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Liquid crystalline behaviour of hydrogen-bonded complexes of alkoxycinnamic acids with octyloxystilbazole

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Hydrogen-bonded liquid crystalline complexes have been obtained through 1 : 1 (molar ratio) complexation of 4-*n*-alkoxycinnamic acids (*n*CNA: $n = 4$, 8, 10, 12, where *n* is the number of carbons in the alkyloxy chain) and *trans*-4-octyloxystilbazole (8Sz). These hydrogen-bonded complexes (*n*CNA8Sz) form stable mesophases. The mesomorphic range was extended by the mixing of complexes. Hexatic modification of smectic B ($\mathrm{SmB_h}$), smectic C (SmC), smectic A (SmA), and nematic mesophases of these complexes were determined by a combination of X-ray diffraction and polarizing optical microscopy. Transitions between the various smectic phases were deduced from the temperature-dependent layer spacing of *n*CNA8Sz. The layer spacing of these complexes in the $\mathrm{SmB_h}$ and SmA phases gradually increased with increasing alkoxy chain length. The favouring of smectic phases in these complexes is believed to originate from the increment of polarity of the mesogen by intermolecular H-bonding.

1. Introduction

The formation of supramolecular structures using small molecular components is of great chemical and biological interest [1]. In liquid crystals, mesomorphism results from the proper combination of molecular interactions and the shape of molecules. Conventional low molar mass liquid crystals comprise molecules consisting of a semi-rigid anisometric core attached normally to one or two alkyl chains [2]. In these molecules, the mesogenic core often contains phenyl rings connected via short unsaturated linkages. In recent years, however, increasing research activity has focused on materials in which the core is assembled via non-covalent bonding [3–5].

Hydrogen bonding is one of the key interactions for chemical and biological processes in nature due to its stability, directionality, and dynamics. For molecular aggregates, hydrogen bonding plays an important role in the association of molecules. The first compounds found to exhibit liquid crystalline behaviour due to hydrogen bond formation were carboxylic acids [6]. These compounds dimerize through intermolecular hydrogen bonds leading to a lengthening of the rigid-rod moiety, which in turn induces liquid crystallinity. Many liquid crystal systems containing hydrogen bonds that function between identical molecules have been reported [7–9].

A new type of thermotropic supramolecular liquid crystal, obtained by molecular recognition processes through intermolecular hydrogen bonds, was reported by Kato and Fréchet [10] and Lehn and co-workers [11]. The novelty of these studies lies with the use of hetero-intermolecular bonds in order to assemble the mesogenic core, i.e. the two interacting molecules are not identical. After these findings, many liquid crystalline materials, including low molar mass liquid crystals and liquid crystalline polymers, have been prepared using the intermolecular hydrogen bond [12–26].

Mixtures of unlike hydrogen bonded molecules producing liquid crystals frequently involve donor molecules derived from benzoic acids, with acceptor molecules derived from pyridine : alkyloxystilbazole [23]. As these systems had proved to be mesomorphic, we modified the complexes to include an additional conjugated C=C bond, thus enhancing the structural anisotropy. It was reported that benzoic acids with $\mathrm{p}K_\mathrm{a} \sim 4$ can yield H-bonded complexes with pyridine derivatives [27]. The $\mathrm{p}K_\mathrm{a}$ of benzoic acid is 4.19, while that of cinnamic acid is 4.44 at 25°C [28]; thus cinnamic acids, like benzoic acids, will be able to complex with various pyridine derivatives.

In this paper we describe the phase behaviour of hydrogen-bonded liquid crystalline complexes of *n*-alkoxycinnamic acids with *trans*-4-octyloxystilbazole. Various mesophases, especially hexatic modification of the smectic B phase, were elucidated through temperature-dependent layer spacing of the complexes. These results show that the hydrogen-bonded mesogen behaves as a rod unit like a covalent bonded mesogen.

