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# Effect of surfactant type on platinum nanoparticle size of composite $Pt/\alpha$ -Al<sub>2</sub>O<sub>3</sub> catalysts synthesized by a microemulsion method



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#### ABSTRACT

A facile method for the generation of platinum-on-alumina hybrid materials with high-surface area is presented, employing a microemulsion-based synthesis of metal nanoparticles. Pt nanoparticles (NPs) supported on  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> were prepared by the reduction of metal ions in water-in-oil microemulsion systems stabilized by a range of different surfactants with cationic, anionic and nonionic headgroups, namely AOT, CTAB, Tween80 and TX-100. The synthesized materials were characterized using Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD) and scanning electron microscopy (SEM). It is demonstrated that choice of surfactant can be used to tailor the size of the generated Pt nanoparticles, and seen that surfactant charge has a determining role in this process. Pt NPs formed in microemulsion systems based on charged surfactants (AOT and CTAB) are smaller than those prepared in nonionic microemulsion systems (TX-100 and Tween80). A solvent-induced demixing process was used to cleanly obtain the hybrid materials from the reaction medium at low energy cost.

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# 1. Introduction

The synthesis of metal nanoparticles (NPs) and exploration of their various potential applications is of considerable interest in fields such as catalysis, electronics, biotechnology and energy materials, due to the unique properties of metals at such small scales [1–5]. In the catalysis field, the preparation of NPs can be achieved by various chemical techniques including the co-precipitation method [6-8], sol-gel process [9-11] and hydrothermal processes [12,13]. An alternative, low-energy method to prepare nanosized particles with a great deal of size and shape control is in water-in-oil microemulsion systems [14]. Microemulsion systems represent excellent media for nanoparticle synthesis, as they are isotropic liquids with nanosize water droplets on the order of 10-100 nm, dispersed in a continuous oil phase and stabilized by surfactant molecules. Reduction of metal salts within the droplets offers 'nanoreactors' that can be adjusted in size using the waterto-surfactant ratio. Thus, using microemulsions, it is possible to control synthesis parameters and produce tailor-made products down to a nanoscale level with new and special properties. In previous work, we have shown that the size of the aqueous droplets in

a water-in-oil microemulsion can be varied by the oil phase [15], also affecting the size of the nanoparticles [16].

Platinum-based catalysts supported on  $Al_2O_3$  show excellent activity and stability for use in important reactions such as catalytic oxidation of naphthalene [17], hydrogen production for fuel cells [18], 1,3-butadiene hydrogenation [19], selective CO oxidation [20], diesel soot and NOx abatement [21], and so on. Karakaya et al. established a simple and rapid chemisorption technique for the quantification of metal dispersion in different types of supported metal catalysts, without the need for a sophisticated apparatus and ultra-high vacuum conditions [22]. They presented exemplary results for diesel oxidation catalysts based on Pt/  $Al_2O_3$ . Another interesting feature of Pt-based catalyst supported on  $Al_2O_3$  is atmospheric desulfurization of diesel fuel. Haji et al. investigated hydrodesulfurization of commercial diesel fuel at atmospheric pressure using Pt/ $Al_2O_3$  catalysts prepared by a supercritical carbon dioxide deposition method [23].

In this work, we explore the effect of surfactant type on the size of Pt NPs synthesized by the microemulsion method and deposited on  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>. It is shown that by changing the surfactant headgroup chemistry, the particle size of Pt NPs supported on  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> can be controlled. To prepare these systems, w/o microemulsions containing an aqueous solution of H<sub>2</sub>PtCl<sub>6</sub> and stabilized by the surfactants AOT, TX-100, Tween80 and CTAB were prepared. By addition of a reducing agent, hydrazine, stable colloidal solutions of Pt NPs were

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obtained from reduction of Pt<sup>4+</sup> ions. A simple phase separation method, using THF as a destabilizing solvent, was used to break the colloidal dispersions and release the Pt NPs onto a colloidal  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> support. Infrared spectroscopy (IR), scanning electron microscopy (SEM) and X-ray diffraction (XRD) were used to characterize all of the prepared samples, showing that nanosized platinum particles were dispersed uniformly on the  $\alpha$ -alumina.

