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Fabrication of Crosslinked Phenolic Polymer LB Films and its Electrical Properties

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We fabricated the crosslinked films using p-hexadecoxyphenol (p-Hp), which is amphiphilic phenol and can form polyion complexes with formaldehyde at the air-water interface. The behavior of monolayers at the air-water interface and the surface structure of LB films was investigated by Brewster angle microscope (BAM) and scanning Maxwell-stress microscope (SMM), respectively. We have provided evidence for the high insulating performance of phenol-formaldehyde thin films by the LB method.

Keywords: crosslinking; BAM; SMM; conductivity; dielectric constant; activation energy

INTRODUCTION

The network structures are 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]. Crosslinking of the LB films is an alternative breakthrough for stability improvement. The crosslinked LB films can be prepared through the polyion complexation technique[2].

In this paper, We have fabricated the crosslinked films using p-hexadecoxyphenol (p-Hp), which is amphiphilic phenol and can form polyion complexes with formaldehyde at the air-water interface. The behavior of polyion complexes at the air-water interface and the surface structure of LB films was investigated by Brewster angle microscope (BAM) and scanning Maxwell-stress microscope (SMM), respectively. Also, the electrical properties for crosslinking in phenol-formaldehyde LB films have investigated by conductivity, dielectric constant and activation energy.

EXPERIMENTAL

Amphiphilic phenols, p-hexadecoxyphenol (p-HP), were synthesized

by the substitution reaction of hydroquinone with 1-iodohexadecane[3].

The surface pressure-area (π-A) isotherms were investigated by NL-LB200-MWC (Nippon Laser and Electronics Lab, Moving Wall Method; trough size: 80 mm ×585 mm). The morphology changes after spreading the monolayer and during the process of compression at the air-water interface were observed with a BAM (MiniBAM, Nanofilm Technologie GmbH.). The images of the LB films were obtained by using SMM imaging in the repulsive mode in air with a commercial system (Nanoscope III, Digital Instruments, USA). For the electrical properties of the LB films, a DC power supply and a Keithley 6517 electrometer were used to measure the current-voltage (I-V) characteristics. The capacitance of the MIM structure was measured by a HP 4192A impedance analyzer from 5 Hz to 13 MHz.

RESULTS AND DISCUSSION

Figure 1 shows surface pressure-area (π-A) isotherms of p-HP on the pure water and on aqueous formaldehyde. The monolayer of p-HP on pure water showed the collapse pressure of over 35 mN/m, while the monolayer of p-HP on aqueous formaldehyde revealed low collapse pressure of ca. 30 mN/m.

The π-A isotherms also showed different monolayer phases, i.e. the monolayer of p-HP on pure water showed the condensed phase, while the monolayer of p-HP on aqueous formaldehyde revealed the expanded phase. The isotherm on aqueous formaldehyde subphase shows the increased molecular area than those on pure water subphase. This difference could be due to formaldehyde attached to the Langmuir film being compacted under the monolayer.

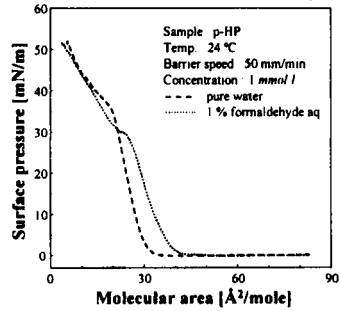

FIGURE 1. π-A isotherms of the p-HP polymer in the subphases of pure water and aqueous formaldehyde.

The phase behavior of the p-HP monolayer observed with BAM is shown in Figure 2. In the monolayer of p-HP on pure water, at a surface pressure of 0 mN/m, the domains started to fuse to one another (Fig. 2(a)) and formed a homogeneous monolayer until the collapse pressure (Fig. 2(c)). When the surface pressure reached the collapse pressure, it was observed that strong light was reflected from the whole monolayer surface (Fig. 2(e)). Finally, when the monolayer was compressed beyond the collapse point, the monolayer was broken in a direction parallel to the compression (Fig. 2(g)). In the monolayer of p-HP on aqueous formaldehyde, a homogeneous monolayer was formed through both the gathering process and the fusing of the domains

during compression (Fig. 2(b), (d)). When the surface pressure reached the collapse pressure, the p-HP monolayer on aqueous formaldehyde was locally destroyed and resulted in the formation and growth of domains with surface pressures as shown in Fig. 2(f) and Fig. 2(h).