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2. Experimental

4-*n*-Alkoxycinnamic acids (*n*CNA: $n = 4$, 8, 10, 12, where *n* is the number of carbons in the alkoxy chain) were prepared according to known procedures [29, 30]. The compounds were recrystallized from ethanol and then from ethyl acetate; the transition temperatures were in agreement with the published values. *Trans*-4-octyloxystilbazole (8Sz) was synthesized as described earlier [31]. The complexes of *n*CNA with 8Sz examined in the present study were prepared by the evaporation technique [32] from THF solution containing equimolar amounts of H-bond donor and acceptor moieties. The complex of *n*CNA with 8Sz is denoted as *n*CNA8Sz.

Infrared (IR) spectra were obtained using a Mattson Infinity Gold series FTIR spectrophotometer. A Perkin-Elmer DSC-7 differential scanning calorimeter was used to determine the thermal transitions, with heating and cooling rates of 10°C min^{-1}. A Leitz Orthoplan-pol polarizing optical microscope (magnification: 140×) equipped with a Leitz Microscope heating stage 350 was used to analyse the anisotropic texture. X-ray diffractograms were obtained with synchrotron radiation at the 3C2 X-ray beam line ($\lambda = 0.154$ nm) at Pohang Accelerator Laboratory, Korea. *In-situ* X-ray measurements were performed in the transmission mode at various temperatures. The sample holder was heated with two cartridge heaters, and the temperature was monitored. A 7 µm thick Kapton film window was used to contain the samples in the fluid liquid crystalline states, and the scattered intensity from the Kapton window was subtracted.

3. Results and discussion

3.1. *Formation of nCNA8Sz complexes*

The thermal properties of *n*CNA compounds are given in table 1. All of the *n*CNAs exhibit liquid crystalline behaviour due to dimerization of the alkoxycinnamic acid unit, as shown in figure 1 (*a*). It has been reported that 8Sz exhibits enantiotropic smectic E and B phases [31].

The H-bonded mesogenic structure obtained through complexation between H-bond donor and acceptor moieties designed for the present study is shown in figure 1 (*b*). The technique used in the formation of these complexes involves the dissolution of both components in THF, which causes the breaking of existing H-bonds within the *n*CNA dimer; subsequent slow evaporation of the THF results in the self-assembly of the final complexes.

Table 1. Thermal transitions of *n*CNA series: data obtained from the second heating scan. Temperatures in °C; enthalpies (in parentheses) in kJ mol^{-1}.

Compound	Phase transitions
4CNA	Cr 154(51.5) N 185(8.8) I
8CNA	Cr 141(43.1) N 172(8.3) I
10CNA	Cr 131(39.6) S 150(4.9) N 169(10.4) I
12CNA	Cr 127(40.6) S 156(5.3) N 164(13.3) I

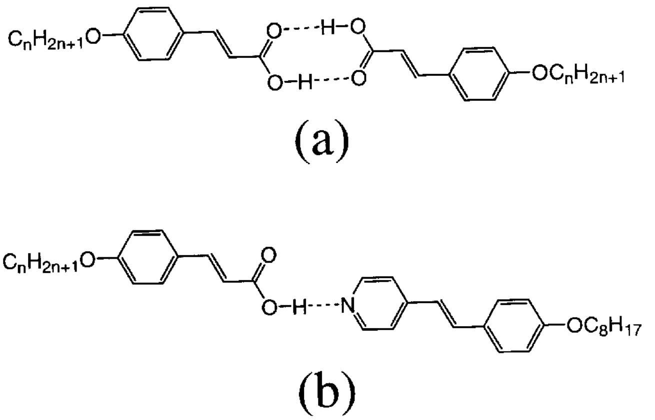

Figure 1. Supramolecular structures of (*a*) *n*CNA, (*b*) *n*CNA8Sz.

The formation of the H-bonded liquid crystal complexes shown in figure 1 (*b*) was confirmed by IR spectroscopy. Figure 2 shows the IR spectra of 10CNA8Sz at room temperature. The IR spectra showed the characteristic stretching bands (two bands centred around 2500 and 1900 cm^{-1}) resulting from self-association of carboxylic acid and pyridine through intermolecular hydrogen bonding [29].

