

Highlight Review

Colloid and Interface Chemistry under Magnetic Fields

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Abstract

Magneto-Science, a new science of materials using magnetic fields, is recently being developed all over the world. Magneto-Science concerns mainly feeble magnetic materials such as soft matter, carbons, ceramics, and organometals. Colloidal and interfacial systems under magnetic fields are very attractive and important because the dimensions are very much sensitive to magnetic fields. In this review we introduce several examples in colloid and interface chemistry under magnetic fields. Molecular assemblies such as micelles, bimolecular membranes, and vesicles are changed in shape and, in some cases, size and composition by magnetic fields. Since mesophases are sensitive to relatively low magnetic fields, magnetic fields control not only structures and functions but also cooperatively other magnetically insensitive materials. Moreover, a few interesting magnetic responses in hydrogels are summarized.

◆ **Introduction**

Magnetism is a universal property of materials, which arises from angular momentum of an electron and nucleus. Diamagnetism and paramagnetism result in orbital motion and spinning of electrons. Therefore, any material will interact with magnetic fields. If the structures and properties of materials are controlled by magnetic fields, there may be available a general method for materials processing.

Magnetic fields affect chemical reactions,^{1,2} chemical equilibria,³⁻⁵ structures and physical properties of materials⁶⁻¹¹ via both direct effects such as quantum mechanical, thermodynamic, and mechanical effects and indirect effects such as convection, concentration changes, and temperature changes. However, since the magnetic energy of diamagnetic and paramagnetic materials is much smaller than the thermal energy at moderate temperature, it was believed to be insufficient to overcome the activation energy associated with chemical and physical processes. The magnetic energy (12.5 mJ mol^{-1}) of an electron spin in a field of 1 T corresponds to the thermal energy of 0.67 K or electric energy of $58 \mu\text{V}$ and is only about 10^{-5} of the thermal energy of 2.5 kJ mol^{-1} at 300 K.¹² Therefore, thermal energy disturbs magnetic field effects in feeble magnetic systems. Consequently, it does not seem that magnetic field effects appear even at moderate temperatures at which materials are processed. However, a variety of magnetic field effects were found in fact using high magnetic fields and sophisticated techniques in appropriate systems.

The Japanese invention of a liquid-helium-free 10 T superconducting magnet in around 1990 was a key technology for

facilitating the new science of feeble magnetic materials under magnetic fields, because chemists can use these easily in academic and industrial laboratories. Magneto-Science became established in 2002 when a book “Jiki Kagaku” (in Japanese) was published,¹³ and subsequently an English version appeared in 2006.¹² Many interesting and important magnetic field effects on feeble magnetic materials were found, but a limited scope of subjects is reviewed here, namely, colloid and interface chemistry under magnetic fields.

◆ **Colloid and Interface Chemistry in Magneto-Science**

The interaction of magnetization $M (= \chi H)$ induced on a material with the applied external magnetic field H gives rise to a magnetic energy E . A rod-like and plate-like molecule and domain having an axial symmetry have the magnetic susceptibilities $\chi_{//}$ and χ_{\perp} , which are in the directions parallel and normal to the axis, respectively. E of a domain (volume $V = Nv$) containing N molecules, whose long molecular axis is at an angle φ to H or the magnetic flux density $B (= \mu_0 H)$, where μ_0 is the magnetic permeability of vacuum), is given by the following equation:^{8,12}

$$E = -(B^2/2\mu_0)(\chi_{\perp} + \Delta\chi \cos^2 \varphi)V \quad (1)$$

where $\Delta\chi (= \chi_{//} - \chi_{\perp})$ is the diamagnetic anisotropy. The second term is the magnetic orientational energy (E_r),

$$E_r = -(B^2/2\mu_0)V\Delta\chi \cos^2 \varphi \quad (2)$$

which causes parallel or perpendicular alignment of the domain axis to the direction of H , depending on positive and negative signs in $\Delta\chi$, respectively.^{14,15} Since the alignment occurs only when E_r overcomes the thermal energy, $|E_r| > kT$, where k is the Boltzmann constant; i.e., $V > 2\mu_0 kT \cos^2 \varphi / |\Delta\chi| B^2$. Since $1 \geq \cos^2 \varphi$, a sufficient volume V_m of a domain for the magnetic orientation is $2\mu_0 kT / |\Delta\chi| B^2$. The minimum size ($V_m^{1/3}$) of a material having $\Delta\chi = 10^{-7} - 10^{-5}$ is estimated to be in the range 101–470 nm at 1 T and 21.8–101 nm at 10 T and 300 K.¹⁴ These sizes, including pore size and crystallite size, are covered by colloid chemistry and agree with target materials and scales in Magneto-Science. The time for the orientation in usual solvents and polymer melts ($1 - 10^3 \text{ Pas}$) is estimated to be between subsecond and several hours.¹⁴

