



Full Length Article

Surface treatments of metal supports for photocatalysis applications

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ABSTRACT

One of the most important challenges, for scaling up a photocatalytic system for VOCs abatement to full-scale, is the design of a suitable photocatalyst support. The support has to firmly immobilize the photocatalyst, without using an organic adhesive, and should also withstand relatively high mechanical stresses. Metals may be effectively implemented as a support material, after a corrugation of the surface with electrochemical treatments. In the present work, we treated stainless steel and aluminum supports, evaluating the surface modifications due to the electrochemical treatments, with scanning electron microscopy (SEM) and confocal microscopy. Five samples showing the highest degree of restructuring were selected and spray coated with P25, a TiO₂ photocatalyst, evaluating the mechanical stability of the coating with a standard tape test method. One particular stainless steel sample presented a superior surface restructuring and coating stability. The photocatalytic activity of this sample, evaluated measuring the complete oxidation of acetaldehyde, was tested for 15 h, and compared with sample of TiO₂-P25 on a ceramic support. The stainless steel exhibited a constant performance after an initial stabilization period. The stainless steel sample showed a slightly higher activity, due to the surface restructuring, increasing the irradiated area available for the coated photocatalyst.

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1. Introduction

Urban air pollution, constituting an important hazard for human health, is one of the most challenging environmental issues the last few decades. The WHO (World Health Organization) states that several hundred thousand deaths per year are attributed to air pollution, a number higher than any other environmental issue for developed countries [1–4]. Furthermore, making the issue even more challenging, urban air pollution generally involves a broad range of compounds from numerous sources, making it hard to pinpoint the exact cause of environmental hazardous pollutants [5].

Among other compounds, VOCs (Volatile Organic Compounds) constitute a very important category of air pollutant. They are present in local areas of the atmosphere and may be very toxic and hazardous even at very low concentrations.

The complexity of an emitted VOC mixture and the toxicity of single compounds may create synergies enhancing the hazard of the whole mixture, sometimes resulting in toxic levels higher than those of well-known toxic compounds, such as benzene [6].

The emitted VOCs, is also involved in reactions, producing ozone and smog at ground level, caused by the reaction of NO, VOCs and sulfur compounds [7–9]. Since ozone is a strong oxidant, it directly attacks proteins and lipids or activates intracellular oxidation mechanisms [10]. Furthermore, smog and particulates are harmful for the respiratory and cardiovascular systems [10].

Another environmental issue, related to VOCs, is odor emission. Some chemical compounds have a very low odor threshold [11–13], often in the ppb range, making the VOCs emissions very odorous. In recent years, the awareness of this issue has increased, manifested as a growing number of complaints and stricter environmental local legislations [14].

There are many different sources of VOCs emission, which may be grouped in four different categories: power generation, transportation, residential and industrial processes [15]. For all these categories, constantly increasing VOCs emissions have been recorded in the last year, with the industrial sector dominating [15,16]. It is therefore crucial to efficiently treat the industrial emissions, possibly with green technologies [17]. Catalytic and thermal incineration has been widely used in the past to purify the industrial exhausts [18]. However, these technologies are neither efficient, due to a significant heat up of the whole gas to high temperature, nor environmental friendly, due to a general use of fossil fuel for supplying the heat. Photocatalysis, based on TiO₂ photocatalysts,

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offers a promising alternative for VOCs abatement [19]. The light irradiation on the photocatalytic surface induces a very fast oxidation reaction already at room temperature, due to formation of hydroxyl radicals [20]. Therefore, there is no need for heating up the gas stream, thus avoiding the use of fossil fuel and, at the same time, reducing the CO₂ emissions. The final result is a significantly more environmentally friendly mitigation process.

When photocatalysis is used under UV light, it is possible to combine this technology with ozonation, using the UV lamps both for ozone generation and for activation of the photocatalyst [21–23]. This combination significantly increases the efficiency of the VOCs removal, making it suitable for an industrial application. There are, however, a few challenges to be addressed before utilizing the technology in operational full-scale photocatalytic systems. One of the most important challenges is the design of an appropriate photocatalyst support. To be suitable for full-scale applications, the support needs to fulfill several requirements. First, it needs to firmly immobilize the photocatalyst, without using adhesives that may be degraded by the UV light, which is the case for organic adhesives [24]. The photocatalyst also needs to be efficiently exposed to the light, maximizing the irradiated surface area, and thus also maximizing the activity of the catalyst. Furthermore, a close contact between the VOCs and the catalyst needs to be ensured, minimizing the mass transfer limitations between the bulk gas phase and the surface. In a full-scale UV reactor, the airflow could reach relatively high speed, up to 10 m/s. It is therefore important that the support withstands the mechanical stresses generated, which may break fragile supports. Finally, the cost of the whole system needs to be minimized. Both the manufacturing process of the reactor and the materials for the support need to be evaluated in order to develop a cost-effective solution.