3.2. *Mesomorphic properties of nCNA8Sz complexes*

*n*CNA8Sz complexes show enantiotropic liquid crystalline behaviour, as revealed by differential scanning calorimetry (DSC) and polarizing optical microscopy (POM). All the complexes behave similarly to the single

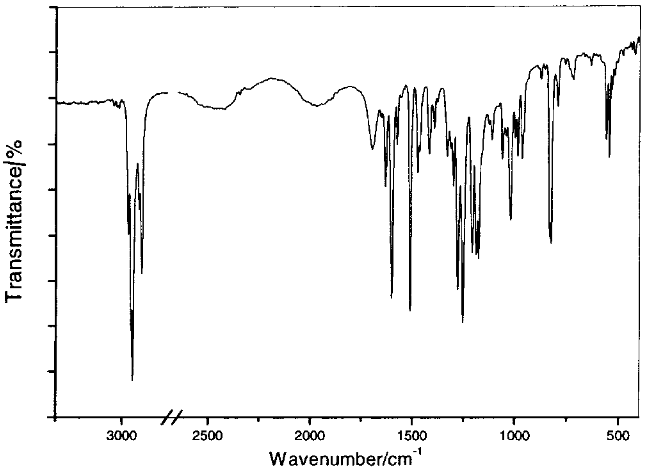

Figure 2. Infrared spectra of 10CNA8Sz at room temperature.

mesogenic component and show clear phase transitions. The detailed phase behaviour of the complexes, however, is different from those of each of the H-bond donor and acceptor compounds. The thermal behaviour of the complexes confirms the selective formation of H-bonded mesogens, as shown in figure 1 (*b*). The phases and transition temperatures of these compounds are summarized in table 2. The enthalpy changes of smectic C (SmC)–smectic A (SmA) transitions for *n*CNA8Sz complexes with $n \geqslant 8$ are too small to be observed in DSC thermograms; in such cases, the transition temperature was determined by X-ray diffraction (XRD) measurement. A comparison of tables 1 and 2 shows that the mixing of complexes extends the mesophase range. For example, the mesomorphic range of 10CNA8Sz is extended to 63°C as compared with 38°C for 10CNA and 13°C for 8Sz.

10CNA exhibits nematic (N) and smectic C phases. On cooling from the isotropic liquid, typical schlieren texture can be observed, figure 3 (*a*), characteristic of a nematic phase [34]. In contrast, the complexation of 8Sz to 10CNA induces the formation of smectic mesophases. Figure 3 (*b*) is a blurred schlieren texture of 10CNA8Sz, which is characteristic of a smectic C phase [33].

Figures 4 (*a*) and 4 (*b*) illustrate the phase behaviour for the *n*CNA series and *n*CNA8Sz complexes, respectively. A comparison of figure 4 (*b*) with figure 4 (*a*) shows that smectics are favorable phases in the *n*CNA8Sz complexes. For example, the *n*CNA series show the nematic phase up to 8CNA, while *n*CNA8Sz complexes display smectic phases from 4CNA8Sz on. Similar phenomena were observed in the complexes of 4,4′-bipyridine with *n*-alkoxybenzoic acids or *n*-alkoxycinnamic acids [25, 26]. Our speculation is that in *n*CNA8Sz, the complexation of 8Sz induces a much stronger H-bond than does the acid dimer; the polarity of the *n*CNA8Sz mesogen must be larger than that of *n*CNA. The complexation-increased interaction energy density between the polar rod and non-polar chain may have given rise to the stabilization of the microphase-separated lamellar structure.

Table 2. Thermal transitions of *n*CNA8Sz complexes: data obtained from the second heating scan. Temperatures in °C; enthalpies (in parentheses) in kJ mol^{-1}.

