

## Experimental

**Fabrication of PPy Nanoparticles:** For the synthesis of the PPy nanoparticles with an average diameter of 2 nm, octyltrimethylammonium bromide (OTAB; 6.0 g) was magnetically stirred in 40 mL of distilled water at 3 °C. Pyrrole (1.0 g) was then added dropwise to the surfactant solution, and iron(III) chloride (5.561 g) dissolved in a small amount of distilled water was added to the reaction mixture. Chemical polymerization proceeded for 3 h at 3 °C. The reaction product was then transferred to a separating funnel and excess methanol was added to remove the surfactant and residual iron(III) chloride. A small amount of isooctane was added to promote the precipitation of the PPy nanoparticles. The upper solution containing surfactant and unreacted iron(III) chloride was discarded and the nanoparticle precipitate was dried in a vacuum oven at room temperature. PPy nanoparticles with a diameter of about 6 nm were prepared using decyltrimethylammonium bromide (DeTAB; 2.3 g), following the same procedure.

**Carbonization Process:** In a typical carbonization procedure, the PPy nanoparticles (about 2 g) collected from two runs of the microemulsion polymerization were precarbonized at 800 °C for 3 h under Ar gas flow (0.2 L min<sup>-1</sup>). Carbon felt was put at the inlet of Ar flow to reduce the oxidation of the polymer precursor. After the pretreatment at 800 °C, the weight of the carbonized product was reduced to 30–35 % of the initial loading weight. The “missing” components, formed as a result of carbonization reactions such as denitrogenation, dehydrogenation, and dehalogenation, are removed by the Ar flow. The products of two runs of the pretreatment were collected and transferred to a quartz tube 4 cm in diameter. The quartz tube was evacuated and refilled with argon repeatedly. The argon-filled quartz tube was then placed in an electrical furnace equipped with a larger diameter alumina tube (6 cm in diameter). The valve of the quartz tube was opened and the alumina tube containing the quartz tube was evacuated and refilled with argon repeatedly. The second carbonization proceeded with streaming argon at a heating rate of 3 °C min<sup>-1</sup>. After 6 h of carbonization, the residual soot was collected and toluene was added to the quartz tube containing the sublimed fullerene film. The quartz tube was sonicated with gentle heating. The crude soot was refluxed with toluene in a standard Soxhlet extractor at 100 °C for 12 h.

**Instrumental Analysis:** TEM images were taken with a JEOL 2010 F microscope and EDX analysis was performed using a Philips CM 20 microscope. MALDI-TOF mass spectra were obtained with a Voyager-DE STR Biospectrometry Workstation (Applied Biosystems) operating in positive mode at an accelerating voltage of 20 kV using diethanol as the matrix. HPLC analysis was performed with a HP1100 liquid chromatograph. A 250 mm × 4.6 mm i.d. monomeric octadecylsilica (ODS) column was used as the stationary phase. A mixture of toluene and methanol (55:45) was used as the mobile phase at a flow rate of 0.6 mL min<sup>-1</sup>. Detection was performed with a diode-array detector at a detection wavelength of 330 nm.

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## Scalable Synthesis of a New Class of Polymer Microrods by a Liquid–Liquid Dispersion Technique\*\*

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Micro- and nanostructured materials formed by assembly of colloidal particles could find a wide variety of applications, ranging from photonics and electronics to catalysis, bioprocessing, sensors, and energy storage.<sup>[1]</sup> The functionality of these materials largely depends on the size, shape, and physical properties of the particles from which they are assembled. The use of anisotropic particles is of significant interest, as it allows the development of materials with an advanced microstructure or anisotropic properties. Enormous progress has been made in the synthesis of rod-like particles on the nanoscale, the most notable structures being various nanowires,

