

†Honda R&D, Wako Research Center,  
Wako 351-0193, Japan

‡International Rice Research Institute, Manila,  
DAPO Box 7777, Philippines

§BioResources Center and ¶Plant Molecular Biology  
Laboratory, Riken, Tsukuba 305-0074, Japan

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Materials chemistry

## Macroporous crystalline vanadium oxide foam

Porous inorganic solids can be synthesized in complex forms<sup>1,2</sup>, and here we describe a simple method to create an ultralight, macroporous, crystalline vanadium oxide foam by bubbling oxygen gas produced *in situ* through a viscous vanadium oxide gel. This foaming process could be extended to other metal oxides.

Vanadium oxide ( $V_2O_5$ ) powder (1 g, or  $5.5 \times 10^{-3}$  mol) is mixed with a solution of a long-chain alkylamine (1-hexadecylamine,  $C_{16}H_{33}NH_2$ ; 1.99 g, or  $8.24 \times 10^{-3}$  mol) in acetone (3 ml). A pasty material is formed, to which is added an aqueous solution of hydrogen peroxide (50 ml, 30%). As  $H_2O_2$  decomposes, oxygen gas evolves and a voluminous bright yellow foam forms spontaneously (Fig. 1). The volume of the foam increases rapidly and reaches 2.5 litres after about 30 min. This new material is ultralight, as only 1 g  $V_2O_5$  is needed to generate the large volume of foam.

Scanning electron microscopy of the foam (Fig. 2a) reveals that it is highly porous and has an irregular, honeycomb-like morphology. Large pores a few micrometres in diameter are surrounded by vanadium oxide walls about 1  $\mu m$  thick. This unusual pattern resembles the hexagonal cellular structures found in the skeletons of some diatoms and radiolarians<sup>3</sup>. The mechanical properties of this porous material are poor — the ultralight foam is brittle and can easily be crushed into a powder.



**Figure 1** Light fantastic: up to 3 litres of a low-density, bright yellow vanadium oxide foam are formed when 1 g of  $V_2O_5$  reacts with hydrogen peroxide in the presence of hexadecylamine.

The vanadium oxide walls are crystalline, and X-ray diffraction reveals five sharp peaks that can be assigned to the 001 peaks of a lamellar solid with a preferred orientation (Fig. 2b). The first 001 peak gives a basal distance,  $d$ , of 33.4 Å, which is much larger than the distance between vanadium oxide planes in the crystalline oxide or in  $V_2O_5 \cdot 1.6H_2O$  gels ( $d = 11.5$  Å). This basal distance corresponds to that noted for hexadecylamine molecules intercalated in vanadium oxide gels in which the alkyl chains are perpendicular to the oxide planes<sup>4</sup>.

Contrary to observations of hybrid organic–inorganic vanadium oxide com-

pounds, the bright yellow coloration of our vanadium oxide foam indicates that all the vanadium ions are probably in their highest oxidation state ( $V^{5+}$ ). The hybrid foams contain no water, even when dried at room temperature, and they have amphiphilic alkylamines intercalated between vanadium oxide layers and are highly hydrophobic, separating readily at the surface of an aqueous solution.

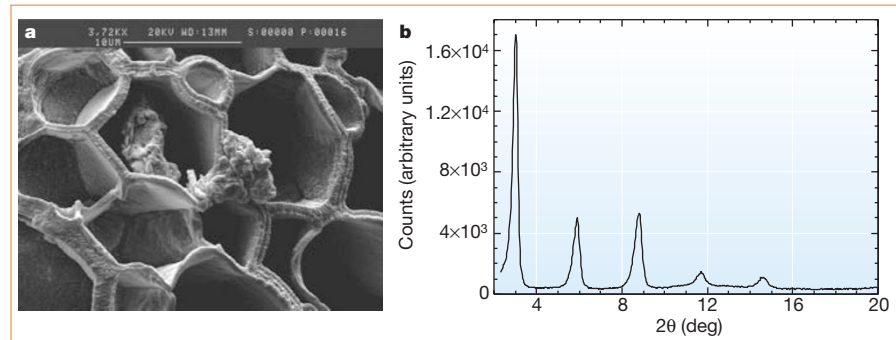
Vanadium oxide reacts with hydrogen peroxide to give  $V_2O_5 \cdot nH_2O$  gels<sup>5</sup> that have a layered structure composed of negatively charged vanadium oxide ribbons. These gels can intercalate a wide variety of inorganic and organic species, such as protonated long-chain alkylamines<sup>6</sup>. In the foaming process described here, gelation and intercalation both occur when the 1-hexadecylamine and hydrogen peroxide solutions are added to vanadium oxide powder. The intercalation of hexadecylamine between the  $V_2O_5$  layers creates a pasty solid, while the hydrogen peroxide decomposes spontaneously in the presence of vanadium oxide. Large pores are formed by oxygen gas released through the viscous gel in the presence of surfactants: as they escape, the oxygen bubbles cause the formation of a voluminous polyhedral foam, without destroying the crystalline structure of the oxide network of surfactant molecules intercalated between the layers of vanadium oxide.

Meso- and macroporous solids are usually made in the presence of organic templates which then have to be removed without damaging the inorganic shell<sup>7,8</sup>. In the foaming method we describe here, amines are simply intercalated between vanadium oxide planes and do not act as templates for the formation of macropores. The experimental requirements are not crucial — foams can form with amine/ $V_2O_5$  molar ratios ranging from 0.5 to 2.5, other surfactants can be used and the process can be extended to many different oxides (for example, those of molybdenum ( $MoO_3$ ), titanium ( $TiO_2$ ) and zirconium ( $ZrO_2$ )).

**G. T. Chandrappa\*†, Nathalie Steunou\*, Jacques Livage\***

\*Chimie de la Matière Condensée, Université Paris VI, 4 place Jussieu, 75252 Paris, France  
e-mail: livage@ccr.jussieu.fr

†Present address: Department of Chemistry, Bangalore University, 560001 Bangalore, India



**Figure 2** Structure of vanadium oxide foam. **a**, Scanning electron micrograph of vanadium oxide foam, showing its honeycomb-like structure. **b**, The vanadium oxide walls are crystalline, as indicated by the 001 peaks of the X-ray diffraction diagram.

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