Complex	Phase transitions
4CNA8SZz	Cr 114(28.1) Cr$_2$ 118(3.2) SmA 150(1.1) N 164(4.2) I
8CNA8S	Cr 112(28.6) SmC 142(—) SmA 158(1.2) N 162(11.3) I
10CNA8Sz	Cr 95(57.1) SmB$_h$ 119(3.7) SmC 133(—) SmA 158(12.9) I
12CNA8Sz	Cr 96(53.0) SmB$_h$ 116(2.8) SmC 140(—) SmA 156(12.5) I

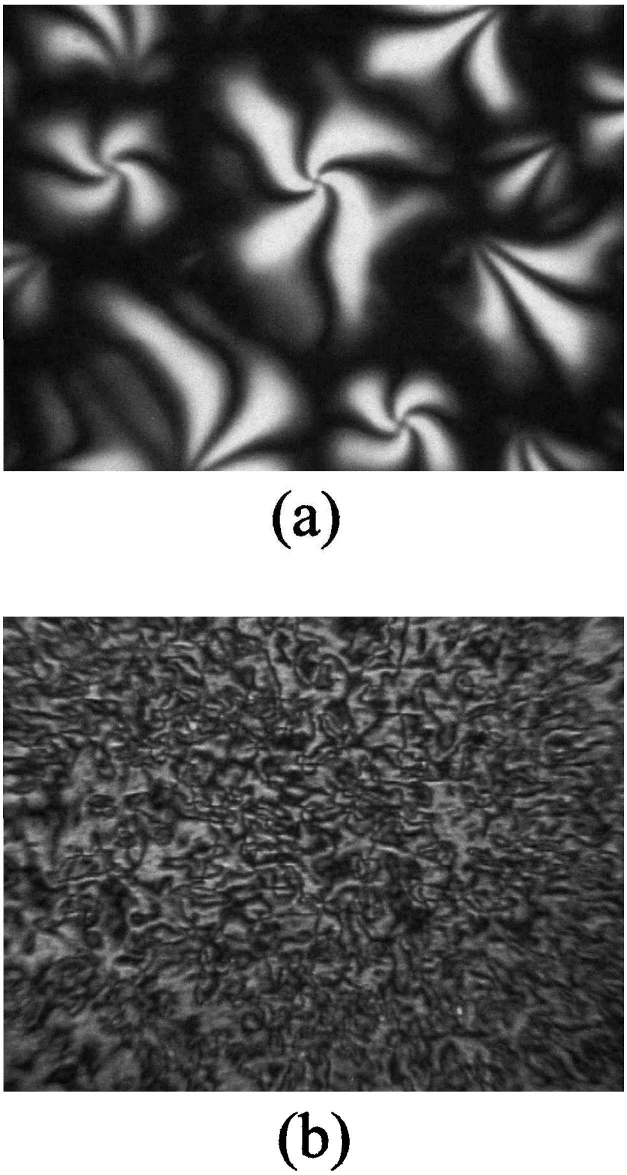

Figure 3. Representative polarized optical micrographs (140X) of the texture exhibited by (*a*) the nematic phase of 10CNA at 160°C on cooling, (*b*) the smectic C phase of 10CNA8Sz at 126°C on cooling.

3.3. *X-ray studies of nCNA8Sz complexes*

To corroborate the POM determination of the molecular packing structure, the mesophases of *n*CNA8Sz complexes were investigated by XRD. Figure 5 displays representative XRD patterns of 10CNA8Sz at various temperatures. Figure 5 (*a*) shows two Bragg reflections at reciprocal space ratios of 1 : 2 in the small angle region. This ratio, together with sharp reflections in the wide angle region as shown in figure 5 (*e*), indicates that 10CNA8Sz is a lamellar crystal at ambient temperature. The diffraction pattern of 10CNA8Sz recorded at 118°C,

