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TiO₂ coatings synthesized by liquid flame spray and low temperature sol–gel technologies on autoclaved aerated concrete for air-purifying purposes



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ABSTRACT

This article reports for the first time in literature the successful application of two TiO₂ synthesis-coating technologies, i.e. low temperature sol–gel (LTS) and liquid flame spraying (LFS), to develop autoclaved aerated concrete with air-purifying properties. Significant differences in crystal and agglomerate sizes, specific surface areas and crystal phase compositions have been observed in the synthesized photocatalytic coatings. These were, however, not reflected in their respective air purification performance as indicated by toluene removal efficiencies around 60% (TOL inlet concentration of 14 ppm_v, 23 °C, 50% relative humidity and 3 min gas residence time) for both coating types. The different nano-scale characteristic effects indicate to compensate each other so that the overall photocatalytic activities as observed in our test set-up and conditions do not differ for both synthesis-coating technologies. The toluene elimination rates reported here (approx. 40 mg TOL·m⁻²·h⁻¹) are significantly higher than that reported in previous research using high temperature sol–gel techniques or commercial TiO₂ embedded in a cementitious matrix. Moreover, LTS and LFS technologies cannot only be easily applied on precast materials during manufacturing, they can also be applied on existing buildings. Based on these promising results and application potential, further research that systematically investigates the photocatalytic effect of different synthesis-coating parameters is highly encouraged to further improve the knowledge about these technologies.

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1. Introduction

The application of titanium dioxide photocatalysis on cementitious materials has received considerable attention in recent years as this technology can efficiently degrade organic (e.g. toluene) and inorganic (e.g. NO_x) air pollutants using light irradiation as the only energy source [1–6]. Furthermore, based on the hydrophilic properties produced by TiO_2 photocatalysis, self-cleaning or easy to clean properties have been also reported [7–12]. TiO_2 is a semiconductor material that when it is light irradiated with high energy photons (higher than its band gap — E_g) promotes electrons (e^-) from the valence band (VB — with electrons) to the conduction band (CB — without electrons). Due to the equilibrium in charges, this process also generates electronic holes (h^+) in the valence band. A portion of the generated electron-hole pairs reaches the surface of the semiconductor and initiates reduction–oxidation processes with adsorbed oxygen (O_2) and water (H_2O) molecules from the surrounding air. Then, reactive oxygen species (e.g. $\cdot\text{OH}$ and $\text{O}_2\cdot^-$) responsible for the oxidation of different organic and inorganic compounds as well as the hydrophilic effect are generated [13]. TiO_2 can crystallize in three different structures: rutile (tetragonal), anatase (tetragonal) and brookite (orthorhombic). Among these crystalline forms, rutile is thermodynamically the most stable while brookite and anatase transform to rutile under heating. Transformation of anatase to rutile is a broadly studied mechanism and occurs at temperatures between 550 °C and 1000 °C, while the less studied transformation of brookite to rutile has been reported between 550 °C and 600 °C [14,15]. The temperature of this transformation strongly depends on the impurities or dopants present in the material as well as on the morphology of the sample [16]. Differences among rutile, brookite and anatase crystals are in the density and hardness but the most important for photocatalytic applications is the difference between their band gaps. Anatase, brookite and rutile have band gaps of 3.19/388.7, 3.11/398.6 and 3.0/413.3 eV/nm, respectively [17]. So far, the use of TiO_2 as a mixed bulk additive in cementitious materials is the most common TiO_2 loading method. Nevertheless, the bulk addition of TiO_2 nanoparticles inside the cementitious matrix is not an ideal solution because of the limited accessibility of light and pollutants to most TiO_2 particles. Furthermore, previous research indicates that also cement carbonation decreases the photocatalytic activity in relation to atrazine degradation [18]. Similarly, also inactivation of the TiO_2 added in the mass has been reported in relation to degradation of toluene, probably due to the presence of hydrated products around the TiO_2 particles as they act as nucleation sites during cement hydration [19].

Therefore, the development of innovative TiO_2 loading methods such as through coatings is an interesting approach. As applied on top of the materials' surfaces, coatings could facilitate the access of pollutants (to be degraded) and photons (activators) to the catalyst. Furthermore, coatings offer not only the potential to apply the photocatalytic properties in a more efficient way, but also to widen the application possibilities to existing buildings or building

products. Numerous methods have been employed to achieve the synthesis of photocatalytic active TiO_2 nanopowders. However, not many synthesis technologies have been directly applied on cementitious materials to produce coatings. Promising technologies for this purpose are liquid flame spraying and sol–gel synthesis based on metal-alkoxide (M-OR) precursors [20–23]. In the liquid flame spraying technology, the liquid precursor is injected into a high temperature flame ($T_{\text{max}} \pm 3000$ K), where it evaporates and nucleates to nanosized particles [24]. In the sol–gel synthesis, metal alkoxides are dissolved into an organic solvent to be followed by reactions with water. Main reactions are hydrolysis and condensation. During hydrolysis, the alkoxide reacts with water producing M–OH bonds. Partially hydrolyzed molecules can link together in the following condensation reaction that occurs between OH groups and finally precipitate as nanosized particles [25]. Typical crystallization temperatures range from 350 to 450 °C in this technology [26].

In this research, nano- TiO_2 particle based coatings for air-purifying purposes have been directly synthesized on autoclaved aerated concrete for the first time using liquid flame spray and low temperature sol–gel synthesis technologies. A detailed characterization of the synthesized coatings from nano- to macro-scale has been performed. At nano-scale, crystal and agglomerate sizes, specific surface areas, crystal phase compositions, band gaps and crystallinity have been determined for synthesized nano- TiO_2 particles using both technologies. At macro-scale, the air cleaning potential of the coatings applied on autoclaved aerated concrete samples has been evaluated at lab-scale by monitoring toluene removal from air using a flow through operated photoreactor.

2. Materials and Methods

2.1. Autoclaved Aerated Concrete

High porosity of the substrate material leads to a positive effect in the degradation of toluene and nitrogen monoxide using TiO_2 photocatalytic cement based materials as reported by Maury Ramirez et al. and Poon and Cheung, respectively [2,6]. Therefore, a substrate that satisfies this demand is selected in this research. Autoclaved aerated concrete is a very porous and rough concrete. The open porosity of this material is previously determined on 4 samples by the vacuum water saturation method described in the ASTM C1202-10 [27] and amounts to $74.9 \pm 2.9\%$. The roughness of the material is evaluated by means of distance measurements using the automated laser measurement system (ALM) described in [28], from which the R_a factor presented in the BS 1134 is calculated [29]. In this case, eight profiles are obtained from each sample: 4 measurements in longitudinal direction (measurement length: 80 mm) and 4 measurements in transversal direction (measurement length: 60 mm). As a result, R_a , the arithmetical mean deviation of the profile from the center line reaches 70 ± 27 μm . For the air purification experiments, autoclaved aerated concrete samples are cut to prisms with dimensions 100 mm \times 80 mm \times 30 mm.

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