Colloidal materials are subject to the Lorentz force^{16,17} and magnetic force¹⁸⁻²² through interactions of magnetic fields with electric current and magnetic gradient, respectively. When gravity is balanced with a magnetic force, magnetic levitation, and pseudo-microgravity may be realized,^{23,24} which for

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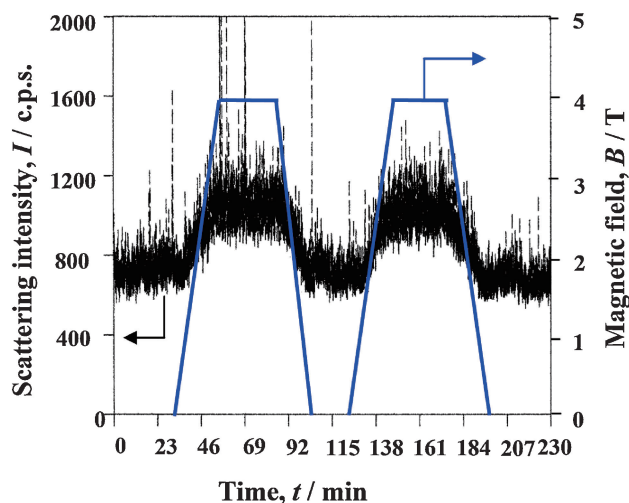


Figure 1. Magnetic gradient effects (4 T; $-141 \text{ T}^2 \text{ m}^{-1}$) on light scattering intensity from a (1 + 2) cyclohexane/aniline solution containing $[\text{Mn}(\text{acac})_2]$ at 301.7 K.

example should lead to enhanced fluctuation of domain size or correlation length. Figure 1 shows that the scattered light intensity of a (1 + 2) cyclohexane/aniline mixture containing $[\text{Mn}(\text{acac})_2]$ was increased by application of a magnetic force at 301.7 K, just above the phase-separation temperature. Since a cyclohexane domain is lighter than an aniline domain, the gravity gives buoyancy. The magnetic force field of $-141 \text{ T}^2 \text{ m}^{-1}$ may depress the buoyancy acting on a cyclohexane domain because a paramagnetic $[\text{Mn}(\text{acac})_2]$ -rich aniline phase should be buoyed by the magnetic force. Therefore, the magnetic force reduced the gravity force to lead to a less gravity field and enhance the equilibrium concentration fluctuation. The correlation length estimated from the Ornstein–Zernike equation increases from $32 \pm 10 \text{ nm}$ at zero field to $54 \pm 10 \text{ nm}$ under the less gravity field due to a magnetic force.

◆ Lipid Membranes under Magnetic Fields

Magnetodeformation of Vesicles.

The structure of organized molecular assemblies of lipids can be controlled by a flow and an electric field. A magnetic field is also a potential method for aligning molecules and domains. It is well established that anisotropic diamagnetic assemblies will become oriented in a steady magnetic field so as to achieve the minimum-energy state. The long molecular axis of lipid molecules in a domain can be cooperatively aligned in the direction of averaged φ ,¹⁵ when H is as large as E_f that overcomes the thermal energy. For example, orientational effects are expected when N is on the order of 10^5 for $\Delta\chi \approx -1 \times 10^{-6} \text{ cm}^3 \text{ mol}^{-1}$ (for long chain hydrocarbons) under 10 T and 318 K or when $H = 4.0 \text{ T}$ for a 200-nm-diameter vesicle with a 5 nm membrane thickness.²⁵ Gaffney and McConnell⁶ and Maret and Dransfeld,⁷ using the electron spin resonance (ESR) and the Cotton–Mouton effect, respectively, showed that phospholipid molecules could be oriented by a magnetic field.

A bilayer membrane is not a homogeneously curved surface in the case that it has different elastic properties because of, e.g.,

the orientational defects. The Helfrich theory for the magneto-deformation, s_2/r_0 , of a spherical bilayer vesicle of radius r_0 , in which the domains in the bilayer have a local curvature c_0 , gives the equation

$$s_2/r_0 = r_0^2 \Delta\chi b H^2 / 3k_c(6 - c_0 r_0) \quad (3)$$

where b is a measure for the thickness of the bilayer, and k_c is the curvature-elastic modulus.²⁶ Helfrich predicted theoretically the magnetic deformation of vesicles of a sphere to an ellipsoid. Tenforde and Liburdy also showed experimentally the magnetic undulation of membrane.²⁵ Moreover, we found using light scattering and small-angle neutron scattering that the magneto-undulation of membranes brought about the magnetodeformation of dipalmitoylphosphatidylcholine (DPPC) and dioctadecyldimethylammonium chloride (DODAC) vesicles into prolate ellipsoids because of $\Delta\chi < 0$.²⁷ Christianen and co-workers persist that the Helfrich theory is not enough to explain their results on magnetodeformation of self-assembled sexithiophene nanocapsules, which associate into oblate ellipsoids because of connected aromatic rings in a sexithiophene molecule.²⁸ They proposed an anharmonic magnetic deformation model to understand the enhanced rigidity of the membrane due to high magnetic fields.²⁹

Magnetofusion and Magnetodivision of Vesicles.

