



Photocatalytic hydrophobic concrete coatings to combat air pollution



M. Faraldos^{a,b,*}, R. Kropp^a, M.A. Anderson^{a,c}, K. Sobolev^d

^a Water Science and Engineering Laboratory, University of Wisconsin–Madison, USA

^b Instituto de Catálisis y Petroleoquímica, CSIC, Spain

^c IMDEA Energía, Madrid, Spain

^d University of Wisconsin–Milwaukee, USA

ARTICLE INFO

Article history:

Received 24 March 2015

Received in revised form 27 May 2015

Accepted 15 July 2015

Available online 17 August 2015

Keywords:

Concrete

Hydrophobic

Photocatalysis

Self-cleaning

Air pollution

NO_x

Siloxane

ABSTRACT

Several low cost, user-friendly photocatalytic surface coatings were applied to cementitious surfaces and tested as a remediation technique for transportation NO_x emissions and organic pollutants. All of the sols (aqueous suspensions of nanoparticles) coatings showed very high nitrogen oxide (NO) conversion (~90%) and Methylene Blue dye bleaching when applied to concrete surfaces, even when the coatings were extremely thin or diluted, independently on the deposition method (spraying or dip coating). A novel hydrophobic coating based on photocatalyst nanoparticles suspended in a siloxane sealant showed similar conversion at low NO concentrations but required a greater loading of TiO₂. At high NO concentrations the hydrophobic coating was less effective, even at greater TiO₂ loadings, suggesting that the adsorption is the rate limiting step in this conversion process. A threshold of 1% TiO₂ loading in hydrophilic coating and 5% in the case of hydrophobic was needed to obtain a quantitative NO_x conversion. A home-made TiO₂ sol showed lower NO_x photodegradation but a more homogeneous and bonded layer.

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1. Introduction

Oxidative photocatalysis based on the semiconducting properties of titanium dioxide (TiO₂) has attracted enormous interest since it was discovered in 1972 [1,2]. However, it is only in the last decade that photocatalytically active materials have been applied to urban infrastructures [3]. The use of these photocatalysts can provide a wide range of beneficial properties to a surface, like self-cleaning [4,5], treatment of gas phase contamination in indoor and outdoor air environments [6–10], mainly with volatile organic compounds (VOCs), nitrous oxides (NO_x), sulfur oxides (SO_x), sooty particles, and reactive oxidative species (ROSs) [11,12].

Application of photocatalytically active material affects the surface area and surface energy of a structure or component and has several implications beyond its photo-oxidative property. Surfaces can be made to be more or less hydrophobic; profoundly different charge and energy, which has implications on the adhesion of coatings and subsequent surface treatments.

Recent advances in measurement techniques and methods have allowed the researcher to probe the oxidative effect of

these coatings at environmentally relevant conditions. Indoor air contamination removal requires understanding the behavior of the system at air concentrations across the range of parts per trillion (ppt_{v/v}) to potentially parts per thousand (v/v) [13–16]. Measurements of the concentration of given pollutants are more viable in a closed space, where the air environment is protected from the vagaries of wind direction and other weather, byproducts from reactions are contained, and the list of common contaminants is relatively fixed [4]. When these same photocatalytic materials are studied in outdoor environments a subtle shift in weather (wind speed, wind direction, atmospheric pressure, humidity, air pressure, etc.) can dramatically affect the contaminant load of the photocatalytic surface [17,18]. The number of potential contaminants is much higher, as outdoor air contamination comes from many sources (industrial, plant and animal sources, fires, dust, water surface aerosolization, etc.), each of which have different composition and concentration profiles. In addition to the greater number of potential contaminants, determining the relevant degradation pathway is more complicated, as byproducts are immediately diluted and confounding or competing reactions occur.

Nitrogen oxides (NO_x) are an important air-polluting agent with impacts on smog formation, ozone generation, and direct human health effects [19–21]. These compounds are produced during high temperature combustion, particularly in internal combustion engines in vehicles [22]. Recent research has shown that making

* Corresponding author at: Instituto de Catálisis y Petroleoquímica, CSIC, C/ Marie Curie, 2 L10. Madrid. Spain.

E-mail address: mfaraldos@icp.csic.es (M. Faraldos).

roadways photoactive through the application of TiO_2 is a promising approach for solving the problems caused by NO_x . The gas phase contaminant nitrous oxides are oxidized to nitrate ions at the concrete interface due to heterogeneous photocatalytic oxidation (PCO). The products of the reaction are in the form of water-soluble nitrate compounds and so can be washed from the active concrete surface by rain [23]. NO_x is typically present at sub-ppm concentrations in highly polluted rural air and even high levels of conversion to nitrates represent a small nitrate load on local water systems.

The incorporation of relatively low concentrations of titanium dioxide (TiO_2) in concrete formulation [24] and the use of the sunlight for the chemical conversion of nitrogen oxides [25,7] have become this technology an interesting alternative for urban construction. In addition, a few studies have shown that nano- TiO_2 can accelerate the early-age hydration of Portland cement, improve compressive and flexural strengths, and enhance the abrasion resistance of concrete [26]. Self-cleaning and “de-polluting” photocatalytic concrete products are already being produced by several companies in Europe and Japan for use in the facades of buildings and in paving materials for roads [27–29].

