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Liquid crystal fibers of bent-core molecules

Antal Jákli
Liquid Crystal Institute, Kent State University, Kent, Ohio 44242

Daniel Krüerke
Stranski-Laboratorium, Technical University, 10623 Berlin, Germany

Geetha G. Nair
Centre for Liquid Crystal Research, P.O. Box No. 1329, Bangalore 560013, India

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Although the liquid-crystal research is well established in science, there are newly emerging exciting systems, that deserve extensive basic studies. One of these areas is the research of the bent-shaped molecules (so-called ‘‘banana liquid crystals’’), which have delicate chirality and polarity properties. In this paper we show that these materials also have very unusual rheological features, such as the formation of stable fluid fibers and bridges. Under electric fields, these objects present striking mechanical effects, such as horizontal and transversal vibrations. Studies indicate that the research of banana-liquid-crystal fibers may lead to new type of artificial muscle systems.

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I. INTRODUCTION

Fibers are very important objects in our life. Examples include natural silk spinned by silkworms and spiders, and synthetic fabrics such as nylon, polyester, or optical fibers for communication networks. Fibers can be drawn only from viscous fluids that harden during the pulling process. The hardening can be achieved either by cooling, such as in glass fibers or by losing water, e.g., in spinning spider silks. As surface tension causes fluids to have as little surface as possible for a given volume, Newtonian fluids (described by a constant viscosity) cannot form fibers, but only short ‘‘bridges’’ with lengths smaller than their circumference (Rayleigh-Plateau instability) [1]. In the case of non-Newtonian materials, where the viscosity depends on the strain, long slender columns of liquids can be stabilized during the pulling for high strain rates characterized by sufficiently large Deborah number [2]: $\mathrm{De}>0.5$ ($\mathrm{De}=\dot{\varepsilon}/\tau$, where $\dot{\varepsilon}$ is the strain rate and τ is the relaxation time of a deformation). In such case, a strain hardening occurs leading to a homogeneous extensional deformation and a uniform column in the midregion.

Liquid crystals are complex fluids [3] with wide range of viscous and elastic properties. Liquid-crystalline polymers are viscoelastic and can easily form fibers, just as conventional isotropic polymers. The stability of nematic liquid-crystalline polymer fibers was extensively studied theoretically [4,5] and experimentally. It was found that liquid-crystalline polymer fibers or tubes have very high oxygen and water vapor permeability, which makes them increasingly important in packaging as ‘‘superbarrier’’ materials [6]. Recently, it was shown [7] that even the spider silks have nematic liquid-crystalline structures in the early duct portion of the silk-producing gland.

The fiber forming ability of low molecular weight liquid crystals is determined basically by the fluid dimensionality. Columnar liquid-crystalline phases of disk-shaped molecules are one-dimensional fluids and they can form as long as $L=0.2$ mm and as narrow as $D=1.5\ \mu$m diameter stable strands [8], which means that their slenderness ratio ($S=L/D$) can be as large as 10^2. Smectic (2D fluid) and nematic (3D fluid) liquid crystals do not form fibers, but only bridges that collapse at slenderness ratios of $S=4.2$ and $S\approx\pi$, respectively [9]. Smectic liquid crystals of rod-shaped molecules (‘‘calamitic smectics’’) generally tend to form thin films [10], similar to membranes. Accordingly, it was a great surprise to find recently that bent-shape molecules (‘‘banana smectics’’) may form stable fibers instead of films. [11,12].

Liquid crystals of banana-shaped molecules attracted considerable interest when it was observed that they are ferroelectric [13] and their structure is chiral without possessing chirality on molecular level [14]. After the first few years of intense studies seven banana-liquid-crystal structures (phases) were distinguished and were tentatively labeled as B_1-B_7 [15], although these names refer only to typical textures and switching properties. The most common phase is B_2, which is a fluid smectic phase, where the molecular plane tilts away from the layer normal. The layer polarization is along the layers and possess mainly antiferroelectric-type switching under electric fields of $E>3$–6 V/μm. The B_7 materials, where the first banana liquid-crystal fibers were found [11,12] form spectacular chiral superstructures [11,16] in an isotropic melt. They have modulated layer structures [17], and it seems [18,33], that they are the least symmetric fluids with C_1 symmetry.

In this paper, we concentrate on the fiber formation properties of the banana liquid crystals, and show that, not only the B_7 materials, but also the B_2 phases and, to some extent, even the banana nematics are able to form fibers.