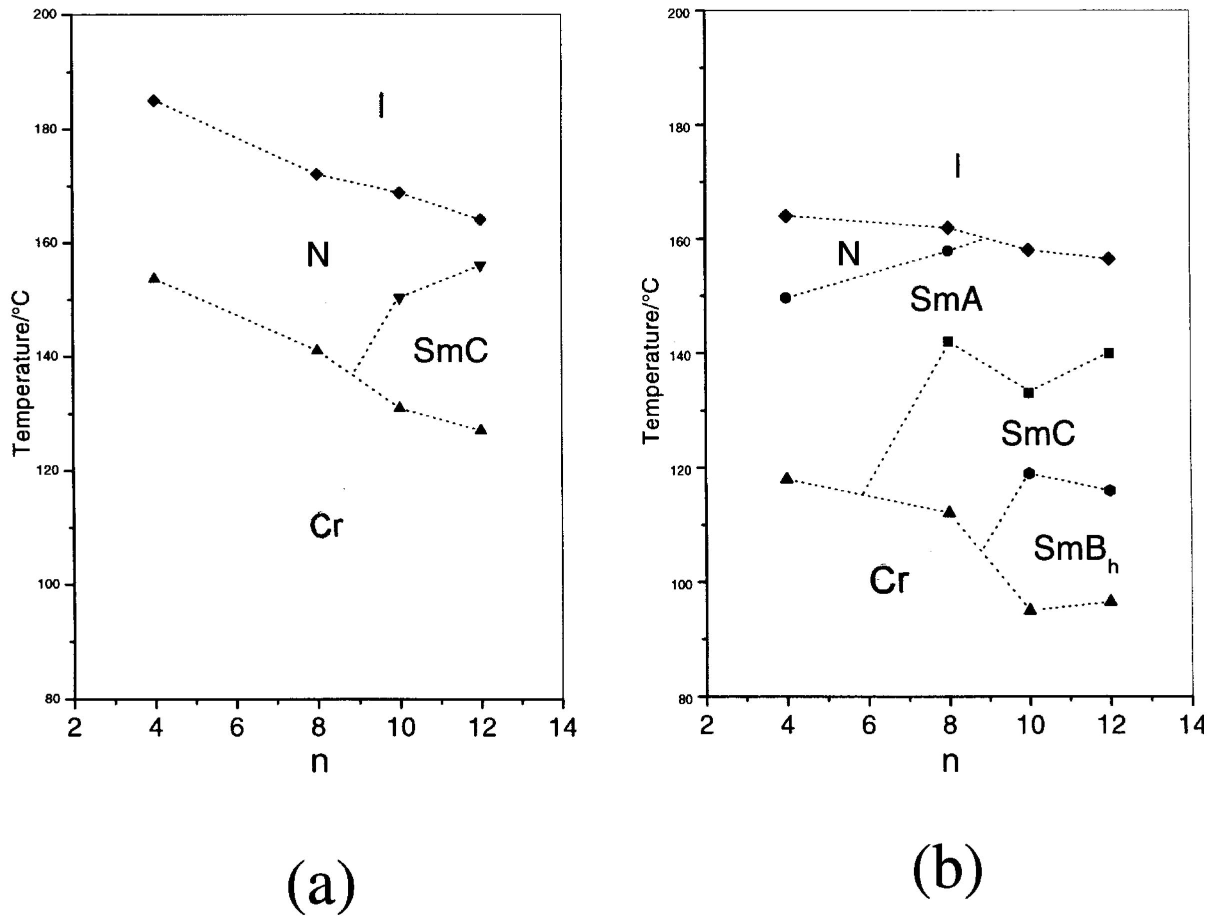

Figure 4. Dependence of the transition temperature of (*a*) *n*CNA compounds and (*b*) *n*CNA8Sz complexes on the number of carbon atoms in the alkoxy chains.

figures 5(*b*) and 5(*f*), indicated the presence of an ordered smectic phase. It contains two sharp equidistant reflections in the small angle region, related to the smectic layering of the molecules, and a unique, rather sharp reflection in the wide angle region at 0.43 nm. This kind of small angle reflection represents a layer spacing in the mesophase structure, and the reflections appearing in the wide angle region may be attributed to the structural order within the layer structures [34–36]. On heating from its higher order smectic mesophase, the sharp reflection disappeared and a diffuse halo was visible in the wide angle region as shown in figure 5(*g*). Although there is a broad amorphous halo in the wide angle region, the half-width Δq (scattering vector $q = 4\pi \sin \theta/\lambda$) of the wide angle halo increases from 2.4 mm^{-1} (SmA) to 3.6 nm^{-1} (isotropic). This result may suggest that there is short range ordering of the mesogens in the smectic A liquid crystalline phase. Figure 5(*c*) depicts the small angle XRD pattern at 140°C. The ratio of the positions of the two sharp peaks denotes that 10CNA8Sz exhibits a lamellar structure for this mesophase. 10CNA8Sz undergoes isotropization at 158°C as shown in figures 5(*d*) and 5(*h*).

