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Three-phase photocatalysis using suspended titania and titania supported on a reticulated foam monolith for water purification

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Abstract

This paper presents the first results on the performance of a pilot-scale photocatalytic oxidation reactor having a TiO_2-coated 15 pores-per-inch (15 PPI) alumina reticulated foam monolith installed in the annular space between a centrally installed 1 kW UV lamp and the internal wall of the reactor. Photocatalytic oxidation of 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) in water was chosen as a test reaction to evaluate the performance of the new reticulated foam photocatalytic reactor and to compare its photocatalytic efficiency with that of the same reactor but with the same mass concentration of TiO_2 suspended in water as slurry catalyst. The reticulated foam monoliths were spray-coated with 10 and 12 wt.% Degussa P25 TiO_2 (equivalent to 18.58 and 21.78 g/L of water, respectively, in suspension). Results were analyzed in terms of overall DBU conversion, TOC conversion, initial reaction rates per unit reactor volume, photonic efficiency and overall process economics. For the test reaction above, results indicate that the 12 wt.% TiO_2-coated reticulated foam photocatalytic reactor was more efficient compared to the reactor using equivalent TiO_2 slurry, with DBU conversion of 100%, TOC conversion of approximately 23% and quantum yield of 3.8 (–) achieved in 60 min. The results clearly suggest that immobilised TiO_2 could be a cost-effective method that should be taken further to large-scale water purification using heterogeneous photocatalysis.
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1. Introduction

The purification or treatment of contaminated or waste water by heterogeneous photocatalytic oxidation has been an increasingly interesting subject of research for the last 20 years due to its potential as a viable alternative to other advanced oxidation processes [1–6]. Titanium dioxide (TiO_2) Degussa P25 is the most commonly used photocatalyst due to its advantages over other known photocatalysts and has often been proposed for the degradation of pollutants in water or air [7].

The majority of published work on heterogeneous liquid phase photocatalysis has reported the use and efficiency of aqueous suspensions of TiO_2. However, downstream separation of the nano-size Degussa P25 TiO_2 from the treated water and the recycling of the photocatalyst have proved very difficult and uneconomical, especially with waste waters [8].

Due to the problems mentioned above, there has been an increase in research into immobilisation of TiO_2 on solid supports to eliminate the difficult separation and recycling of catalyst, thus improving the process economics. A literature review of reactor configurations tabulating photocatalytic research using slurry reactors and immobilised catalyst reactors can be found in [9], while a literature review tabulating methods of immobilisation of TiO_2 and support substrates coated can be found in [10,11]. Some researchers have reported that the efficiency of the slurried photocatalyst is larger than the supported form [8,10,12] while others have reported that the catalytic activity of TiO_2 is not affected when it is immobilised [13]. A direct and precise comparative assessment of the performance of reactors using suspended TiO_2 and

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immobilised TiO_2 particularly on the basis of actual mass concentration of TiO_2 immobilised on the support or in suspension is often hard to find in the open literature.

This work was carried out to develop an efficient photocatalytic support that can be used to eliminate persistent, non-biodegradable and toxic pollutants contained in waste or contaminated waters. The aim is to precisely compare the efficiencies of TiO_2 Degussa P25 in aqueous suspension and the same TiO_2 Degussa P25 immobilised on an inexpensive, custom-made 15 PPI reticulated foam monolith as a new photocatalytic support for pilot-scale water treatment. Only a few researchers [14–18] have carried out related photocatalytic studies using 10, 20 and 30 PPI reticulated foams but only for the gas phase oxidation of volatile organic compounds on a bench scale. This paper presents the first data on the use and precise assessment of the performance of a 15 PPI reticulated foam monolith for water treatment on a pilot-scale. The study was carried out using 1,8-diazabicyclo[5.4.0]undec-7-ene, also known as DBU, as a model substrate. DBU, a nitrogen-containing compound and a tertiary amine, is a recalcitrant compound present in waste water produced from pharmaceutical processes. The release into the environment as well as into surface water is possible during manufacturing processes; hence it is very important to develop the treatment technology for DBU urgently [19].

