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A Borate Ester Network Langmuir-Blodgett Film of 1,3,5-Benzenetriol Monohexadecyl Ether

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A Borate Ester Network Langmuir-Blodgett Film of 1,3,5-Benzenetriol Monohexadecyl Ether

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A two-dimensional borate network of an amphiphilic phenol has been produced through the Langmuir-Blodgett (LB) technique. An amphiphilic phenol, 1,3,5-benzenetriol monohexadecyl ether (C16–1,3,5-BT) was synthesized by the reaction of phloroglucinol dihydrate (1,3,5-benzenetriol) with 1-iodohexadecane. Monolayers of C16–1,3,5-BT were spread on the 1% aqueous boric acid subphases with various pH. The molecular structure of the LB film was determined by the FT-IR and XPS. The mechanical stability of the network film was indirectly evaluated by the SEM observation of the film morphology covered over the porous substrate.

Keywords: borate; monolayer; network; Langmuir-Blodgett

INTRODUCTION

The network structure is important for molecularly-thin films such as the Langmuir-Blodgett (LB) film in order to improve the intrinsic fragility and to make their technological applications possible[1]. For the stability improvement, cross-linked LB films of organic amphiphiles have been obtained by condensation reactions[2], UV irradiation[3], and polyion complexation followed by heat treatment[4]. However, the inorganic network structure was seldom introduced for the cross-linked LB films[5].

In this paper, we report for the first time a two-dimensionally cross-linked borate monolayer film of an amphiphilic phenol, 1,3,5-benzenetriol monohexadecyl ether (C16-1,3,5-BT). The LB films were characterized by FT-IR, XPS, and SEM measurements.

EXPERIMENTALS

An amphiphilic phenol, 1,3,5-benzenetriol monohexadecyl ether (C16-1,3,5-BT) was synthesized by the reaction of phloroglucinol dihydrate (1,3,5-benzenetriol) with 1-iodohexadecane.

A film balance system NLE-LB200-MWC (Nippon Laser and Electronics) was used for measuring surface pressure as a function of molecular area and for LB transfer of monolayer by the vertical mode (trough surface size, 80X585 mm^2). FT-IR spectra of LB films on calcium fluoride plates were obtained on a Perkin-Elmer spectrometer by the transmission method. XPS spectra were obtained on V.G. Scientific X-ray photoelectron spectrometer. SEM micrographs of LB films on FP-010 membrane filters were taken by JSM 35CH scanning electron microscope.

RESULTS AND DISCUSSION

Monolayer Properties at the Air-Water Interface

The surface pressure-area (π-A) isotherms of C16-1,3,5-BT monolayer are shown in Fig. 1. When C16-1,3,5-BT was spread on acidic 1% boric acid subphase (pH 3), the π-A isotherm was almost same as that on pure water subphase. However, the π-A isotherm revealed expanded monolayer phase on the basic boric acid subphase (pH 11). Ionization of the phenolic monolayer and subsequent borate ester formation are supposed to result in the expanded

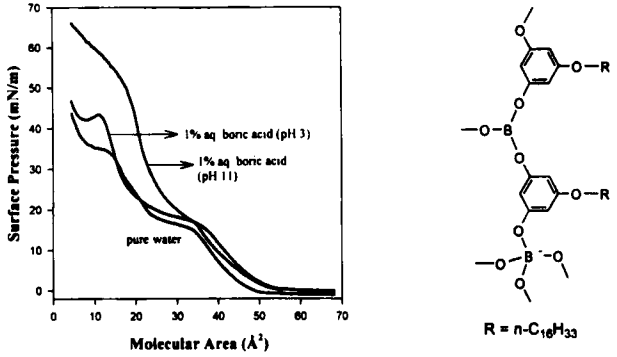

FIGURE 1 π-A isotherms of C16-1,3,5-BT and an aryl borate ester structure.

phase and increased collapse surface pressure of monolayer. Much homogeneous monolayer on the basic subphase was evidenced from Brewster angle microscopy (BAM). The images of BAM (not shown here) represented straight lines after collapse point in case of the basic boric acid subphase, while those of the pure water and acidic subphases were wavy lines.

Characterization of Network LB films

The C16-1,3,5-BT monolayer on the basic boric acid subphase was transferred on calcium fluoride plate as Y type. The FT-IR spectra of the LB film (not shown here) revealed smoothened aromatic bands around 1600 cm^{-1} and additional bands at 939 cm^{-1}, 1330 cm^{-1} owing to borate structure, which were not found in LB film deposited from pure water subphase[6]. XPS spectra (not shown here) also presented the existence of boron in the LB films, the concentration of the boron was calculated to be ca. 0.4 equivalent of C16-1,3,5-BT. The network structure of LB films can be estimated from the formation of borate aryl ester as illustrated in Fig. 1. The network formation could be indirectly evidenced from the coverage capability of the substrate

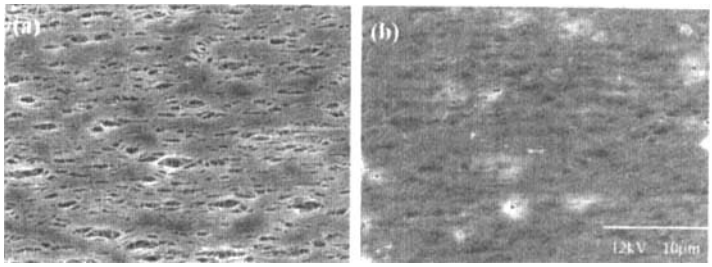

FIGURE 2 SEM micrographs of C16-1,3,5-BT LB films on FP-010 deposited from (a) pure water and (b) aq. boric acid subphase (pH 11).

pores by the network LB films. When the C16-1,3,5-BT monolayers were transferred on porous membrane filter (FP-010, Sumitomo Elect. Co.), the LB films deposited from the basic boric acid subphase could cover the pores with 10 layers. The SEM micrographs are shown in Fig. 2. However, the pores are shown still open in case of the LB films deposited from pure water subphase. As a conclusion, for the first time, we have demonstrated the two-dimensional network films of aryl borate esters stable on the porous substrate by LB technique.

ACKNOWLEDGMENTS

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