Improving air quality by producing photoactive infrastructure will require application of photoactive material to very large areas. In order to prove this treatment technology to be economically viable the cost of application and maintenance should be balanced against the removal or conversion of pollutants and the economic benefit to the public. Some studies have employed concrete blocks prepared by mixing TiO_2 particles with cementitious materials [30,31]. The amount of TiO_2 in these samples is very high and most of the catalyst is in the internal structure, where light is unavailable to promote reactions. Furthermore, mechanical properties of concrete are adversely affected by increasing concentrations of TiO_2 [32]. Other laboratory experiments have employed double layer blocks [17] [33], with only the upper layer having the photocatalyst in relatively high proportions. This preparation, requiring two steps, would ultimately result in increased production costs and some technical inconveniences such as blockage of the porous structure in the concrete and delamination of the photoactive facade. It is important to consider application procedures that are cost effective and these applications should not result in inferior mechanical, physical and chemical characteristics of the concrete [34]. While many contributions in the literature have been addressed to the effect of inlet gas flow, initial pollutant concentration, relative humidity, and irradiance, on the efficiency of photocatalytic degradation rates [17,35], little effort has been expended on optimizing the photocatalyst used, the method of its application or the homogeneity of distribution when preparing photoactive surfaces on concrete. In this sense, taking into account that photocatalytic reactions occur only at an illuminated surface, so an effective, sprayable photocatalytic coating material would be ideal.

On the other hand, siloxane sealers are typically applied to the surface of concrete to reduce ingress of harmful chemicals by creating hydrophobic conditions in the near surface areas, which combined with photocatalytic properties, could originate a new generation of materials offering better performance than conventional TiO_2 products.

2. Experimental procedure

2.1. Preparation of photocatalysts

Several different types of nano-particulate materials based on TiO_2 were used in this study: P25 powder from Degussa Evonik [36] (coded P), PC105 (C), PC500 (D) powders and, S5-300A (A) and S5-300B (B) sols, from CrystalACTIV [37], and an acidic TiO_2

sol (T) that was prepared in the lab by hydrolysis of titanium isopropoxide in diluted nitric acid media under energetic stirring, the mix was kept stirring for days until peptization and afterwards was dialyzed [38,39]. In addition, a home-made acidic silica (SiO_2) sol [40], prepared by hydrolysis of tetraethylortosilicate (TEOS) in acidic aqueous media and continuous stirring until peptization, was used in hydrophilic mixtures of some of these materials. Besides, a polymethyl-hydrogen siloxane (85–100%) oil, PMHS (XIAMETER MHX-1107) from Dow Corning with a specific gravity of 0.997 (at 25 °C) and a viscosity of 30 cSt was used as hydrophobic agent [41–43].

One of the primary concerns when choosing the photoactive materials was particle size. The particles should be small enough to achieve a homogeneous distribution of titania nanoparticles over the surface of concrete blocks, not alter the esthetic, be transparent to translucent, have a high surface area, produce a continuous coating, but not be small enough to be a health concern.

The main physico-chemical properties of photocatalysts were determined: sols composition and titanium quantification were analyzed by using an inductively coupled plasma with optical emission spectrophotometer Perkin-Elmer 3300DV. The crystallite size was estimated by applying the Scherrer equation to X-Ray diffraction data (PANalytical X'Pert). Specific surface area was calculated by BET method from data of nitrogen adsorption isotherms (Micromeritics, ASAP 2420). The surface morphology was analyzed by scanning electron microscopy (Hitachi Tablet Microscope TM-1000).

2.2. Preparation of concrete/mortar samples

Two different construction material supports were employed: concrete blocks and cement tiles. Concrete blocks were fabricated using a mix of crushed limestone coarse aggregate, graded sand, ordinary Portland cement and water in 3.47:1.87:1.0:0.45 proportion, respectively. Water reducing admixture (WRA) and air entraining agent (AEA) were added as necessary to produce a slump of 7.5 cm (± 2.5 cm) and an entrained air content of 6% ($\pm 1\%$) when tested to ASTM standards. The WRA was added to half of the water and added to the coarse and fine aggregates in a mechanical concrete mixer and mixed for 1 min to moisten the aggregate particles. The cement was then added to the mixer, followed by the remainder of the water and the AEA. The concrete was mixed for 3 min, allowed to rest for 5 min, and then mixed for an additional 3 min. The concrete was cast into 10 cm \times 10 cm \times 30 cm molds and demolded after 24 h. The specimens were then transferred into a 100% RH curing chamber for 28 days.

The cement tiles were made from Portland cement mortar (1:1 cement:sand mix at W/C = 0.35) and cast into 15 cm \times 15 cm \times 1 cm stainless steel molds. After casting, the tiles were cured for 28 days in water.

After curing, all the specimens were sliced into small sample coupons (10 cm \times 5 cm \times 1 cm) using a diamond saw.

2.3. Concrete coating

The photoactive coating has been applied by two different procedures, spray and dip coating. The spraying method was used for home-made sols in order to control and minimize the amount employed, when the commercial sols and suspensions were applied spray or dip coating were used, except in the case of hydrophobic suspensions that presented a high viscosity to produce the aerosol. In the case of layers deposited by spraying, the amount of sol was varied to obtain 200, 500 and 1000 nm of theoretical thickness (calculated as, volume = desired layer thickness * specimen length * specimen width).

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