II. EXPERIMENTAL CONDITIONS

The fiber-drawing device used in our experiments consists of two needles with micro-positional accessories built on a

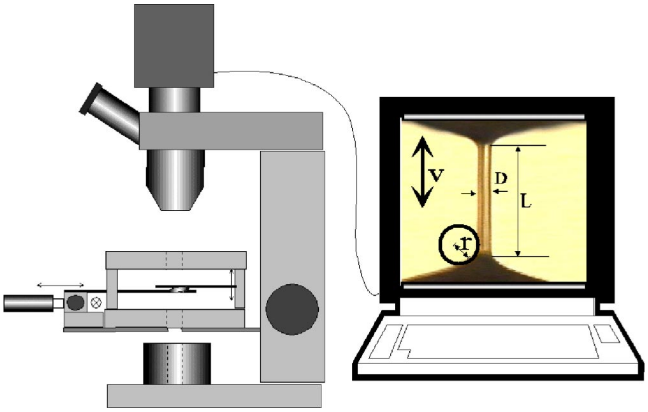


FIG. 1. Experimental setup and a typical fiber seen in microscope with the illustration of the main parameters of the fibers. The curvature radius of the neck of the fiber is r, the diameter is D, and the length of the fiber is L. The speed of the pulling was in the range of $0<v<0.1$ mm/s.

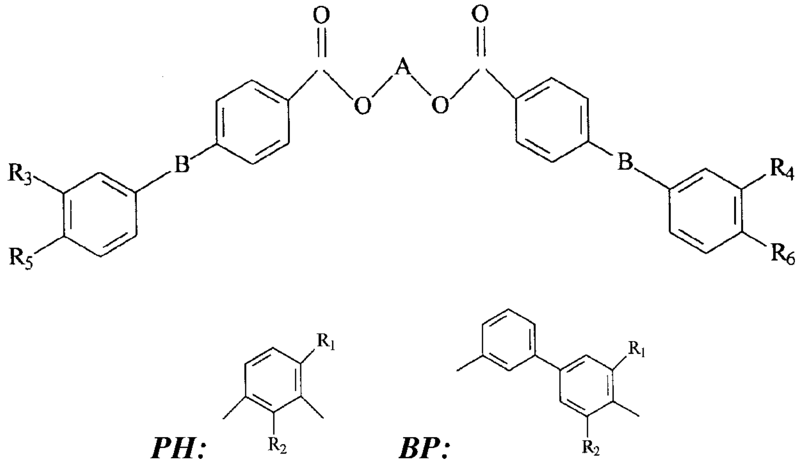
hot stage (HS1 from Instec. Inc.) that allows temperature regulation with 0.1 °C precision. A drop of a liquid-crystalline material is placed on top of the needles when they touch each other. The distance between the pins can be varied by micrometer screws in two directions with speed up to 0.1 mm/s. Optical observations of the suspended liquid-crystal bridges and fibers are carried out by a polarizing microscope (BX60 from Olympus) equipped with a color charge-coupled device camera. (Fig. 1).

The molecular structures and the phase sequences of the materials studied by us are shown in Fig. 2.

III. RESULTS

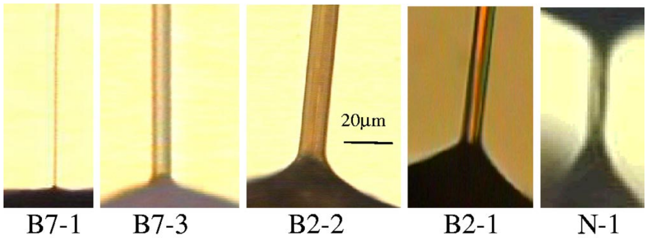
For small strains, the materials behave similar to Newtonian fluids, i.e., as the distance between the needles increases, they develop concave shapes with the decreasing curvature radius. Typically at $D \sim 15$–$20\ \mu$m, a homogeneous elongation deformation occurs and the diameter of the filament remains constant, provided that the fluid reservoirs at the end plates have enough materials. On further increasing the length, the diameter is decreasing in about 1.5–2 μm discrete steps. Fibers with diameters larger than 6–8 μm show no extinction when rotating between crossed polarizers. We found that they are not single filaments, but bundles of twisted coiling strings. The B_7 fibers remained stable until the diameter decreased to 1.5–3 μm. In this case, the filament appears to be uniform and uniaxial with extinction direction parallel to the filament axis. The B_2 fibers remained stable only in bundles at diameters of about 10 μm. (Fig. 3).