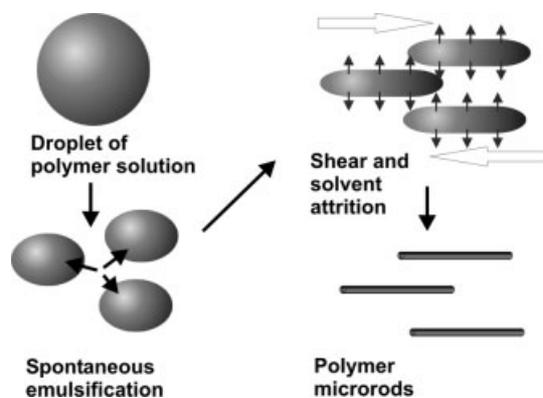
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nanotubes, and nanorods with unique optical, magnetic, and electrical properties.<sup>[2]</sup> Polymer nanocylinders can be formed by self-assembly of block copolymer molecules,<sup>[3]</sup> or by emulsion polymerization of tetrafluoroethylene in the presence of rod-like surfactant micelles.<sup>[4]</sup> Rod-like cylindrical particles on the micrometer scale could also form the basis of materials with unique properties, although very few processes for making such particles have ever been developed. For decades the entropic self-assembly of rod-like colloidal particles has been of intense interest,<sup>[5]</sup> but it has been studied mainly with viruses<sup>[6]</sup> and, in a few cases, with inorganic particles,<sup>[7]</sup> as suitable anisotropic particles were not readily available. Microcylinders can also serve as a medium for longitudinal ordering of smaller rod-like objects, such as carbon nanotubes,<sup>[8]</sup> for enzyme immobilization,<sup>[9]</sup> or for the preparation of composite nanostructures.<sup>[10]</sup> Polymer rods may exhibit new properties in comparison to the bulk material because of the specific alignment of the molecules inside them.<sup>[9,11]</sup>

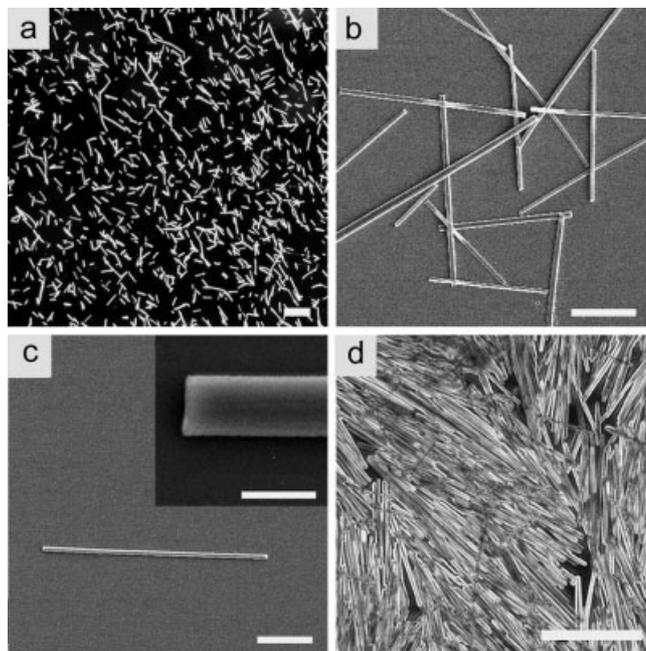
Polymer nanofibers, tubes, or “pencils” have been previously synthesized only by a template method<sup>[9,12]</sup> within cylindrical pores of membranes, zeolites, or mesoporous silicas. This method allows good control over the particle sizes, but only a very limited amount of rods can be synthesized with the template, as at some point it has to be destroyed to extract the products. Because of the lack of methods for facile fabrication of microrods, virtually every material assembled from micrometer-sized particles has been formed from spheres of silica or polymer (latex).<sup>[13]</sup> We report here an efficient scalable process for the formation of a new class of polymer microrods with large aspect ratio that can be as inexpensive and ubiquitous as spherical latex particles, but possess a variety of properties that could be valuable for self-assembly and materials applications.

The polymer microrod synthesis is based on a new liquid-liquid dispersion technique, presented schematically in Figure 1. The microrods were made fully or partially from cross-linked SU-8 (photocurable epoxy resin). The process begins by adding a small amount of concentrated solution of SU-8 in  $\gamma$ -butyrolactone (GBL) to an organic liquid medium (such as glycerol or its mixtures with alcohols or glycols) subjected to a



**Figure 1.** Schematic of the process for the synthesis of rod-like polymer particles.

viscous shearing by an impeller. The emulsification under shear results in a dispersion of rod-like particles with diameters of between 0.5 and 3  $\mu\text{m}$  and lengths of tens of micrometers (Fig. 2).