Applying a high magnetic field to vesicles, the fusion and division may be induced via a deformed state. The total energy ($E^H(r_0)$) of a vesicle which has initially the radius r_0 comprises the curvature-elastic and magnetic energies.²⁶ Roughly, the vesicle growth of radius r_0 to r with the association of n vesicles under a magnetic field may be possible when $E^H(r_0) \geq (1/n)E^H(r)$, assuming $r = n^{1/2}r_0$. When H is large enough or the shape of a vesicle is exactly an ellipsoid of revolution, the following relation is obtained if c_0 is unchanged during the size change:²⁷

$$6(1 - n) - c_0 r_0 (n^{1/2} - n) \geq 0 \quad (4)$$

Solving eq 4 and adding the condition $\Delta\chi(6 - c_0 r_0) > 0$, the possible association in a vesicle having radius r_0 may be estimated for a given radius of curvature ρ ($= 1/c_0$), as shown in Figure 2. The shadowing in the figure illustrates the regions of magnetofusion ($n > 1$) and magnetodivision ($n < 1$) for $\rho = 10$ or 20 nm. When $n = 1$, a vesicle of size r_0 is stable against a certain magnetic field.

The loose condition eq 4 predicts both the magnetodivision and magnetofusion for vesicles of a certain r_0 . Experimentally, small DODAC vesicles less than 500 nm tend to grow with the magnetofusion, but the larger vesicles tend to decrease in size by the magnetodivision. Figure 3 shows that the vesicles having $r_0 \approx 150$ and 1150 nm became larger and smaller, respectively, by application of magnetic fields, depending on kind of aromatic additives. The dynamic process seems to be rapid because a 10-ms-pulsed magnetic field of 20 T promoted significant magnetodivision, although no 1-ms-pulsed field affected the size distribution.

The magnetodeformation as well as the magnetodivision and magnetofusion led to the release of small fluorescent lucigenin molecules from DODAC vesicles, as shown in Figure 4. This demonstrates a possibility of the magnetic field-induced drug release.

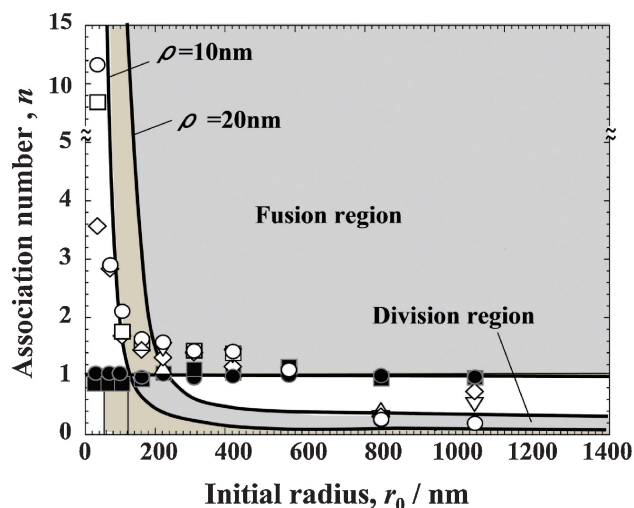


Figure 2. Comparison between experimental (symbols) and theoretical association number (n). Possible association number in vesicles having an initial radius r_0 and a given radius of curvature, $\rho = 10$ and 20 nm under a 10-T-magnetic field. Shading illustrates the regions of magnetofusion ($n > 1$) and magnetodivision ($n < 1$).

