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Short communication

Deposition of activated alumina powder on SiC diesel particulate filters with wall flow type through in-situ hydrothermal process

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ABSTRACT

A modified mesoporous alumina powder by urea mass percent and glycerol addition was deposited on SiC diesel particulate filters (SiC-DPFs). The activated alumina layer was successively synthesized by a surfactant-templating route through in-situ hydrothermal technique with the aim of depositing homogeneous and well-adhered layers. The effect of the urea mass percent and glycerol addition on the deposition process was also examined.

The mineral phase, surface area and structural morphology of the activated alumina layers were investigated by different physicochemical techniques such as X-ray diffraction, N_2-physisorption and scanning electron microscopy. The resultant deposited alumina powder layers possessed surface areas and pore volumes of about 20–28 $m^2\ g^{-1}{}_{supp}$ and 0.15–0.31 $cm^3\ g^{-1}$, respectively.

1. Introduction

Structured reactors have been successfully used in numerous applications ranging from the treatment of automotive exhaust gases to chemical and refining applications. The main advantages of honeycomb and foam reactors over conventional packed bed reactors include high geometric surface area, low pressure drop when associated with high flow rates, efficient mass transfer, high thermal and mechanical stability [1]. The choice of honeycomb structure is a function of the operating conditions adopted in the specific application. In fact, the well recognized use of honeycombs is due to their unique combinations of several critical characteristics such as high melting point and excellent thermal shock resistance, high porosity and pore size distribution suitable for ease of washcoat application and good washcoat adherence [2]. Because of its low surface area, the prevailing activation technique, widely used in the industry, is the washcoating, i.e. dip coating from a slurry or suspension. The catalyst, usually available as a powder of pure catalyst or as an impregnated porous support that provides a large surface area typically γ-Al_2O_3, is deposited on the structured support from a slurry (solid dispersion), according to standard recipes. Dip coating by several cycles of immersion of the monolith in the slurry, each one followed by blowing and drying, is the most common procedure to achieve a uniform coating [3], preventing ageing and viscosity modification of the slurry [4]. Thus, several studies performed to optimize the washcoating technique suggest that the washcoat characteristics of homogeneity, reproducibility and adhesion are affected by the slurry solid content, particle size, viscosity, pH and drying temperature [5–8]. Drying steps also affect the uniformity and nature of the coating [9]. The procedure is time-consuming and its results are determined by a number of practical factors, mostly set by tradition, internal standards and laboratory habits. There is continuous research effort directed towards improved methods, aiming at reducing the variability in the final catalyst quality, saving precious precursors while maintaining the same effectiveness [10]. Orbital stirring in slurry is an interesting variation of the traditional dipping and drying procedure; less efficient in depositing catalyst, it apparently leads to better results if compared to dip coating [10]. The adhesion of powders to the support is not obvious, particularly on metallic ones [11–13], nor it is granted, and assessment of the structural stability of the washcoat could be required, e.g. by ultrasounds [3,14]. In particular, deposited alumina powder is an important substance with a large domain of applications such as high energetic reactions and petrochemical processes. Kong et al. succeeded in preparing alumina and boehmite powders by aqueous precipitation method with mixed surfactants [15]. Depending on calcination temperatures of alumina, different allotropic varieties can be obtained [16].

In this paper, the deposition of activated powder alumina as coating layers was performed on SiC diesel particulate filters (denoted: SiC-DPFs) with wall flow type through in-situ hydrothermal technique. In fact, the influence of urea mass percent and glycerol addition has been observed and discussed. The coated SiC-DPF supports were characterized by sensitive physico-chemical techniques.

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2. Materials and methods

2.1. Materials

Wall-flow SiC-DPF usually removes 85% or more of the soot, and under certain conditions can attain soot removal efficiencies approaching 100%. Some filters are single-use, intended for disposal and replacement once full of accumulated ash. Others are designed to burn off the accumulated particulate either passively through the use of a catalyst or by active means such as a fuel burner which heats the filter to soot combustion temperatures. Fig. 1 showed fresh SiC-DPF support before treatment which physically traps emission particles in the exhaust. The SiC-DPF is a porous filter material that combusts particulate matter through a combination of filtration and chemical reactions, similar to an oxidation catalyst. Diesel particulate filters can reduce hydrocarbons (HC), carbon monoxide (CO) and particulate matter (PM) by 90%.

Geometric parameters of SiC-DPF support were evaluated and represented in Table 1. The cell shape and size, which can be designed into the extrusion die, affect the geometric properties and hence the size of SiC-DPF. The cell size has a strong bearing on cell density (n), geometric surface area (GSA) and open frontal area (OFA). The SiC-DPF support has an important GSA which is useful parameter for estimation of thickness layers.

2.2. Pretreatment

SiC-DPF supports were pretreated to remove impurities such as water vapor and CO_2, and to increase the porosity. High porosity was proved to favor the adherence of prepared alumina layer with metallic supports.