**Figure 2.** Typical optical microscopy (a,d) and SEM (b,c) images of SU-8 microrods taken with different magnification. The inset in (c) shows the tip of the rod at higher magnification. The scale bars are: a) 100  $\mu\text{m}$ , b,c) 10  $\mu\text{m}$  (1  $\mu\text{m}$  in the inset of (c)), and d) 50  $\mu\text{m}$ . The rods were prepared in a 50:50 vol.-% mixture of glycerol and ethylene glycol. After UV crosslinking, the particles were transferred to water (a–c) or dodecane (d). The samples were imaged after drying of suspension drops on glass slides.

The formation of rods in the mixing vessel appears to be a result of three concurrent processes: emulsification of the polymer solution in the organic medium, shear-driven deformation of the emulsion drops into cylinders, and their successive solidification into rod-like particles. All of these processes are assisted by the fact that the solvent for the SU-8 (GBL) is soluble in the dispersion media (while SU-8 itself is not). The GBL diffuses out of the dispersed sheared droplets, leaving the solid polymer behind. The solidification of the elongated drops does not allow them to restore their spherical shape, and they can be kept for a long period (hours and days), during which time they could be turned into an extremely stable polymer by UV light crosslinking.

The emulsification of the polymer solution is facilitated by the high shear-rates during stirring. The process of droplet emulsification, deformation, and breakup is characterized mainly by two dimensionless parameters: the viscosity ratio,  $p = \mu_1/\mu_0$  (where  $\mu_1$  and  $\mu_0$  are the viscosities of the drops and the suspending medium, respectively), and the capillary number, defined as the ratio of the shear stress and the Laplace pressure,  $Ca = \mu_0 Ga/\sigma$  (where  $G$  is the shear rate,  $a$  is the non-deformed drop radius, and  $\sigma$  is the interfacial tension).<sup>[14]</sup> In

our case, the interfacial tension is low (due to the miscibility of the SU-8 solvent, GBL, and glycerol), corresponding to high values of  $Ca$  ( $Ca \geq 1$ ) where the polymer solution droplets are deformed into long, thin cylinders that may further break into droplets with roughly equal volumes.<sup>[14]</sup> The breakup of the elongated droplets is also probably assisted by the spontaneous emulsification of the oil caused by mass transfer of GBL molecules from the oil phase into the medium. The long, thin liquid cylinders later solidify due to the attrition of the polymer solvent. The resulting rods have uniform diameter, and, surprisingly, very straight perpendicular edges (Fig. 2c). These straight edges suggest that the mechanism may involve the initial formation of even longer cylinders that are broken into shorter pieces by the shear flow after the droplets have solidified.

The synthesis of SU-8 microrods is well reproducible and in most of the experiments more than 97% of the emulsion drops are sheared into particles with a regular cylindrical shape, as shown in Figures 2b–d. The rest of the obtained particles are mostly rod-like but possess some defects. Less than 1% of the oil drops produce irregularly shaped particles, probably resulting from coalescence of the drops during the dispersion stage, or irregular shearing conditions in some parts of the vessel.

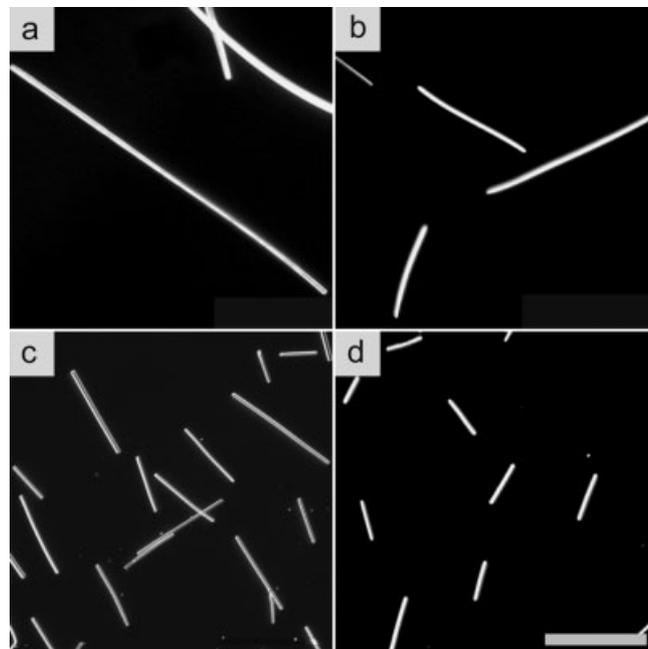
The procedure is flexible and allows control over microrod sizes, aspect ratios, and polydispersity in a variety of ways, a number of which were tested; the results are summarized in Table 1. The first important operational parameter is the shear stress, which can be varied either by the rate of stirring or by