It was observed that dust particles stuck to the bundles of fibers rotate during the change of length, and the direction is opposite for increasing and decreasing end plate distances. These illustrate the spontaneous chirality of the banana liq-



Code	A	B	R_1	R_2	R_3	R_4	R_5=R_6	Phase sequence in (°C)
B7-1	Ph	NCH	H	NO_2	H	H	$C_8H_{17}O$	Cr 116 B_7 177 I
B7-2	Ph	NCH	H	H	F	F	$C_{10}H_{21}O$	Cr 110 B_2 147 B_7 160 I
B7-3	Ph	NCH	H	H	F	F	$C_8H_{17}O$	Cr 112 B_2 174 B_7 206 I
B2-1	Ph	NCH	H	H	H	H	$C_{12}H_{25}O$	140 B_2 170 I
B2-2	Ph	NCH	Cl	H	H	H	$C_{14}H_{29}$	Cr 68 B_2 127 I
B2-3	BP	NCH	Cl	H	H	H	$C_{14}H_{29}$	Cr 58 B_2 160 I
N-1	Ph	OOC	Cl	H	H	H	$C_9H_{19}O$	Cr 83 N 127 I

FIG. 2. Structures and phase sequences of the studied bent-shaped molecules having liquid crystalline mesophases. The central parts A of the molecules of the present studies were either a phenyl group (Ph) or a biphenyl group (BP). $B=-N=CH$(NCH) or $-OOC$ $-$(OOC). R_1, R_2, R_3, R_4 are independently hydrogen or a halogen, or cyanide, and R_5 and R_6 are independently C_8-C_{16} alkyl or C_8-C_{16} alkyl-oxy. In the phase sequences "I" stands for isotropic fluid and "Cr" stands for crystal. $B7-1$ [11,16] has only a B_7 mesophase; $B7$-2 [32,18] and $B7$-3 [33] have both B_7 and B_2 mesophases; $B2$-1 [15], $B2$-2 [15], and $B2$-3 [34] have B_2 banana liquid crystal phases; and N-1 [20] has a nematic phase.



Code.	L_{max}(mm)	D_{min}(μm)	$S_{max} = L_{max}/D_{min}$
B7-1	8	2	4000
B7-2	9	3	3000
B7-3	3	3	1000
B2-1	1	8	125
B2-2	0.5	8	60
B2-3	0.3	10	30
N-1	0.3	20	15

FIG. 3. Typical fibers at the neck of some banana materials, and the list of the main parameters of the fibers: L_{max} is the maximum length, D_{min} is the minimum diameter, and S_{max} is the largest slenderness ratio obtained.

uid crystals. When the distance between the end plates decreases abruptly, typically a bent or twisted pattern appears, but the string straightens in a few seconds again when keeping the end plates distance constant. This demonstrates their viscoelastic nature.

It is remarkable that fibers in the B_2 and B_7 textures [see Fig. 4(a)] are thinner than conventional extruded fibers, and completely stable in air with slenderness ratio as high as 4000.

Unlike all low molecular weight nematic liquid crystals, fibers could be pulled even in the nematic phase of bent-shaped molecules (banana nematic), such as material $N-1$ [20]. Although they become unstable and rupture in a few minutes, the slenderness ratio is much larger than those observed in nematic liquid crystals of rod-shaped molecules [9]. This behavior is typical for viscoelastic fluid fibers [19], and recent light scattering and electro-optic measurements [22] indeed indicate that the banana-nematic material $N-1$ can be considered as a fluid agglomerate of smectic clusters.

