructure of TiO$_2$@Zeolite Composite**

**XRD Analysis**

As depicted in Figure 2, characteristic diffraction peaks of anatase were observed at 20 (25.305°, 37.799°, 48.038°, 55.062°, and 62.690°) in the diffraction pattern of TiO$_2$ according to PDF#73-1764. This result illustrates that hydrolysis of TBOT and followed hydrothermal treatment can promote the formation of TiO$_2$, and TiO$_2$@zeolite composite based on this method will possess excellent photocatalytic activity. In the diffraction pattern of zeolite, characteristic peaks of quartz and K$_2$Na$_{0.06}$Al$_7$Si$_{17}$O$_{62}$.8O$_{20.7}$ were observed according to PDF#89-1629, indicating zeolite was symbiotic with quartz. For the 500-TZ sample, the main crystal phases were still quartz and K$_2$Na$_{0.06}$Al$_7$Si$_{17}$O$_{62}$.8O$_{20.7}$, suggesting that thermal treatment did not destroy the structure of zeolite. After thermal treatment, the intensity of quartz and K$_2$Na$_{0.06}$Al$_7$Si$_{17}$O$_{62}$.8O$_{20.7}$ became stronger, indicating that its purity was improved. A weak diffraction peak was detected at 20 (25.305°), which is the strongest diffraction peak of Anata, confirming that TiO$_2$ was successfully loaded on zeolite. Due to the small amount of TiO$_2$ loading, only one peak of anatase was observed. And there is another possibility that because the crystalline grain size of TiO$_2$ was too small, the anatase diffraction intensity was weak (Jansson et al., 2015).

**Micro-Morphology**

As shown in Figure 3a, zeolite is made up of plate-like units, forming a rough surface and some micron-sized slit-like pores. This structure contributes to the capture of TiO$_2$ particles when immersed in TiO$_2$ sol, thus leading to the homogenous

![Figure 1](https://example.com/fig1.png)  
**Figure 1** | Schematic diagram of the TiO$_2$@zeolite composite coated PCM.

![Figure 2](https://example.com/fig2.png)  
**Figure 2** | X-ray diffraction patterns of TiO$_2$, Zeolite, and 500-TZ.
distribution of TiO₂ particles on the surface of zeolite. Figure 3b depicts the morphology of pure TiO₂, after thermal treatment the nano-structure of TiO₂ is not observed and it presents a block-shape. This indicates that if nano-TiO₂ particles were not dispersed evenly and anchored firmly, they are prone to agglomerate, thus forming a densified structure and reducing its surface area. The rough surface of zeolite combined with the nano-structure of TiO₂ provides a possibility to synthesize the zeolite supported nano-TiO₂ composite (Ito et al., 2014; Maraschi et al., 2014). In Figure 3c, the surface of 500-TZ is coated with a flocculent layer and the edges and corners of zeolite gradually disappear after loading TiO₂, suggesting that porous TiO₂ had been mounted on the surface of zeolite. Figure 3d presents the primary elements distribution of TZ-500 (Si, O, Al, and Ti), Ti element is well dispersed on the surface of 500-TZ, further confirming the existence of TiO₂ and the homogeneous distribution of TiO₂. The unique structure of TZ-500 is attributed to TiO₂ modification. More specifically, in the TiO₂ sol, nano-TiO₂ particles existed stably due to the electrostatic repulsion. When heating TiO₂ sol, water was evaporated and surface charge was changed, and thus TiO₂ particles agglomerated. However, when zeolite was immersed in TiO₂ sol, the micro-nano structure of zeolite interacted with TiO₂ under the action of surface charge, nano-TiO₂ particles were fixed on the surface of zeolite, and zeolite provided a large surface to disperse TiO₂ particles. Upon heating, water was evaporated, but TiO₂ particles will not get into agglomeration due to the anchoring of zeolite (Easwaramoorthi and Natarajan, 2009).