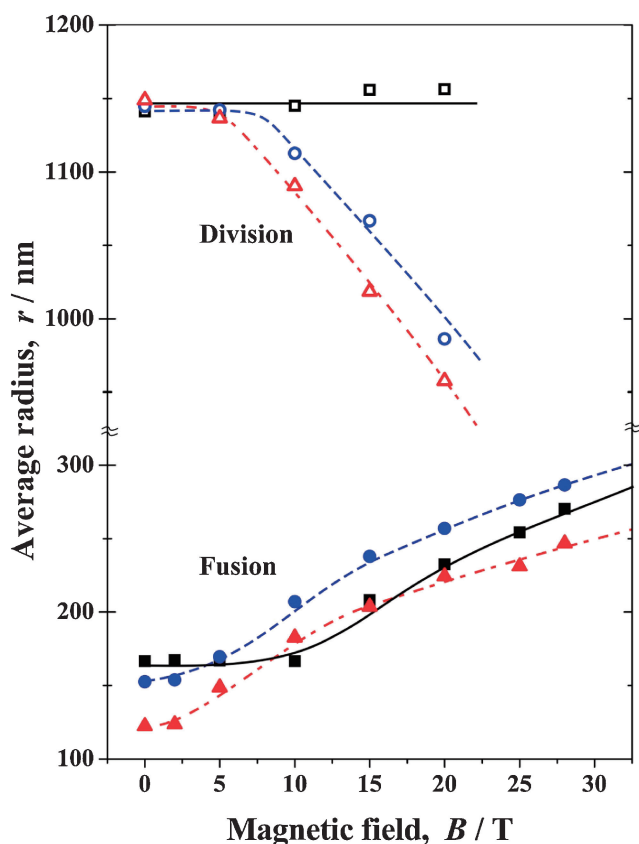


Figure 3. Magnetic field dependence of averaged size of DODAC vesicles (square) of $r_0 = \text{ca. } 150$ and 1150 nm containing naphthalene (circle) and anthracene (triangle) at 318 K.

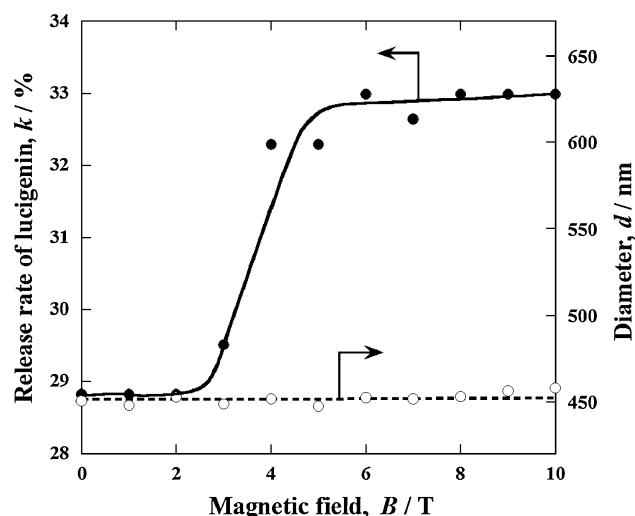


Figure 4. Magnetic field dependence of molecular release from DODAC vesicles including a fluorescent dye, lucigenin, at 318 K. Open circle, average diameter of vesicles; solid circle, release rate.

Magnetic Regulation of Black Lipid Membranes.

There are many reports on magnetic field effects on biological systems; the structural changes of organisms,^{30,31} catalytic activities,^{9,10} material transport,¹¹ etc. Since biological cells contain many components and elementary processes, net magnetic response may be undetectable. To elucidate the magnetic effects on, e.g., biomembranes, an artificial bimolecular or black lipid membrane (BLM) should be used as a simple model membrane.

The membrane potential (Ψ) and resistance of BLMs, comprising didodecyl phosphite, DPPC, and DODAC, changed remarkably at low magnetic fields less than 0.5 T.^{32,33} The electrical properties seem to be affected not via the Lorentz force on the ion flux but via a cooperative orientation of lipid molecules. Figure 5 shows a typical example of changes in Ψ of a BLM with the application of magnetic fields (H_{\perp}) perpendicular to the membrane. The general features in the magnetic response are its reversibility: the maximum response at around 0.2 T and the reverse response at $H > 0.4$ T. Ψ changed by ca. 2 mV even at 0.025 T, whose response seems to be notably large compared to the magnetic field (roughly 24 T) theoretically required to change a nerve impulse.³⁴

The membrane potential is generated by the apparent fixed charge density (σ) of the membrane.³⁵ When a magnetic field was perpendicular to the direction of ion flow, no magnetic effect was observed at 0.5 T, suggesting that an ion flow would not be directly affected by the Lorentz force. Thus, the change in σ due to magnetic fields may be brought about by the molecular orientation in a direction perpendicular to H ($\varphi = \pi/2$). When a lipid molecule in the membrane of $N > 10^7$ for $\Delta\chi \approx -1 \times 10^{-6}$ tilts under a magnetic field, the occupied molecular area increases monotonically with φ , and thus σ would also decrease monotonically with magnetic fields. With further increasing φ , the hydrocarbon/water interface at the membrane surface should increase and destabilize the tilted structure. Thus, the critical tilt angle must exist at the critical magnetic field. A higher magnetic