These results can be interpreted as a growth of a polymeric monolayer complex containing attached formaldehyde. Compression of the monolayer decreases the distance between the formaldehydes and increases formaldehyde concentration. As the formaldehydes come into closer proximity, there is an increase in the van der Waals attraction and in the electrostatic repulsion between the formaldehydes.

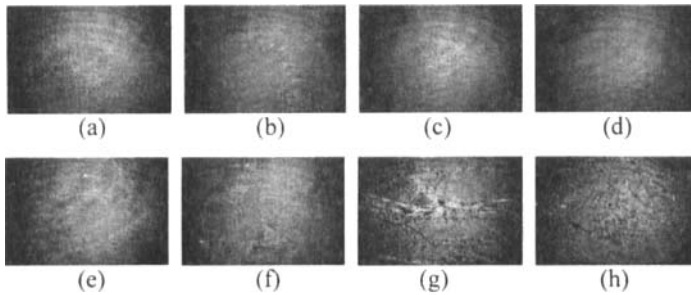

FIGURE 2. The BAM images of the p-HP monolayer: (a) 0 mN/m on pure water, (b) 0 mN/m on aqueous formaldehyde, (c) 15 mN/m on pure water, (d) 15 mN/m on aqueous formaldehyde, (e) 35 mN/m on pure water, (f) 30 mN/m on aqueous formaldehyde, (g) 45 mN/m on pure water, and (h) 45 mN/m on aqueous formaldehyde.

Figure 3 shows the SMM topography and surface potential images of p-HP LB films deposited on a silicon substrate. The Z type deposition was achieved at a surface pressure of 25 mN/m and its transfer ratio was 0.85 ~ 1.02. When the monolayer of p-HP was deposited on the Si wafer substrate from a pure water subphase, the large domains were seen as bright spots in the SMM images (Fig. 3(a), 3(b)). However, a good deposite of network film was found in the monolayer films and was transferred from the 1% aqueous formaldehyde subphase (Fig. 3(c), 3(d)). It is thought that the crosslinked structures were formed by reaction between p-HP and subphase of 1% aqueous formaldehyde. The substrate potential distribution was homogeneous over the film before the LB film transfer. The spatial distribution of the surface potential was, however, created after formation on the Si wafer surface with the crosslinked network monolayer because the surface potential would be different between the network film and the substrate. The surface potential between these two parts was attributed to the difference in the surface dipole moments due to the terminal CH_2- and CH_3- groups of the C-H, C-O and C=C chains, respectively.

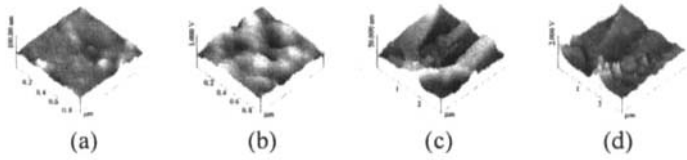

FIGURE 3. The SMM images of the p-HP LB films: (a) topography and (b) surface potential of p-HP LB film deposited on pure water, (c) topography and (d) surface potential of p-HP LB film deposited on aq. 1% formaldehyde.

Electrical properties for phenol-formaldehyde LB films deposited in the pure water (PW) and 1% aqueous formaldehyde (1AF), and heat-treated 1AF LB films (H1AF) have investigated such as conductivity, dielectric constant and activation energy. The heat treatment of LB films deposited in the 1AF worked for 24 hours in a vacuum in order to improve crosslinking reaction. We have provided evidence for the high insulating performance of the phenol-formaldehyde thin films by the LB method. The dielectric constant, activation energy and conductivity of their LB films was as follows: pure water > 1 % aq. Formaldehyde > heat treatment, in the current-voltage (I-V) characteristics (Table. 1). It is demonstrated that insulation properties of the crosslinked p-HP LB films were improved.

TABLE 1. Conductivity, dielectric constant and activation energy of p-HP LB films.

Condition	Conductivity	dielectric constant	Activation Energy
PW	$1.33 \sim 1.74 \times 10^{-14}$ S/cm	8.36 ~ 9.05	0.495 eV
1AF	$7.36 \sim 8.34 \times 10^{-15}$ S/cm	5.76 ~ 8.23	0.367 eV
H1AF	$3.76 \sim 4.76 \times 10^{-15}$ S/cm	2.47 ~ 2.73	0.264 eV

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