**BET Analysis**

Figure 4A displays isotherm adsorption-desorption curves of as-prepared samples. Hysteresis loops were observed in all samples, confirming the existence of mesoporous structure (Sing et al., 1985), but the shape of isotherms was different, indicating that the pore structure and surface property were altered through thermal treatment under different temperatures. At low relative pressure (<0.5), the N₂ adsorption volume of 200-TZ was approximate to that of 300-TZ and 400-TZ, but higher than that of 500-TZ. This demonstrates that the adsorption ability of 200-TZ, 300-TZ, and 400-TZ was higher that of 500-TZ, which was also verified by the results of SSA in Figure 4C. In general, the higher the SSA is, the stronger the adsorption capacity is. It proves that thermal treatment can lead to an agglomeration of TiO₂, thus reducing its SSA, and the higher the temperature is, and more serious the agglomeration is. However, at high relative pressure (>0.5), the N₂ adsorption volume of 200-TZ was noticeably lower than that of 300-TZ, 400-TZ, and 500-TZ. This is probably caused by the pore size difference. As shown in Figure 4B, the most probable pore diameter shifted right with temperature, and 200-TZ had
the smallest pore diameter (about 3.5 nm). This phenomenon also verified the TiO$_2$ agglomeration resulted by thermal treatment.

**Photocatalytic Degradation of TCACAm**

It is well known that photocatalytic degradation contains three main processes: mass transfer, interphase adsorption, and photocatalytic oxidation. Figure 5 compares different samples in terms of adsorption rate toward TCACAm without light. The adsorption rate increased with time and stayed constant after 5 h. And the stable adsorption rate of 200-TZ-25-PCM reached up to about 50%, which was higher than that of zeolite-PCM and TiO$_2$-PCM. This can be explained by the fact that, although zeolite is porous and has good adsorption capacity, after loading TiO$_2$ its pore structure was modified, so the adsorption capacity of 200-TZ-25-PCM was enhanced. On the other hand, compared with TiO$_2$-PCM, 200-TZ-25-PCM can disperse TiO$_2$ more evenly on the surface of cement paste and provide more adsorption sites, thus leading to a higher adsorption rate.

Figure 6 displays the effect of calcination temperature on the photocatalytic degradation rate toward TCACAm. The degradation rate was the highest at 200°C (97.6%), and it gradually decreased as the calcination temperature increased. It shows that the prepared photocatalytic PCM has the greatest photocatalytic activity under the calcination condition of 200°C.

Because as the temperature increases, the size of the TiO$_2$@zeolite
composite particles became larger, resulting in the redshift of the optical energy gap, and the redox potential of electrons/holes became lower, and the activity of the catalyst decreased (Poon and Cheung, 2007).

Figure 7 shows the results of photodegradation of TCAcAm on PCM with different TiO$_2$ content after 5 h of light. Zeolite-25-PCM stands that the TiO$_2$ content was 0, and TiO$_2$-25-PCM stands that the TiO$_2$ content was 100%. It can be seen that the degradation rates of Zeolite-25-PCM and TiO$_2$-25-PCM were relatively low, reaching 40 and 34.2%, respectively. The degradation rate of Zeolite-25-PCM was almost equal to its adsorption rate, indicating that Zeolite-25-PCM can only adsorb TCAcAm under light, and TiO$_2$ particles will get into agglomeration in the cement hydration environment, so the degradation rate of TiO$_2$-25-PCM was the lowest (34.2%). At low TiO$_2$ addition, its photocatalytic degradation effect is poor and the adsorption governs the reaction, so the degradation rate of TiO$_2$-25-PCM was lower than that of Zeolite-25-PCM. Moreover, the degradation rate of 200-TZ-25-PCM was close to its adsorption rate as shown in Figure 5. With TiO$_2$ content increased, the degradation rate rose rapidly, and it reached its highest point (97.8%) as TiO$_2$@zeolite composite content was 40%. This was owned to two effects. Firstly, the abundant channels and pores, and the large SSAs of zeolite benefited the mass transfer and adsorption. Secondly, the zeolite was an optimal support for TiO$_2$ due to the unique pore shape, internal pore volume, and channel size (Hashimoto, 2003; Matsuoka and Anpo, 2003; Corma and Garcia, 2004), and TiO$_2$ was proved to be homogeneously dispersed on the surface of the TiO$_2$@zeolite composite as the results of N$_2$ adsorption-desorption and SEM-EDS indicated, thus more active sites were provided. The synergistic effect led to a high photocatalytic efficiency. It should be noticed that the degradation rate of 200-TZ-45-PCM was lower than 200-TZ-40-PCM. This can be explained by the fact that when the content of TiO$_2$@zeolite composite was high,
it could not be dispersed homogenously on the surface of cement and some active sites will be covered, so its photocatalytic degradation rate was lower even if it had higher TiO$_2$ content. The intermediate carrier method is effective to improve the photocatalytic performance of the PCM.