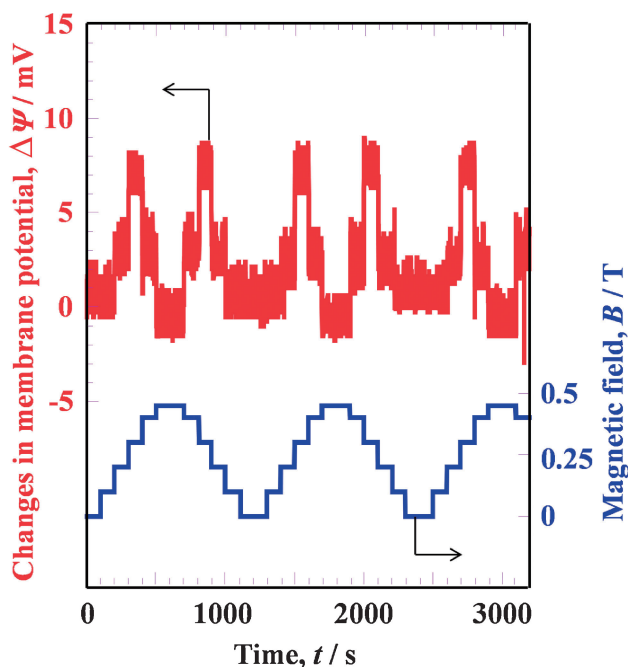


Figure 5. The time course of changes in membrane potential Ψ of a black lipid membrane of DODAC containing 20 mol % perylene with the application of various magnetic fields perpendicular to the membrane at 318 K.

field would introduce membrane undulation such as a ripple phase.²⁷ The undulation structure must be frozen or stable under a 11.7-T magnetic field during cooling. Such irreversible deformation has been observed by means of ³¹P and ²H NMR.⁸

Lipid monolayer and multilayer on metal electrodes also are affected by magnetic fields.^{36–39} Yonemura et al. demonstrated the magnetic-field-induced photocurrent of photo-semiconductors via sodium dioctyl sulfosuccinate membrane on an electrode.^{36,37} Saravanan and Ozeki⁴⁰ showed the magnetic field effect on tunneling current through a self-assembled monolayer (SAM) on a gold electrode. The electron-transfer path changed with tilt angle of SAM molecules to switch from bond-through to space-through tunneling path by applying a 6-T magnetic field perpendicular to the membrane (Figure 6).

◆ Magnetic Field Effects on Amphiphilic Assemblies

Amphiphilic molecules will form micelles with various shapes and sizes. Since rod-like micelles have the persistence length of about 20 nm or less, long rod-like micelles are flexible and behave as worm-like micelles. Adding salicylate ions to a rod-like micellar solution of hexadecyltrimethylammonium bromide (CTAB), very long rod-like micelle or thread-like micelles are formed to be entangled. Magnetic fields lengthened the persistence length to extend the worm-like micelles having R_G more than 120 nm. Also, the entangled micellar coil was somewhat extended, e.g., from 145 to 165 nm in a direction of a 10 T magnetic field, to form an ellipsoid.

Amphiphiles in the higher concentration region also form various mesophases such as hexagonal and lamellar structures or middle and smectic liquid crystals. It is well known that liquid

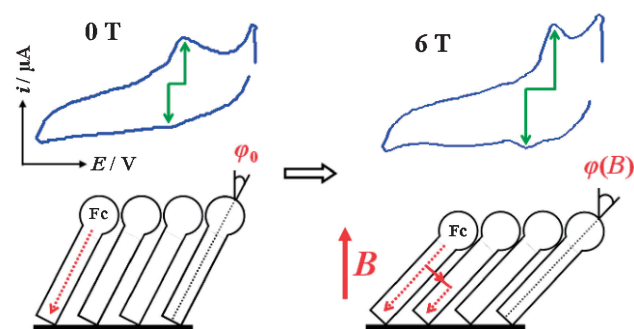


Figure 6. Magnetic field effects on cyclic voltammograms of the self-assembled monolayer of (ferrocenylmethyl)dodecyltrimethylammonium bromide on a gold electrode and schematic model for magnetic field effect on the electron tunneling through the SAM membrane.⁴⁰ A 6-T magnetic field B is applied perpendicular to the membrane.

crystals composed of small molecules and oligomers are affected by steady magnetic fields.^{41–43} Therefore, it is plausible that CTAB/silicate hybrids having hexagonal structure were ordered under steady magnetic fields in order to prepare highly ordered honeycomb mesoporous silica (MCM-41).^{44–46} Helical silicate hybrids, prepared using sodium dodecyl sulfate and trimethoxysilylpropyltrimethylammonium bromide as a template instead of CTAB, was aligned, elongated, thinned, and changed their pitch under magnetic fields more than 6 T (Figure 7). The helicity of the hybrids was not clearly regulated even by high magnetic fields, although magnetic control of chirality should be a dream in chemistry.⁴⁷ Lorentz force under magnetic fields or magnetohydrodynamics (MHD) enabled control of the helicity in silicate tubes^{17,48} and the chirality of metal dendrite and organic film on an electrode.^{49,50}

The magnetic alignment of surfactant molecules as a template was insufficient for the perfect orientation of hexagonal layer on a quartz plate.⁴⁶ Expecting the larger tendency of magnetic orientation of a template molecule, a triblock copolymer Pluronic P123, high magnetic fields were applied to a SBA-15 hybrid membrane using P123 as a template on a glass plate. An SBA-15 film with hexagonal pores perpendicular to a glass plate was obtained under a 30-T magnetic field.⁵¹ By the addition of aromatic additives having large magnetic anisotropy, magnetic orientation of organosilicates in preparing SBA-15 hybrids was cooperatively promoted to lead to perpendicular and uniaxially parallel alignment of microchannels in an SBA membrane (Figure 8). The threshold magnetic field intensity for perfect perpendicular orientation indicating no XRD peaks was reduced with increasing the number of benzene rings in the additives homologues, e.g., the threshold magnetic field decreased from 30 T for no additive to 12 T for pentacene.