In order to identify the character of the various smectic phases observed, a systematic X-ray measurement of *n*CNA8Sz was carried out in the temperature range from room to 160°C. Plots of temperature-dependent layer spacing for *n*CNA8Sz complexes are shown in figure 6. The layer spacing for 4CNA8Sz decreases steadily with increasing temperature, which is characteristic of a smectic A phase; thus, 4CNA8Sz has SmA and N phases. On the other hand, 8CNA8Sz displays an increase in layer spacing as a function of increasing temperature, which becomes almost level at the transition to the SmA phase. From this result, it can be concluded that 8CNA8Sz has SmC, SmA, and N phases. Besides the SmC and SmA phases, a higher order smectic phase was observed for *n*CNA8Sz ($n \geqslant 10$) complexes. Close inspection of the data shows a steady, though very weak, decrease of the layer spacing throughout the temperature range of this

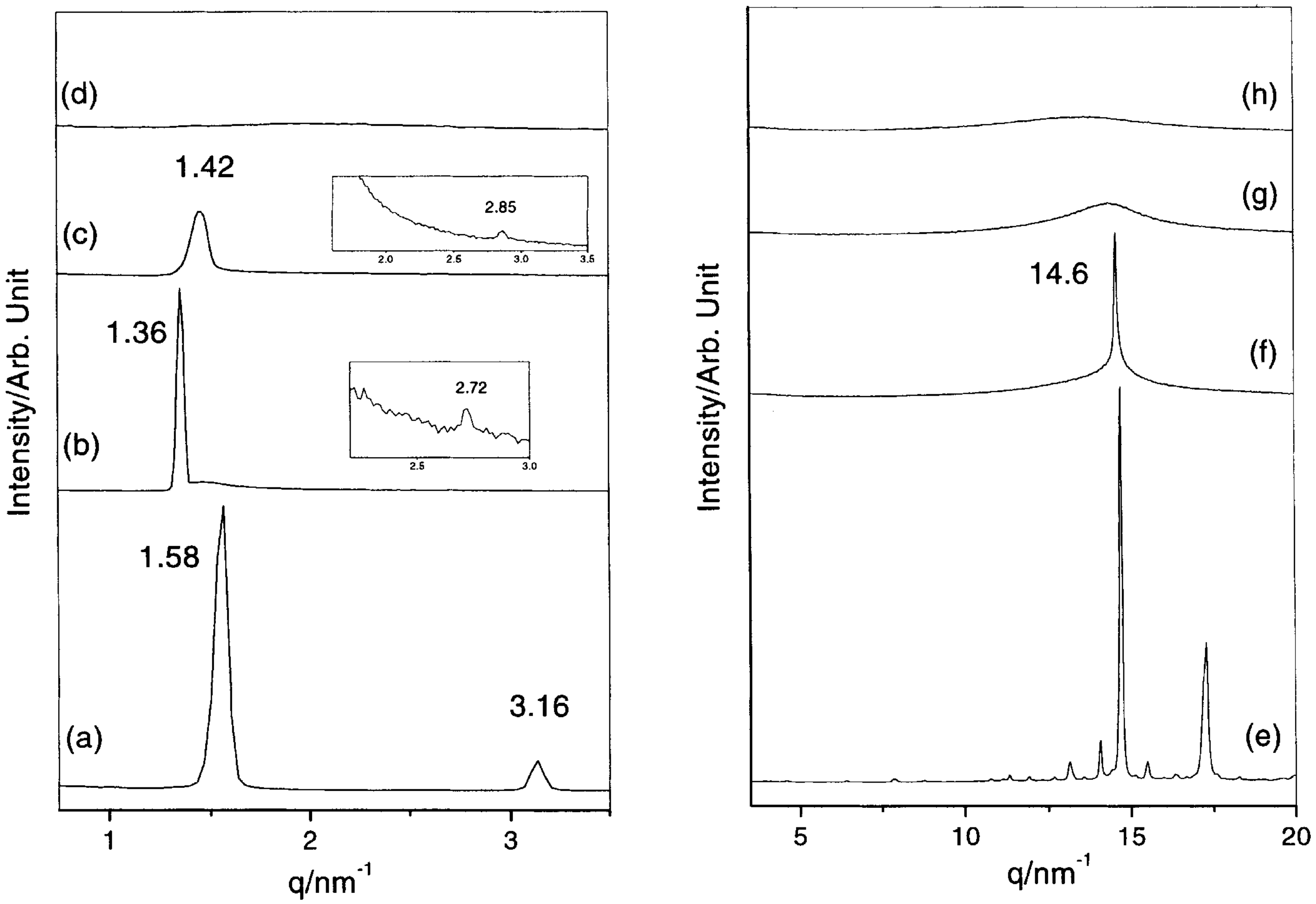

Figure 5. X-ray diffractograms measured at different temperatures plotted against $q(=4\pi \sin\theta/\lambda)$ for 10CNA8Sz: (*a*) small angle and (*e*) wide angle XRD of the crystalline phase at room temperature; (*b*) small angle and (*f*) wide angle XRD of the hexatic modification of smectic B phase at 118°C; (*c*) small angle and (*g*) wide angle XRD of the smectic A phase at 140°C; (*d*) small angle and (*h*) wide angle XRD of the isotropic phase at 175°C.