It can be seen from Figure 8 that the degradation rate of 200-TZ-40-PCM toward TCAcAm can reach 98.7 and 99.2% at 5 h under acidic (pH = 5) and alkaline conditions (pH = 11), respectively. It shows that in the process of photocatalytic degradation of TCAcAm, the initial pH has little effect on the degradation rate. The reasons for the above phenomenon may be as follows: the pH value of the solution is an important factor that determines the physical and chemical species in the solution and the state of the molecules in the solution. In the case of acidic initial conditions (pH = 5) and alkaline initial conditions (pH = 11), since the pKa value of TCAcAm is 8.75, when the pH value of the solution was lower than 9, TCAcAm existed in the form of C$_2$H$_2$Cl$_3$NO molecules, and as the pH value was lower than 8.75, it existed in the form of C$_2$HCl$_3$NO$^-$. Therefore, regardless of acidic or alkaline conditions, TiO$_2$ had no additional adsorption effect on TCAcAm due to the protonation of the charge on the TiO$_2$ surface, so acidity and alkalinity have no great effect on the photocatalytic degradation of TCAcAm.

The results of the recycle degradation experiment of 200-TZ-40-PCM are shown in Figure 9. The first degradation rate reached up to 98.6% for TCAcAm within 5 h. However, from the second cycle onward, the degradation rate decreased slightly. When the tests were recycled three times, the third degradation rate was 56.6%. The decrease in degradation rate is probably due to the occupation of TiO$_2$ active sites by TCAcAm or the reaction products.

**CONCLUSION**

(1) Through the sol-gel method, TiO$_2$@zeolite composite catalyst was successfully prepared. SEM images and BET results showed that zeolite was porous and had large SSA. XRD and EDS results confirmed that nano-TiO$_2$ particles were homogeneously dispersed on the surface of zeolite.

(2) TiO$_2$@zeolite composite coated PCM had excellent adsorption ability. The synergistic effect of the surface adsorption of zeolite and the photocatalytic degradation of TiO$_2$ can significantly improve the photocatalytic degradation performance of the TiO$_2$@zeolite composite coated PCM. 200-TZ-40-PCM (TiO$_2$@zeolite composite content was 40% and thermal treatment temperature was 200$^\circ$C) had the highest degradation rate (97.8%) within 5 h, which was about 2.86 times than pure TiO$_2$ coated PCM.

(3) The TCAcAm degradation rate was regardless of pH value, and decreased with thermal treatment temperature. After several repeated experiments, the degradation rate had a declining trend.

**DATA AVAILABILITY STATEMENT**

The original contributions presented in the study are included in the article-supplementary material, further inquiries can be directed to the corresponding author/s.

**AUTHOR CONTRIBUTIONS**

GL: literature search, figures, and writing. WC: data collection. JZ: data analysis. WY: study design. AS: funding and data interpretation. All authors contributed to the article and approved the submitted version.

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Conflict of Interest: JZ was employed by the company Shanghai Construction Group Co., Ltd., Shanghai, China.

The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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