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A Two-Dimensionally Extended Conjugation from an Amphiphilic Melamine and Glyoxal

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A two-dimensional reaction of a melamine derivative with glyoxal was carried out at the air-water interface for melamine-based conjugation Langmuir-Blodgett film. An amphiphilic derivative of melamine, N-(4,6-diamino-1,3,5-triazin-2-yl) octadecanamide (NDTOA), was synthesized by the reaction of melamine with stearic acid. Monolayers of the NDTOA were spread on aqueous 3% glyoxal subphases with various pH. The surface pressure-area isotherms revealed more expanded monolayer area on the acidic or basic subphases. The molecular structure of the LB films was confirmed by the FT-IR and UV spectroscopy. Characteristic red-shifts of UV absorption peaks (λ_{max}) were detected from 201 nm to 218 nm and 240 nm, 2 or 3 conjugation number could be evaluated from the peak shift.

Keywords: melamine; conjugation; glyoxal; Langmuir-Blodgett

INTRODUCTION

Melamine has been widely used for thermosetting plastics[1]. Its mechanical and insulating properties were adopted for various electrical appliances. Recently, however, melamine-based conducting plastics were produced from reaction of melamine with glyoxal[2]. The extended conjugation of melamine and ethylene units is attributed to the electrical conductance. Although the interest on melamine-related bulk technology has been increased, however, the

investigation on thin films of the melamines has seldom carried out[3].

In this paper, we report for the first time a two-dimensional reaction of an amphiphilic melamine, N-(4,6-diamino-1,3,5-triazin-2-yl) octadecanamide (NDTOA), with glyoxal at the air-water interface. The monolayer properties were examined by surface pressure-area (π-A) isotherm and Brewster angle microscopy (BAM). The LB films were characterized by FT-IR and UV measurements.

EXPERIMENTALS

An amphiphilic melamine, NDTOA, was synthesized by the reaction of melamine with stearic acid. Aqueous glyoxal solution was purchased from Aldrich Co. and used as diluted.

A film balance system NLE-LB200-MWC (Nippon Laser and Electronics) was used for measuring the surface pressure as a function of molecular area and for the LB transfer by the vertical mode (trough surface size, 80X585 mm^2). FT-IR spectra of LB films on calcium fluoride plates were obtained on a Shimadzu 8201PC spectrometer. BAM images were taken by Mini BAM (Nanofilm Technologie Gmbh.) and UV spectra were obtained by a Shimadzu UV-1601PC spectrometer.

RESULTS AND DISCUSSION

Monolayer Properties at the Air-Water Interface

The π-A isotherms of NDTOA monolayers are shown in Fig. 1. When NDTOA was spread on aq. glyoxal subphases with various pH, all the π-A isotherms showed more expanded monolayer area than that on pure water subphase. In particular, when the subphase was highly acidic (pH 1.5) and

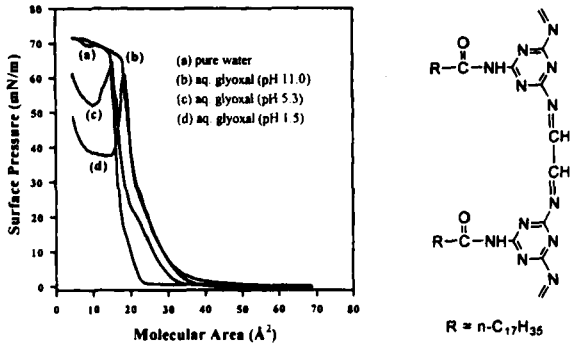

FIGURE 1 π-A isotherms of NDTOA and a melamine-glyoxal conjugation structure.

basic (pH 11.0), the monolayer areas were most expanded. The collapse surface pressures were over 60 mN/m, and drastic decrease of the surface pressure was observed on acidic subphases. The pH dependence was supposed to be related with the pH-sensitive reaction between amine and aldehyde.

The BAM images (not shown here) represented different type of collapse modes of monolayers. While many dots were seen after the collapse point on pure water and basic (pH 11.0) subphase, round islands were observed on acidic (pH 1.5) subphase. The patterns are thought to have relation with the reactions at the interface, but the reason is not clear at this point.

Characterization of Conjugation LB films

The NDTOA monolayers on pure water and basic subphase were transferred on calcium fluoride plates as Y type. The FT-IR spectra of the LB film (not shown here) revealed strong bands around 3400 cm^{-1} and 1000 cm^{-1} in case of the basic subphase system, which were attributed to the additional O-H and C-O bonds together with N-H and C-N bonds. The band owing to the alkene

structure was not clear. The formation of conjugation structure was monitored from UV spectra of the MDTOA LB films (Fig. 2). Acid and following heat treatments were applied for the complete conjugation formation. Characteristic red-shifts of UV absorption peaks (λ_{max}) were detected from 201 nm to 218 nm and 240 nm. As a conclusion, although two-dimensionally conjugated polymers were not obtained, the peak shifts showed that a melamine-based LB film with 2 or 3 conjugation number could be fabricated.

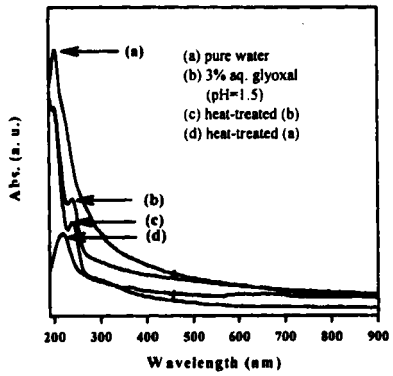

FIGURE 2 UV spectra of NDTOA LB films on quartz plates.

ACKNOWLEDGMENTS

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