mesophase. This decrement, together with the sharp reflection in wide angle XRD pattern, indicates that the higher order smectic phase is a hexatic modification of the smectic B phase (SmB_h).

The existence of a SmB_h phase for 10CNA8Sz could also be demonstrated by POM. On cooling, a dark texture was observed by POM in the SmA phase temperature region. A dark texture was also observed by POM between birefringent SmC and crystalline textures. These isotropic textures under a homeotropic alignment indicate the optical uniaxial nature of the molecular arrangement in the SmA and SmB_h phases [34]. Our examination of SmA and SmB_h phases depends greatly upon XRD because we could observe only homeotropic textures. However, the textures could be clues to the existence of a non-tilted smectic phase. So, on heating, *n*CNA8Sz complexes have SmB_h, SmC, and SmA phases in the range of $n = 10, 12$. The layer spacing of *n*CNA8Sz complexes in their SmB_h and SmA phases gradually

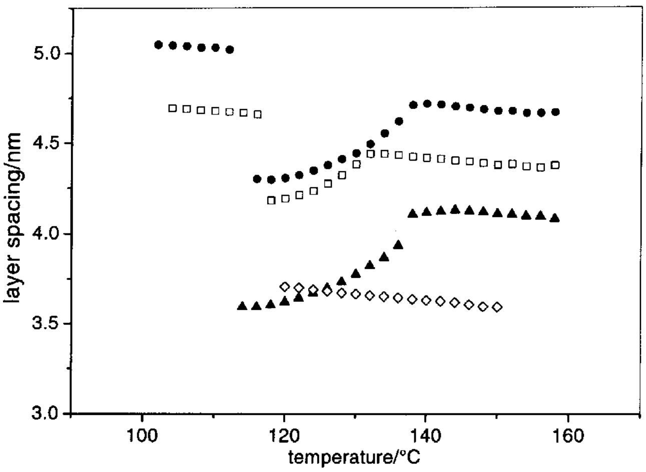

Figure 6. Temperature dependence of the layer spacing of 4CNA8Sz (open diamond), 8CNA8Sz (solid triangle), 10CNA8Sz (open square), and 12CNA8Sz (solid circle).

increases with increasing alkoxy chain length, as shown in figure 6.