Different materials can be used as photocatalyst support [25]. Previously, glass and quartz have been used in different forms: beads, fibers, pellets or sheets. Ceramic materials have also been widely investigated, especially cyclosilicates as cordierite, or phyllosilicates as organoclays. Metals are another important category investigated, especially stainless steel. The porosity of ceramic and glass supports, generally, simplifies the coating procedure, making it attractive for use in R&D at lab scale. Traditional dip-coating or pipetting procedures may then be applied to achieve a coating stable enough for laboratory experiments [26,27]. In the case of metallic surfaces, the general lack of porosity makes it inappropriate to use the same coating procedures for these materials, and therefore they are not commonly used in laboratory applications. The situation changes significantly, when a full-scale case is considered. In an industrial reactor, the natural choice for coating a photocatalyst should be on the walls of the reactor or on structures, specifically designed for the purpose. Metals are generally the material used for the walls, supporting the entire reactor. Having glass or ceramic on the walls, means adding another layer on the metal, increasing the production costs of the reactor. In case of structures (e.g. plates or foams), specifically built to support the photocatalyst, the manufacturing process is much simpler and more inexpensive if metal is used, since it is easier to shape. Furthermore, metal can withstand the mechanical stresses generated inside the reactor, ceramic or glass supports may not sustain the load, resulting in cracks and fractures of the support. For these reasons, metal is definitely the preferred material in full-scale applications. However, a catalytic powder is difficult to coat on metal, due to the particularly smooth surface, and the coating process is therefore challenging.

One way to ensure a stable coating is the surface treatment of metals by corrugation. The treatment produces a very rough surface, possibly with a positive skewness, enabling a deposit of the photocatalyst particles in the valleys of the corrugated surface [28]. The photocatalyst particles are then protected from the

mechanical stresses, caused by, for instance, an air stream flowing at high speed on the surface of the support. Several treatments may be used on metallic materials, such as stainless steel and aluminum, to modify the surface. These treatments can be divided into three main categories: chemical etching, electrochemical DC (direct current) etching and electrochemical AC (alternate current) etching. The first category is performed exposing the metallic surface to a solution of an etching agent, normally, an acid. Sulfuric acid and “H₂SO₄–H₂O₂” solution (a concentrated solution of H₂SO₄ and H₂O₂ with a v/v ratio of 2:1, occasionally called “piranha” solution) have both previously been used to treat stainless steel [29,30]. The treatments proved to greatly increase the roughness of the sample surface, improving the adhesion of the coated layer. On the other hand, very few works have been reported for aluminum, especially using the “H₂SO₄–H₂O₂” solution. For this material, the electrochemical DC etching with sulfuric acid of aluminum has been thoroughly investigated. The process is called anodization and has been studied for a wide range of applications, mainly focused on corrosion protection [31]. Recently, a porous surface, produced by anodization, was used to improve the adhesion of a coated layer [32]. Giornelli et al. [33] successfully dip-coated a V₂O₅/TiO₂ catalyst on aluminum after anodization. Nevertheless, it is also important to address how anodization is effective in forming a surface, mechanically stabilizing the catalyst in comparison to other treatment techniques. Other works investigated the effect of voltage and temperature on porosity and pore diameter [34,35]. Fewer works, presenting the treatment with DC current of stainless steel rather than aluminum, are present in the literature. Lee et al. [36] used aqua regia in DC current, observing a very high restructuring of the steel surface, improving the retention properties of a lubricant oil. Therefore, we expect that the same treatment could be successfully implemented to stabilize a photocatalyst coating.

The treatment of aluminum surface has also been investigated applying an AC current [37–39], while the same treatment has not been applied, to the best of our knowledge, on stainless steel yet. In this case, the situation is more complex, since the current can be either pulsed [37,38] or sinusoidal [39]. In both cases, the treatment produces cubical terraces on the surface of the aluminum metal, with a cube size depending on the current frequency [39] and on the number of applied cycles [37]. Furthermore, Ono and Habazaki [38] analyzed the effect of H₂SO₄ on cubical pits generation and distribution. However, the work is not directly connected to a coating application and it is not well defined how the pit properties are affecting the stability of a photocatalyst coating.

Since there is broad range of treatments applicable on metals, the produced surfaces also have a wide range of applications, e.g. corrosion protection [31], oil retention [36] or improving electrolyte capacity [40]. However, to the best of our knowledge, very few works have been performed evaluating the effect of surface restructuring on the mechanical stability of a catalyst coating. We believe that the improved surface properties, observed to be beneficial for other applications, could be transferred to coating applications.

Electrochemical treatments for tailoring photocatalyst metal supports have not been very well investigated, especially in terms of how the morphology affects the coating stability and activity of the photocatalyst. In the present work, we investigated how various electrochemical treatments modify the surface structure of photocatalysts supports. Stainless steel and aluminum supports were considered, since they are more suitable than ceramic ones for industrial applications. We investigated several treatments, both novel and adapted from the literature, evaluating the resulting roughness. Samples showing the highest degree of surface modifications were selected for coating with the TiO₂-P25 photocatalyst. The mechanical properties of the resulting metal-catalyst couple were assessed, evaluating how the obtained support morphology

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