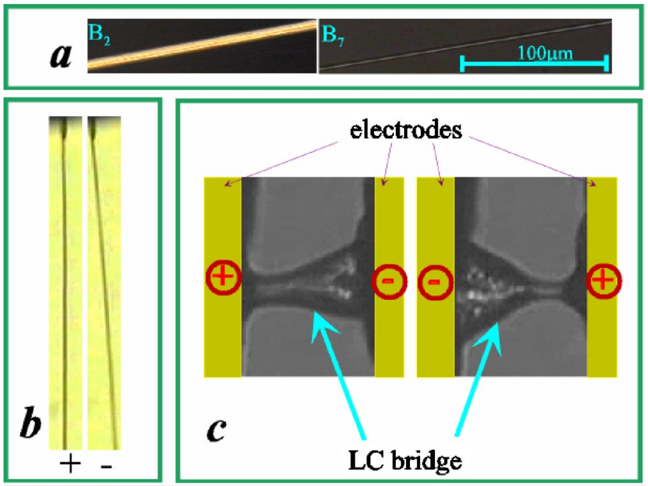


FIG. 4. Freely suspended fibers and bridges. (a) Fibers viewed between crossed polarizers. Left side: a bundle of fibers in the B_2 phase of material $B2$-1. The length of the entire fiber is 2 mm. Right side: a single fiber in the B_7 phase of material $B7$-2. The length of the whole fiber is 1 cm. (b) Photomicrograph of a fiber with 2 μm diameter of material $B7$-1 at one edge where positive and negative voltages (200 V) are applied. (c) Push-pull effect in the B_7 bridge of the compound $B7$-2. (a) $U=+100$ V, (b) $U=-100$ V. The pictures represent 125×125 μm areas.

We note that all of our attempts to pull fibers from other smectics of rod-shaped molecules (including those that form filaments in isotropic melts) were unsuccessful.

Single fibers of the B_7 materials show polar responses to external fields applied along the fibers. Under dc fields, some of them rupture, while others remain stable. For periodic fields, a transversal vibration could be observed in long single B_7 fibers. Interestingly, the vibration takes place with the frequency of the electric field. An example is shown in Fig. 4(b) for the material $B7$-1. The string is anchored at the end plates and become bent for one of the polarities of the voltage and straighten again under reversed field polarity. The maximum deviation from the straight direction is about 5°. The natural frequency of a $D=2$ μm diameter and $L=1$ mm length fiber was found to be about 10 Hz. From this we can estimate the elastic constant of the B_7 fiber. The first harmonic of the natural oscillation of an elastic spring with Young modulus E, diameter D, and length L fixed at the two ends can be expressed as [23]

$$f_{\mathrm{min}}=\frac{1}{2\pi}\frac{22.4}{L^2}\sqrt{\frac{E\theta}{\rho\Omega}}. \tag{1}$$

Here, $\Omega=(D/2)^2\pi$ is the area of the cross section and $\theta=\pi D^4/64$ is the inertial moment with respect to any axis in the plane of the cross section. With the observed $f_{min}\sim 10$ Hz, Eq. (1) gives $E\sim 3\times 10^4$ N/m^2. The contribution of the surface tension of the fiber-air interface to the Young modulus is $E_\gamma=4\gamma/D$. With $\gamma\sim 2\times 10^{-2}$ N/m, which is typical value for smectic liquid crystals, we get that $E\simeq E_\gamma$. This clearly shows that the single fibers are fluids along the fiber axis. Such a situation can be achieved by rolling the smectic layers into concentric cylinders (so-called "jelly roll" [21] structure). Observations of the fibers with a λ/4 wave plate inserted at 45° with respect to the crossed polarizers indicate that the refractive index along the fiber is lower than normal to it. In addition, we find that the birefringence increases toward the outer part of the fiber. Considering the principal refractive indices illustrated in Fig. 5(a), one can see in Fig. 5(b) that the effective birefringence is n_2-n_3 at the core, which is smaller than the birefringence (n_1-n_2) at the bark of the fiber.

In the bundles, electric fields induce a flow along the fiber axis. The direction of the flow depends on the sign of the electric fields, but in different fibers it can be opposite. In the B_2 bundles, the flow effects average out and the overall diameter remains unchanged. In the B_7 fibers, the flow direction appears to be the same in all strands and a net flow is observed. For relatively thick and short bundles, this results in a dramatic "push-pull" effect: depending on the polarity