Figure 7 displays the packing structures in *n*CNA8Sz complexes in their smectic phases. In the SmB_h phase, the layer spacing is larger than that of any other smectic phase. This behaviour is believed to originate from the shortest rod–rod distance of *n*CNA8Sz in the SmB_h phase. The paraffinic chains are stretched out when the rod–rod distance becomes shorter. At the $SmB_h \rightarrow SmC$ transition, the molecular interactions lose their efficacy, and molecules tilt drastically in order to relax the elastic constraints imposed upon the alkyl chains [37]. With the increase of temperature, the tilt of the molecules with respect to the layer normal decreases, thus causing the increase in layer thickness for the SmC phase. So the tilt angle of H-bonded complexes in the SmC phase is strongly temperature dependent, and decreases to zero at the phase transition to a smectic A phase. In the smectic A phase, the layer spacing decreases slowly with increasing rod–rod distance on heating, as shown in figure 6 [38]. From the temperature-dependent layer spacing behaviour, it can be concluded that the H-bond complexes behave like conventional calamitic (rod-like) mesogens. The layer spacings of *n*CNA8Sz complexes in the smectic mesophases are in the range of 3.6–5.0 nm, and the length of the rod unit of *n*CNA8Sz complexes is calculated to be 2.3 nm. Therefore, we speculate that the packing structure of *n*CNA8Sz in the smectic phases is a monolayer lamellar as shown in figure 7.

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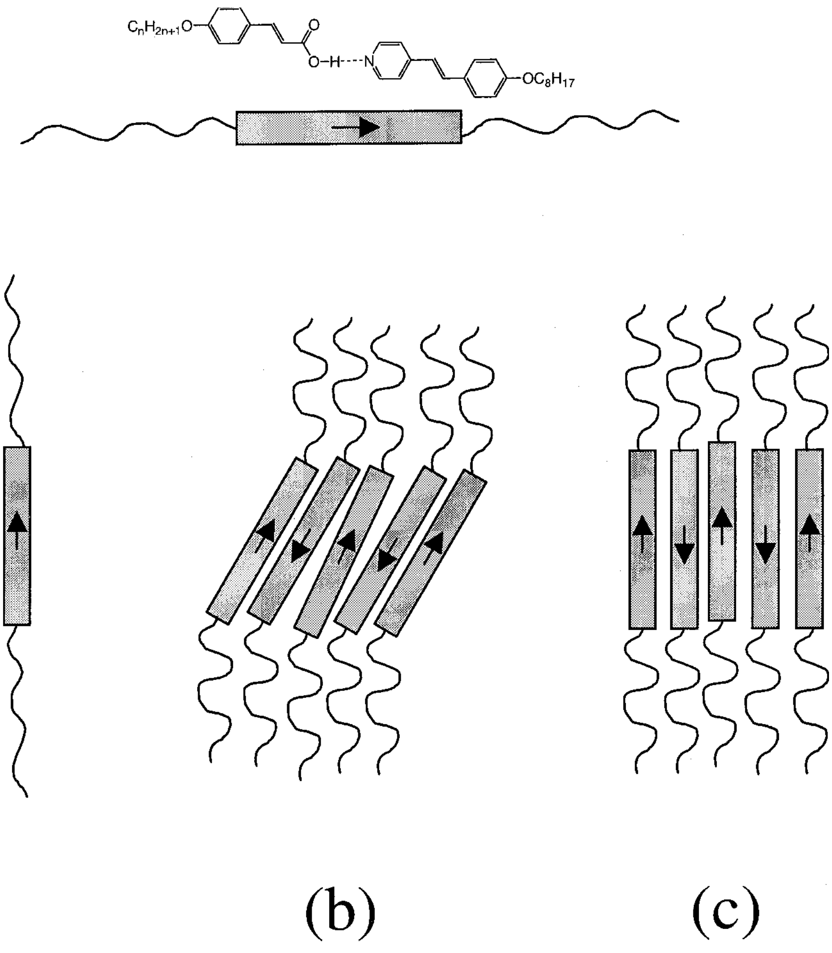

Figure 7. Schematic representation of the structures of *n*CNA8Sz in the (*a*) hexatic modification of smectic B phase, (*b*) smectic C phase, (*c*) smectic A phase.

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