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Ring-Opening Reactions of 2-Heptadecyl-2-Oxazoline at the Air-Water Interface

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The monolayer behavior of 2-heptadecyl-2-oxazoline (C17-OXZ) was examined. C17-OXZ was synthesized from condensation and cyclization of stearic acid with 2-aminoethanol. C17-OXZ was spread to monolayers on subphases with various pHs, poly(acrylic acid), and poly(allylamine). The surface pressure-area isotherm and Brewster angle microscopy were employed to investigate the monolayer properties. The chemical and morphological structures of the LB films were respectively examined by FTIR and scanning electron microscopy. The C17-OXZ monolayer showed preference to hydrolytic ring-opening rather than ring-opening polymerization.

Keywords: 2-oxazoline; monolayer; LB film; ring-opening

INTRODUCTION

2-Oxazolines (4,5-dihydrooxazoles) have been polymerized with various kinds of initiators to produce the corresponding derivatives of poly[(N-acylimino)ethylene] via ring-opening isomerization^[1]. Specifically, 2-oxazolines have received great attention for surface coating application^[2]. On the other hand, the stabilization of Langmuir-Blodgett (LB) films is important in order to improve the intrinsic fragility and to make their technological applications possible^[3]. Polymerization or cross-linking of the monolayers have been employed as alternative methods for the stabilization^[4].

In this paper, we report for the first time the reactions of 2-heptadecyl-2-oxazoline (C17-OXZ) at the air-water interface and the chemical and morphological structures of their LB films. The monolayer properties of C17-OXZ at the air water interface were investigated by surface pressure-area (π -A) isotherms and Brewster angle microscopy (BAM). The chemical structures of the resulting LB films were determined from FTIR spectra. The stability of LB films was indirectly evaluated by considering the least layer numbers necessary to cover the pores of porous substrates, which was carried out by scanning electron microscopy (SEM).

EXPERIMENTALS

Material

The preparation of C17-OXZ included the condensation reaction of stearic acid with 2-aminoethanol and following cyclization of N-(2-hydroxyethyl)octadecanamide. Dean-Stark apparatus was used for dehydration and p-toluenesulfonic acid for catalyst. The chemical structure was determined from $^1\text{H-NMR}$, FTIR, and elemental analysis.

Measurement

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, $80 \times 585 \text{ mm}^2$). BAM images were obtained from Mini BAM (Nanofilm Technologie GmbH). The employed substrates were fluorocarbon membrane filter (FP-010, Sumitomo Electric Co.) for SEM (JSM 35CH) and calcium fluoride plates (GL Sciences) for FTIR spectroscopy (Perkin-Elmer).

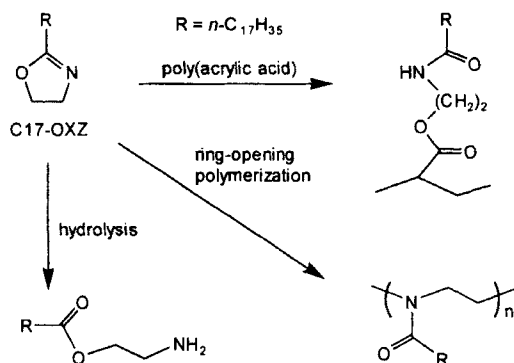


FIGURE 1 Possible reactions of C17-OXZ at the air-water interface.

RESULTS AND DISCUSSION

C17-OXZ Monolayers at the Air-Water Interface

As shown in Fig. 2, π -A isotherms of C17-OXZ monolayers revealed more expanded phases on acidic, aq. poly(allylamine), and aq. poly(acrylic acid) subphases than on pure water. Specifically, spontaneous increase of surface pressure was observed on aq. poly(allylamine) and aq. poly(acrylic acid) subphases during the incubation after monolayer spreading. Characteristic area changes were found when the monolayers were kept at a constant surface pressure. Gradual reduction of monolayer area was produced on acidic water subphase, while the area increase on aq. poly(acrylic acid) subphase. When the monolayer images were taken by BAM, any noticeable structures were not detected before the monolayer collapse. However, island-like images were found on pure water subphase after the collapse point, while band-type images were produced on aq. poly(allylamine) or poly(acrylic acid) subphases.

Characterization of C17-OXZ LB Films

FTIR spectra of the C17-OXZ LB films showed characteristic carbonyl bands which could be attributed to ester or amide bonds. The totally polymeric monolayer via two-dimensional ring-opening polymerization was not obtained. Instead, ring-opening reactions by hydrolysis or carboxylate of poly(acrylic acid) were favored. Fig. 1 shows possible reactions of C17-OXZ estimated from the results. The stability of LB films was evaluated indirectly by counting the least numbers necessary to cover the pores of porous substrates. The results showed the best stability on the LB film obtained from aq. poly(acrylic acid) subphase. In that case, almost of the pores with 0.1 μm diameter were covered by 10 layers deposition.

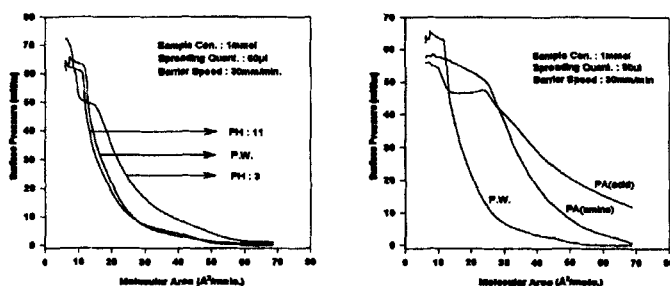


FIGURE 2 π -A isotherms of C17-OXZ on various subphases.

Acknowledgments

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