# 2. Materials and methods

#### 2.1. Reactants

Hexachloroplatinic acid (H<sub>2</sub>PtCl<sub>6</sub>) (99%) and cyclohexane were obtained from Merck–Schuchardt and used as received. The purity of the cyclohexane was checked by gas chromatography and confirmed to be >99%. The surfactants Sodium 1,4-bis(2-ethylhexoxy)-1,4-dioxobutane-2-sulfonate (AOT, 96%), cetyltrimethylammonium bromide (CTAB, 99+%), poly(oxyethylene) sorbitan monooleate (Tween80, 99%) and poly(ethylene glycol) *p*-(1,1,3,3tetramethylbutane)-phenyl ether (TX-100, 98%) were purchased from Acros Organics and used without future purification (see Table 1).  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> was obtained from Shijiazhuang Baicheng Chemical Co., Ltd. (surface area 16–18 m<sup>2</sup>/g and particle size 80-100 nm).

#### 2.2. Synthesis of platinum colloidal suspensions

Colloidal suspensions of Pt NPs were prepared in w/o microemulsions containing cyclohexane as the continuous oil phase and one of four surfactants: AOT, TX-100, Tween80 and CTAB. A suitable amount of 0.01 M aqueous H<sub>2</sub>PtCl<sub>6</sub> was added to the 0.12 M surfactant solution in cyclohexane such that [H<sub>2</sub>O]/ [surfactant] = 5. Thus four separate microemulsions containing H<sub>2</sub>PtCl<sub>6</sub> were prepared, each stabilized by a different surfactant. These samples were equilibrated for 3 hours with stirring, after which the platinum ions were reduced by addition of a separate microemulsion system containing N<sub>2</sub>H<sub>4</sub>. The molar ratio of N<sub>2</sub>H<sub>4</sub>:Pt was kept at 10:1. After 4 h of stirring to ensure complete mixing and reduction, the reaction was considered complete. UVvisible spectroscopy was used to monitor the formation of Pt NPs for all prepared colloidal systems. The stability of all colloidal systems as prepared was more than 2 months at ambient conditions.

#### 2.3. Preparation of $Pt/\alpha$ - $Al_2O_3$

The  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, as a supporting material on which to adsorb the Pt NPs, was added to the Pt NPs colloidal suspensions in a 50:1 Al<sub>2</sub>O<sub>3</sub>:Pt mass ratio. After addition of the alumina, the colloidal system was destabilized by slow addition of THF to 3 times the volume of the initial colloidal dispersion. The suspension was then stirred for 20 h to facilitate demixing and adsorption of Pt NPs onto the  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>. The harvested products were filtered and washed with excess THF. The absence of a UV–visible absorption peak of Pt NPs in the remaining solution confirmed that the Pt NPs had fully adsorbed on Al<sub>2</sub>O<sub>3</sub> and were eliminated from the colloidal precursor systems. Subsequently, the final Pt/ $\alpha$ -Al<sub>2</sub>O<sub>3</sub> products with 1.2 wt% Pt were dried at room temperature for 24 h and calcined for 1 h at 600 °C to remove adsorbed organic material.

#### 2.4. Characterization

### 2.4.1. Scanning electron microscopy (SEM)

SEM images were made using a Philips XL30 instrument. The prepared samples were sonicated for 20 min using a Pulse 570s POT Riscaldamento W.400 instrument, and one droplet of the sample was transferred to an aluminum plate, dried at room temperature and then sputtered with gold.

# 2.4.2. X-ray diffraction (XRD)

XRD measurements were obtained using a Philips diffractometer with a 40 kV generator tension and 30 mA generator current with Cu K $\alpha$  radiation (Cu K $\alpha_1$ :1.54056 Å, Cu K $\alpha_2$ :1.54439 Å). The  $2\theta$  angular region between 20° and 100° was explored at a step time of 2.0 s and temperature of 25 °C.

# 2.4.3. Spectroscopy

Infra-red spectra were obtained using a Galaxy FT-IR 5000, and samples were prepared using KBr pressed disks. UV–visible spectra were acquired using a Specord S600 JENA AG Model spectrometer.

# 3. Results and discussion

#### 3.1. UV-visible study of Pt NPs in colloidal systems

Fig. 1 shows the typical change in the UV-visible absorption spectrum of the  $H_2PtCl_6$  microemulsion system after addition of

Table 1

Chemical structures and corresponding abbreviations of various surfactants used in microemulsion systems.



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