INTRODUCTION

Sugar surfactants are a group of environmentally benign surfactants. Sugar surfactants have attracted special interest because they are both biobased, as they are made from renewable materials such as plant-based sorbitol or mannitol, and readily biodegradable. Sugar surfactants in solution can also self-assemble into various types of aggregates, such as micelle, emulsions, and lyotropic liquid crystals. Now, most of the work focused on hydrocarbon-tailed sugar surfactants [1]. However, hydrocarbon surfactants generally have many \(-\text{CH}_2\)\(-\) groups in their hydrophobic portions, which have an intrinsically higher surface energy than methyl groups. Based on the general geometrical considerations, hydrocarbon-tailed sugar surfactants packs into micelle in dilute solution.

Siloxane surfactants have attracted special interest [2–4]. Compared to their hydrogenated counterparts, siloxane surfactants show much lower critical micellar concentrations, higher surface activity. In our previous study, we report that siloxane surfactants can more easily form bilayers, because of a higher rigidity of the siloxane chain compared to an analogous hydrocarbon chain [5, 6].

Here we have prepared a tetrasiloxane-tailed sugar surfactant Si4N2-LA:

![Chemical structure of Si4N2-LA](image)

These tetrasiloxane-tailed sugar surfactants self-assemble into a vesicular structure in aqueous solution due to the incorporation of a bulky tetrasiloxane moiety at the terminus of the hydrocarbon chain.

EXPERIMENTAL SECTION

**Materials.** \(N\-(2\text{-Aminoethyl})\-3\text{-aminopropyltetrasiloxane} \) was prepared according to the our previous reported procedure [7]. The purity was ascertained by \(^1\text{H NMR} \) in CDCl₃. All other chemicals were analytical grade and were used without further purification. The water used was redistilled from potassium permanganate.

**General method.** \(^1\text{H NMR} \) spectra were recorded on a Varian INOVA-400MHz spectrometer. Since TMS cannot be easily used as internal standard because of the overlapping with other methyl signals,
we used the residual protons of the solvent (DMSO) δ 1H = 2.50 ppm.

Equilibrium surface tension measurements. Surface tensions of aqueous solutions were measured with a Kruss 12 tensiometer equipped with a Wilhelmy-plate. The critical aggregation concentration (CAC) and surface tension at the CAC were determined from the break point of the surface tension and concentration curve.

Dynamic light scattering (DLS). Light scattering measurements were made using a spectrometer of Zeta Plus Particle Size Analysis (Brookhaven, USA). The scattering angle was set at 90°, and the intensity autocorrelation functions were analyzed using the methods of non-negatively constrained least-squares algorithm. The solutions were filtrated though 0.45 μm millipore to remove the dust before the experiment.

Transmission electron microscopy (TEM). The TEM images were obtained using a negative-staining method. A carbon Formvar-coated copper grid (300 mesh) was laid on one drop of the sample solution, and the excess solution was wiped away with filter paper. Then the copper grid was put onto one drop of phosphotungstic acid solution (2%) as the staining agent. The excess liquid was also wiped with filter paper. After drying, the samples were imaged under a JEM-100CXII electron microscope at a working voltage of 100 kV.

X-ray diffraction (XRD). Self-supported cast films were prepared by dispersing the solutions over pre-cleaned glass plates, and then air-drying these samples at room temperature. Finally, the plates were placed under low vacuum for 15 min. X-ray diffraction (XRD) studies were carried out using an X-ray diffractometer (Rigaku model D/MAX2500). The X-ray beam was generated with a Cu anode at 40 kV and 20 mA, and the wavelength of the Kα1 beam was 1.5406 Å. The X-ray beam was directed to the edge of film, and the scanning 2θ was recorded from 1° to 6°, using a step width of 0.01°.

Synthesis of Si4N2- LA. N-(2-aminoethyl)-3-amino propyltetrasiloxane (1.98 g, 0.005 mol), was dissolved in 50 mL of dry methanol. Lactobionic acid (0.893 g, 0.005 mol) was added, and the mixture was heated to reflux temperature for 24 h. After cooling to room temperature and evaporation of the solvent the solid residue was gently crushed, washed several times with hexane at 25°C, and dried under reduced pressure to a constant mass. The white solid 2.61 g (yield 91%). IR (KBr): 3310 cm –1 (ν(O–H)), 1650 cm –1 (ν(C=O) in amide), 1468 cm –1 (ν(C=H) in –CH2–), 1259 cm –1 (ν(Si–Me3)), 1035–1150 cm –1 (ν(C–O), δ(O–H), and ν(Si–O–Si)), 840 cm –1 (ν(Si–Me3)), 760 cm –1 (ν(Si–Me3)).

1H NMR(D2O, ppm) δ: 0.085 (s, 27H, Si(CH3)3 · 3), 0.405 (t, 2H, SiCH2), 1.422 (2H, SiCH2CH2CH2), 2.466 (2H, –CH2NHCH2CH2N), 2.555 (2H, –CH2NHCH2CH2N), 3.162 (2H, –CH2NHCH2CH2N), 3.30–4.26 (m, 13H, protons of sugar group), 7.641 (s, 1H, proton of amide).

Elemental analysis calcd for C26H60N2O14Si4: C, 42.37; H, 8.20; N, 3.80; Found: C, 42.31; H, 8.23; N, 3.75.

RESULTS AND DISCUSSION
Synthesis and characterization. The synthesis of Si4N2-LA is shown before. The amidation of the primary amine proceeded in the presence of the methanol at reflux temperature. The reaction conditions were moderate. In the cases the amide of the primary amine function was formed, whereas the secondary amino group did not yield a considerable amount of by-products. The amidation of amine function has been reported previously by other author [8]. The structure of amide was confirmed by proton nuclear magnetic spectroscopy (Fig. 1). The protons of the carbon adjacent to the free amine (CH2—NH2) are seen at 2.80 ppm, while the same protons after amide formation through coupling with the lactone (CH2—NHCO) shift to 3.16 ppm. This shift is observed in NMR data, verifying the absence of free amine groups. In amide the hydrogen of C(h) and C(i) are adjacent to the carbonyl function and shows a low-field shift, 4.09 and 4.00 ppm respectively. The signals of the hydrogen atoms in lactobionic acid were assigned according to