**Table 1.** Parameters of the formation process providing control of microrod dimensions.

Parameter	Effect when parameter increases	Explanation
Shear rate	Decreased diameter, $D$ , decreased length, $L$ , lower polydispersity	Droplet deformation and breakup rate increase at higher shear
Media viscosity	Decreased diameter, $D$ , decreased length, $L$ , lower polydispersity	Higher viscosity leads to higher shear stress, similar to increased shear rate
SU-8 concentration in GBL	Increased $D$ and $L$	Polymer rod solidifies faster, losing less material

the viscosity of the dispersion medium. At larger capillary numbers theory predicts that smaller droplets will form that should solidify into thinner, shorter, and less polydisperse cylinders. An increase of the shear rate indeed decreased the characteristic sizes and the polydispersity of the microrods (Fig. 3). The decrease in the rod length,  $L$  (see Figs. 3a–d) was more pronounced than that of the diameter,  $D$ , probably because of the faster GBL dissolution and solidification of the smaller drops. In general, a five- to sevenfold increase in the rate of stirring leads to approximately a five- to ninefold decrease in the microrod length and a two- to threefold decrease in rod diameter.

A uniform and constant shear stress was required for producing rods of relatively narrow size distribution and aspect



**Figure 3.** Optical micrographs of SU-8 rods formed in a 50:50 vol.-% glycerol/ethylene glycol mixture at increasing stirring speed (corresponding to increasing shear rates): a) 200 rpm, b) 500 rpm, c) 900 rpm, and d) 1400 rpm. The scale bar is 50  $\mu\text{m}$ .

ratio; we used an electronic mixer with a high-shear impeller. The role of the shear stress for formation of uniform particles was demonstrated by performing synthesis in pure alcohols, the viscosity of which is low (1.07 mPa s at 25 °C for ethanol compared to 934 mPa s for pure glycerin). In this case, the process results in a high fraction of large, irregularly shaped particles. Thus, a relatively high medium viscosity,  $\mu_0$ , is required for high yield and low polydispersity, again in correlation to theoretical expectations that high values of  $Ca$  lead to formation of shorter and less polydisperse rods.

The concentration of SU-8 in GBL was another controlling parameter. The decrease of SU-8 concentration in the initial solution was expected to reduce the diameter of the rods, since the amount of SU-8 in an oil droplet will be smaller. However, lowering the SU-8 concentration in the GBL decreased both the length and diameter of the microcylinders, possibly because of a decrease of the droplet size during the emulsification process. At least three more experimental parameters—the interfacial tension, the rate of solvent attrition, and the temperature—can be used to control the particle sizes, aspect ratios, and polydispersity. A detailed study of the mechanism of action of all these parameters is under way.

We also experimented with various dispersion media, including pure glycerin and its mixtures with ethylene glycol, ethanol, methanol, or isopropanol. The procedure worked very well with most of them. The key to the successful synthesis is the miscibility of the SU-8 solvent, GBL, with the dispersion medium. If the two are poorly miscible the process results in an oil–oil emulsion that separates into two phases. The microrods may also be made from other polymers dissolved in

the emulsified phases, which we proved with experiments with polystyrene/SU-8 mixtures at a weight ratio of 1:2. Mixed polymer rods with characteristic sizes similar to pure SU-8 rods were formed.

