

A selective optical sensor for antimony based on hexagonal mesoporous structures

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Abstract

Mesoporous materials show promise in fabricating ordered sensing systems in a reproducible manner. Here, the fabrication of optically selective and sensitive sensors up to subnanomolar concentrations of Sb(III) ions was reported via simple and reproducible techniques in which the hexagonal mesoporous silicas in powder and monolith forms were used as probe carriers. Evidence of successful fabrication of the optical sensors was investigated by extensive characterizations using powder X-ray diffraction, nitrogen adsorption/desorption isotherms, ²⁹Si NMR spectroscopy, and transmission electron microscopy. The mesoporous features allowed high adsorption capacity and accessibility of probe molecules and efficient transport of toxic species via much more direct and easier diffusion to the network sites without significant alteration of their physical characteristics, leading to excellent sensing systems in terms of stability and sensitivity with rapid response time of detection. In addition, the high performance of the hexagonal sensors was dependent on key factors such as the number of support-based sensors, the reaction temperature, and the pH value that led to possible naked-eye detection of Sb(III) ion concentration with a detection limit as low as 3×10^{-9} mol/dm³ and a wide detection range of 1 ppb–2 ppm. Of particular interest was that our mesoporous sensor design provided control over the retention of the potential functionality of the naked-eye sensing system of Sb(III) ions upon the storage and even after several regeneration and reuse cycles, indicating large-scale reversibility of sensing systems.

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1. Introduction

Antimony is present in the aquatic environment as a result of rock weathering, soil runoff, and anthropogenic activities. Typical concentrations of dissolved antimony in unpolluted waters are less than 1 ppb. However, in proximity to anthropogenic sources, concentrations can reach up to 100 times natural levels. Antimony and its compounds are considered as priority pollutants by the United States Environmental Protection Agency (US-EPA) and the European Union Council of the European Communities due to their toxicity. The antimony content in drinking water is regulated to be less than 10 ppb in the European Community, 5 ppb in the United States, and 2 ppb in

Japan [1,2]. The oxidation state of antimony Sb(III) is reported to be considerably more toxic and mobile than Sb(V). In addition, the chemical form of its compounds considerably influences toxicity [1,2]. It has also been found in geological, biological, and water samples as a result of various anthropogenic activities [3–5]. Since antimony-containing additives are used in brake linings and the tire vulcanization process, road traffic is also a significant source of pollution [6]. Due to the high toxicity of antimony (III), there is a significant demand for accurate and specific determination of its trace concentration.

In the literature, the use of solid sensors to detect Sb(III) ions is negligibly reported. However, in general, the detection of Sb(III) is based on a preconcentration process using solid sorbents to improve the detection limit and for efficient removal of matrix interferences by a wide range of well-controlled equipment and techniques such as UV spectroscopy, electrothermal AAS, inductively coupled plasma mass spectrometry (ICP-MS), PSTH-Dowex microcolumn, and determination by FI-

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ETAAS and chromatographic and fluorimetric detection approaches [7–13]. Therefore, the metal-chelating resins play a leading role for the preconcentration of trace metals, including Sb(III) ions from natural waters. Although these techniques are the most useful and sensitive approaches for detection of Sb(III) ions, limitations exist in the requirements for sophisticated equipment and extreme control of experiments and environments, and therefore lead to high capital, consuming-time response, and operating costs. In this respect, increased selectivity, response speed, and sensitivity in the chemical and biological determination of gases and liquids are of great interest. Particular attention is paid to sensor materials, which are applicable to sensors exploiting various energy transduction principles, such as radiant, electrical, mechanical, and thermal energy [14].