At first, the supports were ultrasonically cleaned in nitric acid (Wako Japan, 60%) and then in distillated water, each for 2 h. After that, the supports were dried at 120 °C for 1 h. Supports were then calcined in air at 300 °C for 2 h. After calcination, SiC-DPF supports were ultrasonically treated by toluene and distillated water for 2 h. In fact, this treatment was able to make cells permeable to macromolecules.

2.3. Washcoating process

The activated alumina layers were synthesized via surfactant-templating method [17,18]. The corresponding layers were deposited on SiC-DPF supports with an in situ hydrothermal technique. The mass of 2 g of sodium hydroxide (NaOH) pellet was dissolved in 10 ml of distilled water. The NaOH solution was added to 150 ml triethanolamine (TEA) and heated at 120 °C for 10 min in order to totally evaporate the water content. After that, the mass of 40 g of aluminum sec-butoxide was added by drop-wise to the resulting TEA solution under stirring, and the corresponding solution was heated at 150 °C for 10 min to obtain the first solution. The mass of 30 g of cetyltrimethylammonium (CTAB) was dissolved in 250 ml of water at room temperature to form second solution. The first solution was slowly added to the second prepared solution under vigorous stirring at 60 °C. After that, the addition of desired quantities of urea or glycerol was performed in order to control the viscosity of the prepared solutions. Finally, the solution was stirred for 4 h. Subsequently, the mixture was brought into a Teflon-lined autoclave and the surfactant coated SiC foam substrates were placed horizontally in the autoclave. After hydrothermal reaction at 70 °C for 48 h, the resulting gel was filtered, washed with ethanol and dried in air. The washcoating process consists to dip the SiC-DPF in the prepared suspensions for different contact times. Finally, the coated SiC-DPF substances were calcined at 500 °C for 4 h to obtain alumina as coating layers. It should be mentioned that the resultant supports were denoted SiC-DPF-A (A corresponds to alumina). SiC-DPF-A-U and SiC-DPF-A-G correspond to the resultant supports with addition of urea or glycerol contents during suspension preparations, respectively.

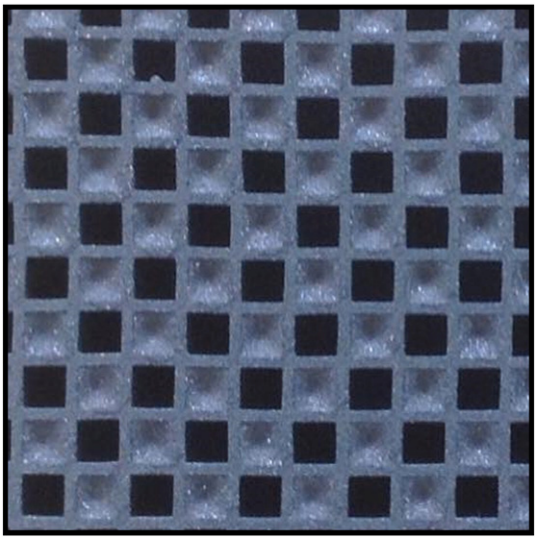

Fig. 1. SiC-DPF support with wall flow type.

Table 1
Geometric parameters of SiC-DPF support.

Geometric parameters	
Density: n (cpsi)	178
Density: n (mm^{-2})	0.44
Wall diameter d_w (mm)	1.50
Wall thickness t_w (mm)	0.36
GSA (mm^{-1})	2.01
OFA	0.57

2.4. Characterization

Coated SiC-DPF support was characterized by X-ray diffraction (XRD); 0.5 g of sample was placed in a special holder for non-powdered sample. The XRD pattern was recorded using a Cu detector; $\lambda_{Cu\ Ka} = 1.5406$ Å; over a 2θ range of 30–70°.

Brunauer–Emmett–Teller (BET) technique was used to measure the surface areas through Micromeritics Flowsorb II device at −196 °C with the following conditions: before analysis, the samples were first degassed at 250 °C for 5 h. The surface area was calculated according to the BET equation at a relative pressure ranging from 0.05 to 0.20.

The morphology of powdered-alumina and coated SiC-DPF support was observed through scanning electron microscopy (SEM) using an accelerating voltage of 200 kV.

3. Results and discussion

The coated SiC-DPF support was characterized by XRD and the results were presented in Fig. 2. The main peaks are those relevant to the presence of SiC structure. Two other peaks are attributed to the presence of γ-alumina. The alumina content of the structure was 5 wt.% which was responsible for the low intensity of the alumina peaks. Moreover, the low peak intensities indicate that the alumina was highly and homogeneously distributed over SiC-DPF surfaces and their layer thicknesses were in micrometric scales.

The SiC-DPF supports were characterized by N_2-physisorption before and after washcoating. The N_2 adsorption–desorption isotherms were presented in Fig. 3. In both cases, the adsorption isotherms were the type IV and the hysteresis loops are close to type H2. For high pressures ($P/P_0 > 0.5$), they were characterized by a saturation plateau whose length varies: the adsorption isotherms were obtained with mesoporous adsorbents in which capillary condensation occurs [19,