The structure of porous organometallic complexes such as $[\text{Cu}_2(\text{pzdc})_2(\text{pyz})] \cdot 2\text{H}_2\text{O}$ (CPL-1)⁵² can be changed by applying steady magnetic fields to reactant solutions during synthesis. Aqueous pyrazine-2,3-dicarboxylic acid (H_2pzdc) solutions was added to an aqueous $\text{Cu}(\text{ClO}_4)_2$ solution containing pyrazine (pyz) under magnetic fields at 298 K and stood under the magnetic field for 1 h. Blue precipitates CPL-1 prepared under zero field were spherical aggregates comprising plate-like microcrystals, but those prepared under magnetic fields con-

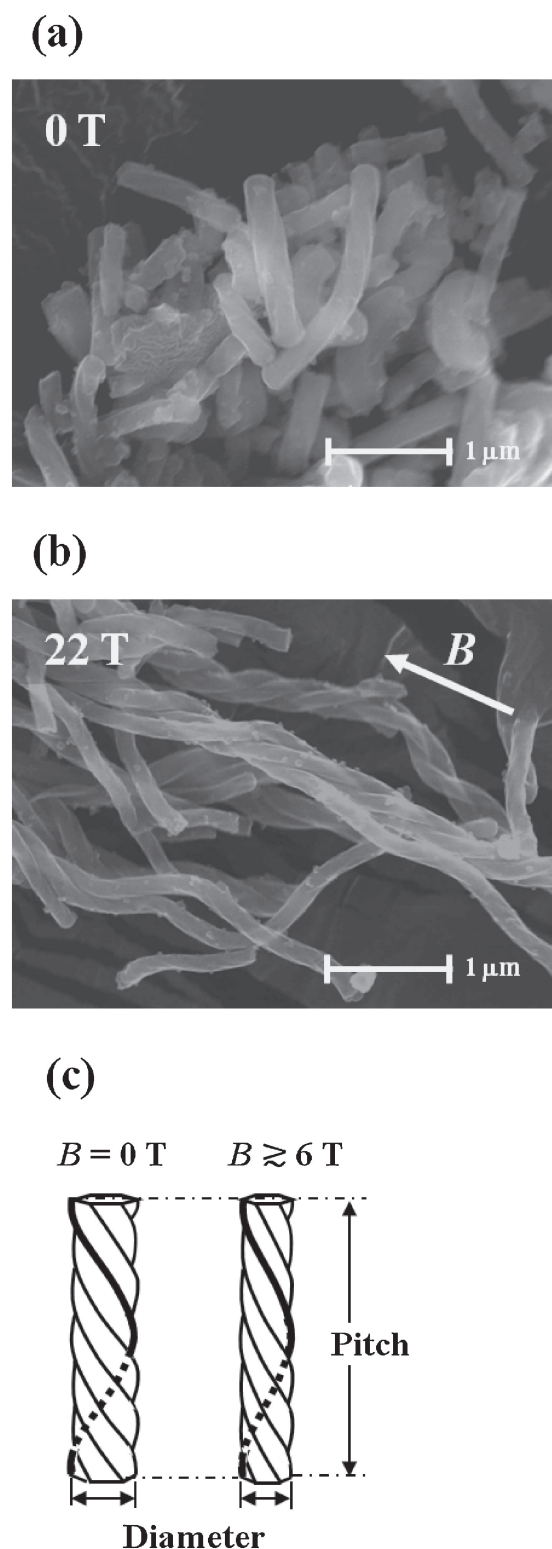


Figure 7. SEM images of helical silica hybrids prepared under zero field (a) and 22 T (b) at 298 K. An example of change of helical structure is illustrated (c).

tained needle-like microcrystals. The needle-like crystals prepared at 6 T were separated and used as a seed crystal in the same reactant solution under magnetic fields. The grown single-crystal