Reticulated foam monoliths are a family of materials whose light weight, flow through properties are easily customised to meet design requirements of reactor catalyst support. They exhibit a three-dimensional pore structure with versatility of shape, pore size, permeability, surface area and chemistry. A desirable property of reticulated foams is their high bed porosity which results in a much lower pressure drop in a reactor. In addition, and unlike honeycomb monoliths, reticulated foam monoliths have a tortuous pore structure which enhances intense turbulence and mixing [20]. The aim of this work, in addition to comparing the efficiency of TiO_2-coated reticulated foams with suspended TiO_2, is to show how randomly structured catalysts can improve the efficiency of a photocatalytic reactor and minimise or remove waste or by-products.

2. Experimental work

2.1. Experimental set-up: the cocurrent downflow contactor reticulated foam photocatalytic reactor (CDC-RFPR)

All experimental work was carried out using a pilot-scale cocurrent downflow contactor reticulated foam photocatalytic reactor (CDC-RFPR) having a total system volume of 15 L. However, in comparative experiments using suspended TiO_2 the reticulated foam monolith was removed from the reactor converting it to a 'slurry mode' photocatalytic reactor. In all cases, the outer wall of the photocatalytic reactor consists of a standard QVF glass tube with internal diameter 0.1 m while the inner wall is a replaceable Pyrex sleeve (external diameter 0.0465 m, length approximately 0.5 m) mounted axially at the centre of the reactor. The annular photocatalytic reaction zone was 0.5 m long and the irradiated volume was 4.0 L. A TiO_2-coated custom-made 15 pores-per-inch (15 PPI) alumina reticulated foam monolith was installed in the annular space between a centrally installed 1 kW UV lamp and the internal wall of the reactor. The schematic diagram of the CDC-RFPR is shown in Fig. 1A while Fig. 1B is the photographic illustration of the photocatalytic reactor. UV radiation was provided by a UVA 1 kW Hanovia medium pressure lamp, which provides a maximum emission at 365 nm. Oxygen and liquid streams are introduced co-currently and downwards through an orifice in the entry zone at the top of an oxygen/air absorption column (QVF glass tube, diameter 0.05 m and length 0.5 m). A 5 mm orifice plate is attached to the underside of a metal cap fitted to the top of this glass column. In this column, the energy and turbulence due to a high-velocity gas–liquid jet causes efficient mass transfer of oxygen into the liquid phase and a close approach to equilibrium of the gas and liquid phase (approximately 97%) is typical of this section of the reactor [21]. The reactor was operated in a recirculation batch mode, where liquid stream containing reactants is re-circulated back into a well mixed recirculation reservoir with the aid of a centrifugal pump. A thermocouple inserted into the reaction zone through the CDC cap monitors the reaction temperature while a cooling coil inserted into the reservoir controls the reaction temperature, keeping it at 50 °C.

TiO_2 Degussa P25 (80% anatase, 21 nm primary particle size and 55 ± 15 m^2/g specific surface area) was used for all photocatalytic reactions.

2.2. The reticulated foam monoliths and preparation of the coatings

Two 15 PPI (15 pores per inch) alumina reticulated foam monoliths were custom fabricated by Vesuvius (Hi-Tech Ceramics), Alfred, NY, USA. The annular shaped monoliths, with external diameter of 86 mm, internal diameter of 66 and 450 mm high, were designed to fit perfectly into the annular space between the vertically and centrally positioned UV lamp and the internal wall of the photocatalytic reaction zone. This allows the gas and liquid stream to flow downwards through the irradiated annular space between the internal wall of the monolith and the external wall of the UV lamp. The reticulated foam monoliths were coated by Johnson Matthey Catalysts using a customised spray-coating technology. This method was chosen to ensure uniform anchoring of the catalyst (TiO_2) onto the surface of the reticulated foam monoliths. TiO_2 Degussa P25 was made into slurry with ultra pure (Millipore) water (18.58 or 21.78 g of TiO_2 per litre of water corresponding to 10 or 12% solids). Prior to coating, the foams were dried at 105 °C for 2 h and weighed. The wash coat slurry was then sprayed onto the foams to achieve a uniform coating and a loading of 10 or 12 wt.% TiO_2 on the foams. The foams were then dried at 120 °C for 2 h and then calcined at 500 °C overnight. The catalyst loading was determined by calculating the difference in weight before and after coating.

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