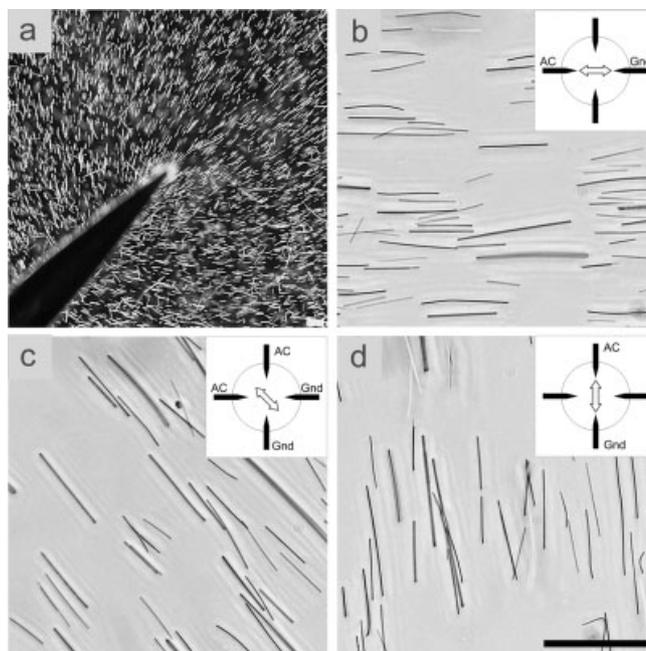
Irradiation of the SU-8 particles with UV light led to irreversible crosslinking of the photocurable resin, and the polymeric rods obtained could be easily separated by mild centrifugation and/or re-dispersed in water or in another liquid medium. Dispersal of the rods in water could be strongly enhanced by adding small amounts of anionic or nonionic surfactants (sodium dodecyl sulfate or Tween 20) that provide additional electrostatic or steric repulsion. If surfactant was not used, the hydrophobic SU-8 rods tended to aggregate reversibly in water, but dispersed easily again upon adding water-soluble surfactants.

The suspensions formed by this process allowed us to demonstrate at least three of the unique properties of the microrods that make them interesting for materials science. When rod-like particles in suspension are concentrated, they minimize their free energy by aligning in the same direction and forming "colloidal-liquid-crystal" phases.<sup>[6,7]</sup> Microrod suspensions concentrated above 2 wt.-% demonstrated the expected correlated alignment. Optical microscopy observations of the films deposited by drying drops of concentrated SU-8 rod dispersion in dodecane on glass substrates showed that they are made from multiple domains of ordered SU-8 rods (Fig. 2d). Concentrated rod suspensions probably form nematic liquid-crystal phases.

Two other valuable properties of these rod-like microparticles are their alignment in hydrodynamic flows and electric fields. Hydrodynamic rod alignment was immediately seen both by microscopy and by observing the macroscopic optical appearance of the suspension. Even a slight flow oriented the microrods so that their long axis pointed in the direction of the local flow. This could be used for effective visualization of liquid flow-lines on the microscale. Macroscopically, when the rod dispersions were shaken slightly by hand they acquired a silver color and were visually different when viewed from various angles, similar to liquid-crystalline phases; however, no significant degree of light polarization was measured with these diluted samples.

The SU-8 rods could also be manipulated and aligned by applying an external alternating current (AC) electric field. When suspended in water, the rods have higher net polarizability due to the conductivity and mobility of the counterion environment. The polarizability is highest along the long axis, and results in dielectrophoretic torque, leading to rod orientation parallel to the direction of the electric-field vector.<sup>[15]</sup> When a drop of rod dispersion was placed between two coplanar electrodes, the particles polarized and oriented perpendicularly to the electrodes, i.e., parallel to the lines of the electric field. When the electric field was inhomogeneous, the rods visualized the field lines, as illustrated by their orientation around a needle-like electrode in Figure 4a.

Rotating AC electric fields could be used to align the rods in arbitrary directions and spin them in the plane of their long



**Figure 4.** Optical microscopy images of SU-8 microrods dispersed in water and subjected to an AC electric field, created by needle-like electrodes: a) Rod orientation in the vicinity of a needle electrode at an AC field of 50 V/cm, 200 Hz; b–d) images of the particles in the middle of the experimental cell taken with higher magnification. The configurations of the energized electrodes are shown in the insets. The rods orient in the direction of the electric field and can be rotated. The scale bars are: a) 100  $\mu\text{m}$ , and b–d) 50  $\mu\text{m}$ .

axis. We used four point electrodes evenly distributed on the sides of a chamber of diameter 20 mm. By switching different electrode pairs on and off we could rotate the SU-8 microrods through 360° by eight steps of 45° each. Microscopy images of rods oriented in three different directions depending on the configuration of the energized electrodes are shown in Figures 4b–d. Such microrod orientation and rotation in electrical fields can be used in micromixers, for studying microrheology, or in various optical and display devices.