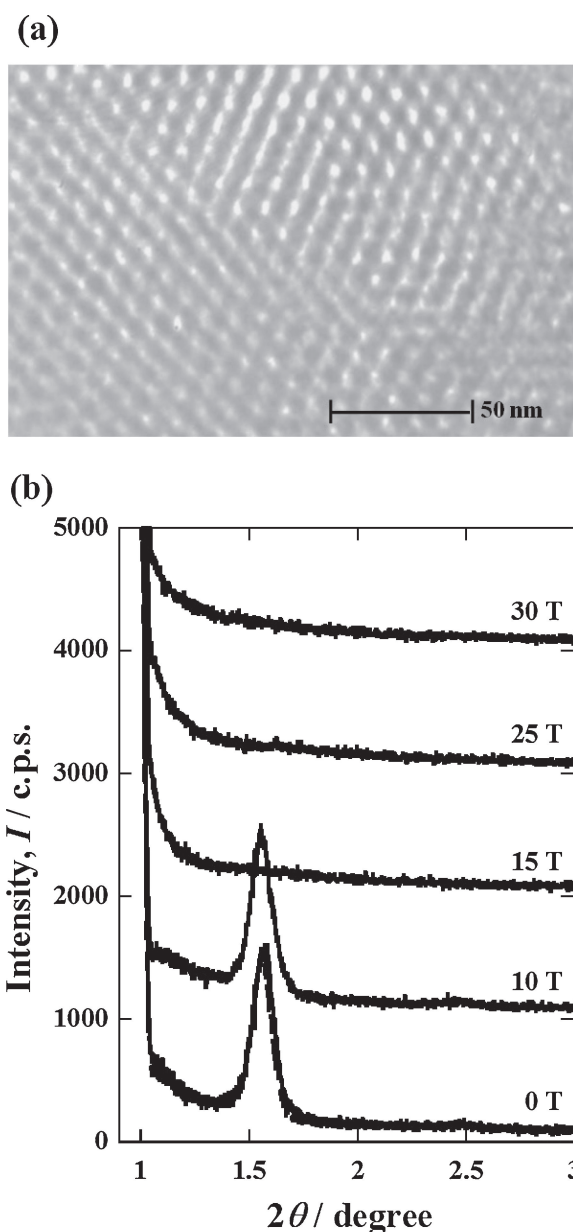


Figure 8. A TEM image (a) and XRD patterns (b) of SBA-15 membrane prepared at various magnetic fields perpendicular to the membrane surfaces.

($[\text{Cu}(\text{pzdc})(\text{pyz})] \cdot \text{ClO}_4$) (six-coordinated Cu^{2+}) under a 6-T magnetic field was nonporous and completely different from the structure of microporous CPL-1 (five-coordinated Cu^{2+}), as seen in Figure 9. This may be the first example of the composition and structure of a crystal being controlled by magnetic fields.

◆ **Polymers and Macromolecular Hydrogels under Steady Magnetic Fields**

Since organic and inorganic polymers have high molecular weight and magnetic anisotropy, magnetic orientation may occur easily under relatively low magnetic fields. Thus, there are many

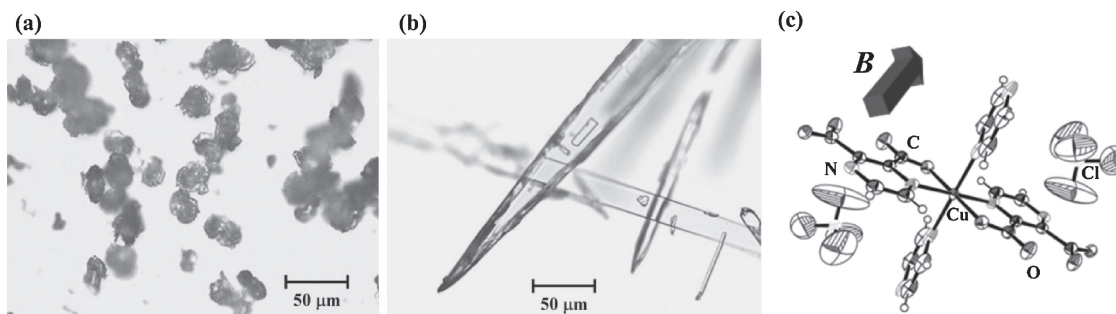


Figure 9. Magnetic-field-induced structural changes in CPL-1 (a). Preparation conditions: (a), zero field; (b), 6 T. The crystal structure of (b) is given in (c).

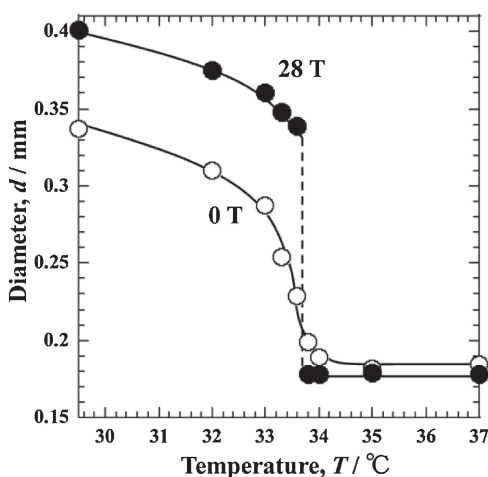


Figure 10. Magnetic-field-induced volume phase transition. NIPA gels prepared at zero field (open circle) and 28 T (solid circle) show continuous and abrupt volume changes, respectively.