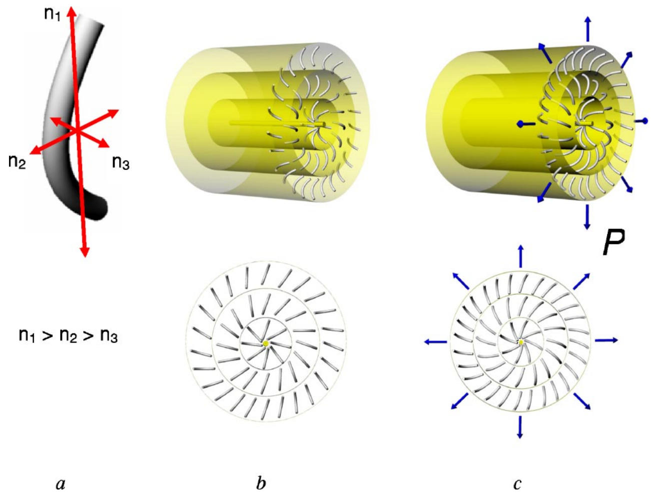


FIG. 5. Proposed structure of the banana-smectic fibers. (a) A banana-shaped molecule with the principal refractive indices. (b) Proposed layer and director structure (jelly-roll [21] configuration) of the B_2 phase. Upper row: three-dimensional 3D view. Lower row: cross section. (c) Proposed layer and director structure of the B_7 fibers. Upper row: 3D view. Lower row: cross section. Note that the difference between the director structures in the B_2 and B_7 phases is that in the B_2 the long axes of the average molecules are in the plane of the cross section, whereas in the B_7 phase they are tilted away. [18].

of the electric field, the material can be pulled or pushed from one side to the other. An example is shown in Fig. 4(c) for the material $B7\text{-}2$. The push-pull effect is not present in the B_2 bundles and also disappeared when the materials $B7\text{-}2$ and $B7\text{-}3$ were cooled to their B_2 phase.

Since the compound $B7\text{-}2$ is antiferroelectric along the smectic layers (and because the threshold for switching is much larger than the applied 1 V/μm field) [18], the push-pull effect cannot be attributed to a permanent electric polarization along the smectic layers. We propose therefore that it is due to the out-of-layer polarization component, in accordance with recent electro-optic and textural observations [18,33]. The effect of the polarization component normal to the smectic layers is illustrated in Fig. 4(c). The divergence of the polarization is identical to a local polarization charge density ($\vec{\nabla}\cdot\vec{P}=-\varrho_P$), which means that the individual volume elements appear to be charged. An electric field applied along the fiber axis (which is not sufficient to realign the polarization) induces a force density $\varrho_P\cdot\vec{E}$, which would pull the material along the field. Since the fibers are fluids along the fiber axis, this force will cause viscous flows with speed proportional to the field, just as observed in the experiments.

IV. DISCUSSION

Due to the pioneering nature of this research, there area number of unsolved problems and questions, which we have to address even though the present answers are only guidelines for further studies.

1. Why do banana smectics form fibers, but calamitic smectics do not?

Our premise is that the fiber forming ability of the smectics is related to the asymmetry of the surface tension, just as in the case of filament formation in an isotropic melt. Smectic filament formation in isotropic phase of both rod- [24], and bent-shaped [16] molecules was related to surface tension anisotropy $\gamma_a=\gamma_\parallel-\gamma_\perp<0$, where $\gamma_\parallel(\gamma_\perp)$ are the surface tensions measured along (perpendicular to) the smectic layers. The failure to pull fibers in air from smectics of rod-shaped molecules (including those that form filaments in isotropic melts) maybe attributed to strong layer fluctuations (Peierls-Landau instability) [25]. Such fluctuations weaken the intermolecular interactions and the layers can easily break up under pulling forces along the layers. In the case of banana smectics, the fluctuations of the layers are strongly suppressed due to the closed packing requirement that anchors the kinks of the neighbor molecules to align in planes. This is also seen in small-angle x-ray profiles that show higher-order Bragg peaks in banana smectics (which are never present in smectics of rod-shaped molecules). The strong steric and dipole-dipole intermolecular interactions therefore are not weakened by fluctuations, explaining why the banana-smectic fibers are much more stable.

2. What determines the diameter of the stable fibers?

Here, we utilize the analogy between the cross section of the banana-smectic and the columnar fibers, and we evoke the arguments used for columnar strands [8]. Following Rayleigh's treatment [26] for Newtonian fluids in gravity-free environment, we assume a small periodic fluctuation as $D=D_o+\varepsilon\cos(kz)$, which keeps the constant volume, and where D_o is the equilibrium diameter of the cylindrical fiber drawn in the z direction and k is the wave number of periodic distortion. Considering only isotropic surface tension γ and taking into account smectic layer compression modulus B, associated with a change in the layer spacing, the net potential energy per unit length can be written in the form of