The main advantages of this method for fabrication of polymer microrods are its simplicity and easy scalability. Large amounts of polymer microrods could be rapidly fabricated in an inexpensive process similar to that for making latex microspheres. The method allowed control of their characteristic sizes and, to some extent, their polydispersity. The anisotropic particles were synthesized in dispersed state and could easily be concentrated and transferred to another medium. Rods could be fabricated from polymer mixtures. This microrod self-assembly into anisotropic phases could find application in colloidal substitutes for the widely used molecular liquid crystals, or in assembling new classes of microstructures. These anisotropic phases can, in turn, be used as templates for the assembly and alignment of nanowires and nanotubes. The rod-like particles could also be used in electrorheological fluids, as tracers in microfluidic devices, as modifiers of the rheological behavior of suspensions and paints, or as coatings with anisotropic optical properties.

## Experimental

The photocurable epoxy resin SU-8 (widely used as a negative resist for photolithography) was purchased from MicroChem. MA (USA), as a 63 wt.-% solution (SU-8 25) in  $\gamma$ -butyrolactone (GBL). The resin was used as received or diluted further with GBL. Analytical grade glycerin or its mixtures with ethylene glycol, ethanol, methanol, dodecane (all from Fisher Scientific, PA, USA), and isopropanol (LabChem. Inc., PA, USA) were used as dispersion media. GBL is soluble in all of the above solvents, which is a necessary condition for the microrod synthesis. The polystyrene used in the polymer mixtures was obtained from 1  $\mu$ m poly(styrene sulfate) FluoSpheres (Molecular Probes, OR, USA). The dried latex was dissolved in GBL prior to mixing with SU-8 solution.

Mixing of the phases was performed by a Servodyne electronic mixer (Cole-Parmer, IL, USA) providing a high speed of stirring (150–6000 rpm) at low torque. It was equipped with a high-shear impeller of diameter 50.8 mm (Lightin A-320, Cole-Parmer, IL, USA), inserted into a beaker of inner diameter of 62.2 mm. The dispersions were made by injecting 0.1–0.5 mL of 30–63 wt.-% solution of SU-8 in GBL into the continuously stirred viscous medium. Uniformly shaped, rod-like polymer particles were formed in all experiments at high shear rates. The dispersion formation was easily detected by the change in the solution turbidity; a milky color appears in the first 20–60 s after mixing the phases. The optimal time for rod formation at these conditions was about 10 min.

After completion of the initial synthesis, SU-8 rods could be additionally crosslinked by exposing them to 365 nm UV light (BL-100A UV lamp, Blak-Ray, CA, USA) for 15 min. This made them insoluble in any common solvent and they could then be transferred to another aqueous or organic medium. For example, dispersions of SU-8 rods in water were prepared by diluting the initially synthesized dispersion with water containing  $5 \times 10^{-4}$  wt.-% sodium dodecyl sulfate or Tween 20 and repeating a few centrifugation/washing cycles. Dispersions in pure alcohols could be prepared in a similar way.

The alternating electric field (AC) experiments with coplanar electrodes were carried out by applying an AC field of  $75 \text{ V cm}^{-1}$  and 200 Hz in a cell similar to that in [16]. The electrorotation experiments were carried out by using an experimental cell made from a round perfusion chamber with a thickness of 0.5 mm and diameter of 20 mm (Cover Well, Grace Bio-Labs, OR, USA) sealed by a microscope glass slide. Four needle electrodes were positioned at the chamber periphery and connected to an AC generator and amplifier [16]. The dispersion of SU-8 rods in water was inserted into the chamber by a micropipette. An electric field of  $50 \text{ V cm}^{-1}$  and 200 Hz was applied and its direction was changed by switching on and off different electrode pairs (Figs. 4c,d).

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## Mesoporous Silica Platelets with Perpendicular Nanochannels via a Ternary Surfactant System\*\*

By Bi-Chang Chen, Hong-Ping Lin,\* Man-Chien Chao, Chung-Yuan Mou, and Chin-Yuan Tang

Since the discovery of mesoporous silica, there have been numerous reports on the fabrication of thin films of these materials.<sup>[1–3]</sup> In particular, the hexagonally arranged, linear channel systems of MCM-41 and SBA-15 have received the most attention.<sup>[4,5]</sup> Up to the present, in all reports, the chan-

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