reports^{12,13} on magnetic alignment of fibers such as carbon nanotube and Bucky paper^{53,54} and crystalline polymers such as polypropylene in which mesophase formation are involved.^{14,55}

Hydrogels have been investigated from the view point of not only scientific interests but technological aspects such as drug delivery and actuators. Thus, it is very important to investigate methodology for control of the structures and physical properties of gels. Poly(*N*-isopropylacrylamide) (NIPA) gels were prepared from NIPA monomers in a 0.4-mm-diameter glass capillary at 278 K under steady magnetic fields. The volume of NIPA gels increased with magnetic field intensity, and the volume phase transition was much more significant, although no volume phase transition temperature T_c (307.0 K) changed. Magnetic fields thickened the gels in a swelling state, while on the other hand, thinned in a shrinking state.⁵⁶ When a NIPA gel was prepared at high crosslinkage ratio q^{pre} , the opaque gel showed no volume phase transition, as shown in Figure 10. However, when it was prepared under magnetic fields, the gel became transparent and its diameter changed discontinuously at T_c , i.e., magnetic fields in preparation induced the volume phase transition. The discontinuity in the volume phase transition became more significant with increasing magnetic field intensity in preparing gels. The plots of both diameters of swelling and shrinking gels at T_c against magnetic

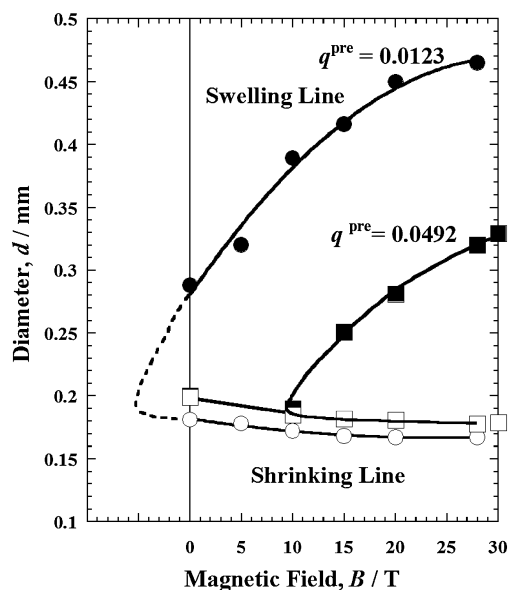


Figure 11. Diagram for the magnetic-field-induced volume phase transition. The diameters of swelling and shrinking NIPA gels at the volume phase transition temperature were plotted as a function of magnetic field.

field intensity gave the swelling line and the shrinking line, as shown in Figure 11. A similar plot was obtained for polyacrylamide (AAM) gels prepared under magnetic fields, although AAM gels are well known as a nonvolume phase-transition gel. These phenomena may arise from orientation of polymer chains, reduction of the effective crosslinkage q^{eff} , e.g., $q^{\text{eff}} < 1/3q^{\text{pre}}$ over 15 T, and hydration promotion due to magnetic fields.

Physical gels also may be affected by magnetic fields. An aqueous solution of a polysaccharide, agarose, shows the sol–gel phase transition. The melting temperature of agarose gel prepared over 3 T was 1.0 K higher than that prepared at zero field.⁵⁷ The phenomenon was ascribed to the self-oriented domains which can rotate by a magnetic field to grow up into larger domains. The chain alignment in a gel was detected by the birefringence which depended on the directions of magnetic field. The anisotropic, aligned gel was applied to electrophoresis for DNA separation.⁵⁸

When external magnetic fields were applied to NIPA gels, the diameter of gels in the swelling state increased linearly with increase in magnetic field less than 10 T to approach gradually a

saturated volume and then reached 40% increase in diameter at 30 T. The notable phenomenon due to magnetic fields is the lower temperature shift of the volume phase transition of NIPA gels in an aqueous phenol solution. This led to the magnetic-field-induced volume phase transition, i.e., abrupt change of a swelling volume to shrinking volume by applying magnetic fields.

◆ Conclusion

The main subjects of Magneto-Science were reviewed, which concern colloid and interface chemistry under magnetic fields. Colloidal dimension will interact with magnetic fields to change detectably ordering, structures, size, and functions of feeble magnetic materials. Potentialities of various magnetic fields such as homogeneous, gradient, and pulsed were demonstrated by many examples of regulation of feeble magnetic materials. These suggest extensive applications for sensors, chirality control, structure-controlled processing, drug release from vesicles and capsules, colloid transport, micropatterning, materials trap, separation, etc. Great possibilities will be open to materials science under sophisticated, designed magnetic fields in space and time. Magnetic energy is dream energy: clean, mild, energy saving, and highly penetrating. We believe that new Magneto-Science will develop leading to new technology.

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