Optical sensors that have high-performance sensitivity and are more selective with fast kinetic response are required today to analyze ultra-trace levels of environmental pollutants. The development of sensing processes, which includes discovery, optimization, and commercialization [15], remains a significant challenge in materials science. In fact, the successful design of sensor materials required high implementation of controlled assessment processes, involving the evaluation of intrinsic properties (sensitivity, selectivity, and active shelf-life or long-term stability) and manufacturability (operating cost, reversibility, and high capital tolerances). However, in the interior of the sensing systems, the complex nature of the interactions between functional groups and compositions, control syntheses, and end-use conditions of sensor materials has often led to their rational design for certain “real world” sensing applications [16–23]. Some challenges that remain for these nanostructured sensors are the achievement of the rational immobilization of surface-bound ligands such as probe moieties into the freestanding support matrices of inorganic oxides to preserve their large analyte-binding ability and to attain a stable and reproducible sensor response upon reuse cycles. However, lower detection limits are desirable to compensate for the low extraction efficiency and to allow monitoring of Sb species in specimens that have not been extensively exposed to Sb(III) ions. In our Sb(III) ion-sensing system, the design of optical nanosensors with 2D hexagonal mesoporous silica carriers that have high surface area, uniformly sized pores, and large particle grains-like monoliths enabled these nanosensors to act as preconcentrators with simultaneous naked-eye detection down to a trace level of concentration. In addition, this simple ion-detection process could be performed over a wide, and tunable range of Sb(III) ions. Therefore, our sensing method is a one-step, simple procedure for separation and simultaneous detection of metal ions without using sophisticated instruments. To the best of our knowledge, this is the first time that an optical solid sensor has been designed based on hexagonally ordered mesoporous silica materials for the efficient detection of Sb(III) ion and for achieving more than the required level of the US-EPA drinking water standards, along with high stability and reproducibility of optical sensors.

Here, we report the fabrication of optically selective and sensitive sensors up to subnanomolar concentrations of Sb(III) ion

via simple and reproducible techniques in which the hexagonal mesoporous silicas in powder and monolith forms were used as probe carriers. This nanostructured sensor design led to excellent sensing systems in terms of stability and sensitivity with rapid response-time of detection. Controlled experimental conditions also made it possible to create possible naked-eye detection of Sb(III) ion concentration with detection limits as low as 3×10^{-9} mol/dm³ and a wide detection range of 1 ppb–2 ppm, indicating high Sb(III) ion loading capability and strong binding affinity even at low concentrations of selected Sb(III) target. Significantly, the optically hexagonal sensors revealed the exceptional retention of the potential functionality of the naked-eye sensing system of Sb(III) ions upon storage or even after several regeneration and reuse cycles, making our sensing systems more applicable to unique sensing applications.

2. Experimental

2.1. Materials

All chemicals used were of analytical reagent (AR) grade and used as purchased without further purification. Tetramethyl orthosilicate (TMOS), tetraethyl orthosilicate (TEOS), triblock copolymer Pluronic P123 EO₂₀PO₇₀EO₂₀, and Brij 56 (polyoxyethylene, C₁₆H₃₃(OCH₂CH₂)₁₀OH, C₁₆EO₁₀) were obtained from Sigma-Aldrich Company Ltd., USA. Anhydrous ethanol and toluene were purchased from Wako, Japan. *N*-Trimethoxysilylpropyl-*N,N,N*-trimethylammonium chloride (TMAC) (50% methanol solution) was purchased from Gelest, Japan.

2.2. Synthesis of hexagonal silica as carriers

2.2.1. Hexagonal silica monoliths (HOM-2)

HOM-2 silica monoliths were synthesized using direct templating with the lyotropic liquid crystalline phase of Brij 56 as template, as previously reported [24]. Under typical conditions, the composition mass ratio of Brij 56:TMOS:HCl/H₂O was 1:2:1. Homogeneous sol-gel synthesis was achieved by mixing Brij 56/TMOS in a 100-cm³ round flask and then shaking at 50 °C for 2 min until homogeneous. The exothermic hydrolysis and condensation of TMOS occurred rapidly by addition of an acidified aqueous solution of HCl (at pH 1.3) to this homogeneous solution. The methanol produced from this hydrolysis of TMOS was removed in vacuo at 40 °C. As the hydrolysis/condensation reactions continued during the evacuation, the liquid viscosity of the material increased, and the resulting optical gel-like material acquired the shape and size of the reaction vessel. After a 10-min evacuation, the translucent glassy monoliths were collected and kept at 40 °C for 16 h. The surfactant was removed by calcination at 450 °C (1 h under nitrogen and 6 h under oxygen) [24].

2.2.2. Hexagonal silica powder (SBA-15)

SBA-15 silica powder was fabricated as previously reported [25]. However, 8 g of Pluronic P123 (EO₂₀PO₇₀EO₂₀) copolymer was first dissolved in 250 ml of 1.9 M HCl at 40 °C,

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