$$V=(k^2\gamma D^2+8BD-4\gamma)\pi\varepsilon^2/4D. \tag{2}$$

The strand is stable against all fluctuations if $V>0$, and instability occurs for cylinder diameters smaller than

$$D_c=\gamma/2B, \tag{3}$$

if $k<k_c=2/D\sqrt{1-D/D_c}$. With values typical for smectic liquid crystals of rod-shaped molecules ($\gamma=2\times10^{-2}$ J/m^2 and $B\sim10^5$ J/m^3) we get $D_c\sim0.15$ μm. Similar to columnar fibers [8], this is an order of magnitude smaller than the experimentally observed smallest diameters. The discrepancy may indicate that the surface tension γ is larger and/or the layer compression modulus B is smaller than in calamitic smectics. Based on the observed frequency of the natural vibrations, we believe that the estimated γ is right, and we assume that the effective layer compression modulus B of banana smectics is an order of magnitude smaller than in calamitic smectics. At this point, it is important to note that in the case of tilted smectic materials, such as the B_2 and B_7

phases, the layer thickness variation does not need to involve permeation, but only the change in the director tilt angle. It is expected [27] that the layer compression modulus B_t, associated with the director tilt, is related to the permeative compression modulus B_p as $B_t/B_p \sim (l/w)^2$, where l (w) is the molecular length (width). Considering a typical banana-shaped molecule, we see that B_t is at least an order of magnitude smaller than B_p. With $B_p \sim K_1/a^2$ (where $K_1 \sim 10^{-12}$–$10^{-11} N$ is the splay elastic constant and $a \sim 4$ nm is the layer spacing) [3], we get that $B_t \sim 6\times10^3 - 6\times10^4$ J/m^3. Accordingly, the relatively large layer spacing and the director tilt may result in such a small layer compression modulus that is consistent with the measured smallest fiber diameters. Additional softening of the layer compression modulus can be due to the presence of defects, and/or intercalation effects (i.e., the overlapping of the long flexible alkyl chains at the ends of the molecules can be relatively easily varied). Independent measurements of the layer compression modulus are needed to clarify these assumptions.

3. Why do the B_7 banana materials form more stable fibers than the B_2 substances?

The higher stability of the B_7 bananas could also be connected to the out-of-layer polarization component that was already considered in explaining the push-pull effect. In the case of uniform in-plane polarization fields, the periodic fiber diameter deformation results in the formation of charged rings around the fibers. The charges of the same sign around the cylindrical cross sections repel each other. These extensional forces are larger in areas with narrower diameters, thus suppressing the thickness fluctuations. Actually, electrostatic stabilization [28] of fibers and electrospinning [29] that result in extremely narrow fibers are known among polymers.

4. What could be the practical use of banana-liquid-crystal fibers?

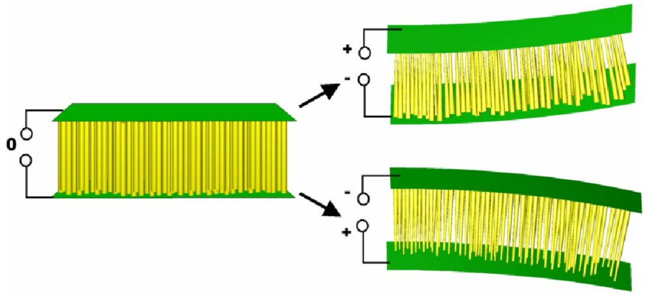


FIG. 6. Possible organic electromechanical device based on the push-pull effect observed in certain banana smectics.

Although the research of banana liquid crystals is relatively new, and it was mainly initiated by pure scientific curiosity, it has been already shown that they can be useful in scattering switching display and storage devices [30]. We believe that the unique electromechanical properties presented here can be useful for making organic electromechanical devices. Imagine, for example, that we disperse B_7 banana-smectic materials in a porous polymer matrix bounded between flexible electrodes. Based on the push-pull effect shown in Fig. 4(c), the B_7 material would be attracted or repelled by one of the electrodes depending on the sign of the electric field. Due to the elastic response of the polymer matrix, this would lead to a flexing of the film as illustrated in Fig. 6. Considering that the typical polarization of banana smectics is about 5×10^{-2} C/m^2, and the applied field is $E \sim 2\times10^7$ V/m, the field-induced stress is of the order 100 kPa, which is comparable to the peak stress of the biological muscles [31]. The research of banana liquid crystals can therefore help us to replicate what is happening in nature to make shape change.

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