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Interfacial Control in Photochromic Systems

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Overview

- Photo-Controlled surfaces
- Photo-Controlled Colloidal System
- Photo-physical effects in Colloids
- 2D and 3D Patterning with Colloids

Contributors

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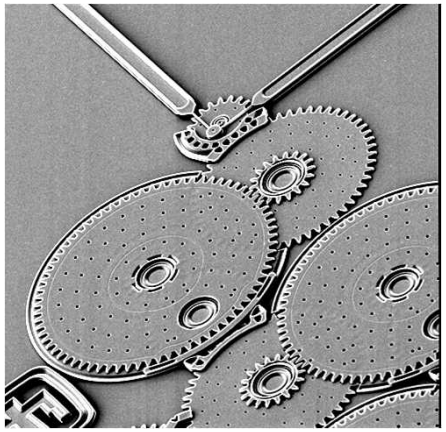
Liu Jiang, Tim Long, John Lean

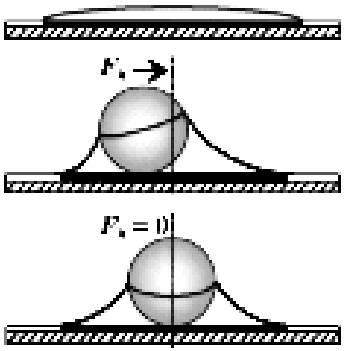
Motivation

Photochemical control of physical interaction processes on the molecular level



MEMS: ***tribology***

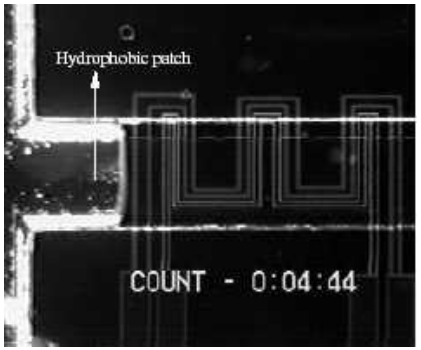
Nano-interaction management



Self-assembled nanostructures:

Microfluidics: ***rheology***

J. Micromech. Microeng.., 13, (2003), p. 261



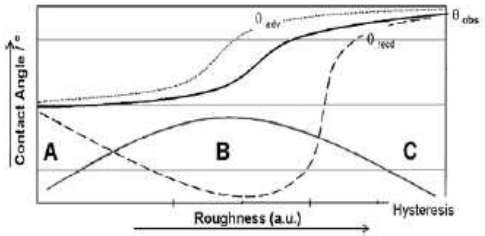


Figure 1. Wettability vs. roughness (schematically). (A) Smooth surface, (B) Wenzel Regime, (C) superhydrophobic surface. θ_{obs}: observable (static) c.a., θ_{adv}: advancing c.a., θ_{recd}: receding c.a.

J. Sol-Gel Sci. Tech., 26, (2003), p.789

- **Fundamental competency for the realization and application of high-surface-area microsystem architectures and the controlled fabrication and utilization of nanostructured materials**

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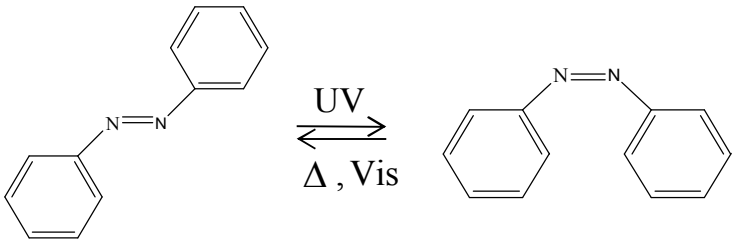
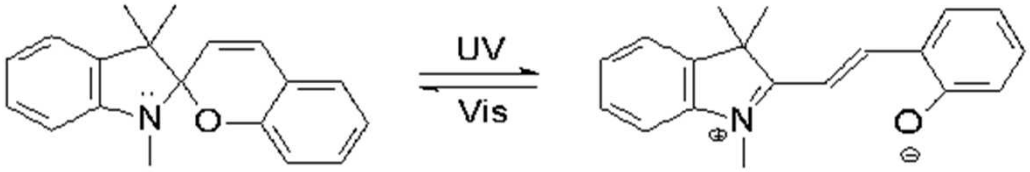
Photochromic Molecules

- Azobenzene



trans cis

- Spiropyran



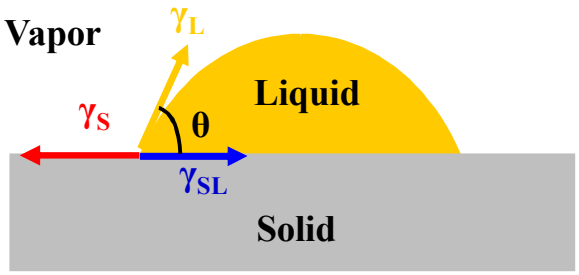
Closed Open

Dipole moment of these molecules are reversible, and monitored by:

- changes in optical profile (UV/Vis spectrum)

- alteration of surface energy (contact angle measurements)

Modeling of Surface Energy from Contact Angle Measurements



Young's equation

$$\gamma_S = \gamma_{SL} + \gamma_L \cos\theta$$

Van Oss theory

$$\gamma_S = \gamma_S^{LW} + \gamma_S^{AB} \qquad \gamma_S^{AB} = 2\sqrt{\gamma_S^- \gamma_S^+}$$

$$\gamma_L(1+\cos\theta) = 2\left(\sqrt{\gamma_S^{LW}\gamma_L^{LW}} + \sqrt{\gamma_S^-\gamma_L^+} + \sqrt{\gamma_S^+\gamma_L^-}\right)$$

Surface energy separated into dispersive (LW), electron donating (-) and electron accepting (+) components

Van Oss Modeling of Azobenzene Surfaces

Azobenzene modified surface energy (mJ/m^2) was calculated by using three fluids of known dispersive, acid and base components.

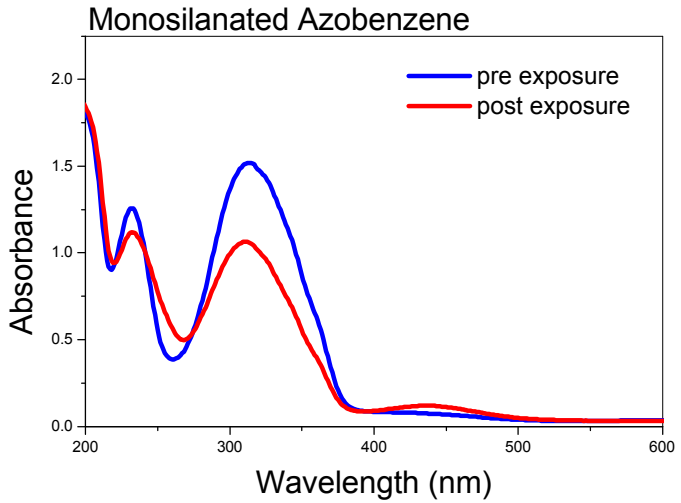
$$\gamma_L(1+\cos\theta) = 2\left(\sqrt{\gamma_S^{LW}\gamma_L^{LW}} + \sqrt{\gamma_S^{-}\gamma_L^{+}} + \sqrt{\gamma_S^{+}\gamma_L^{-}}\right)$$

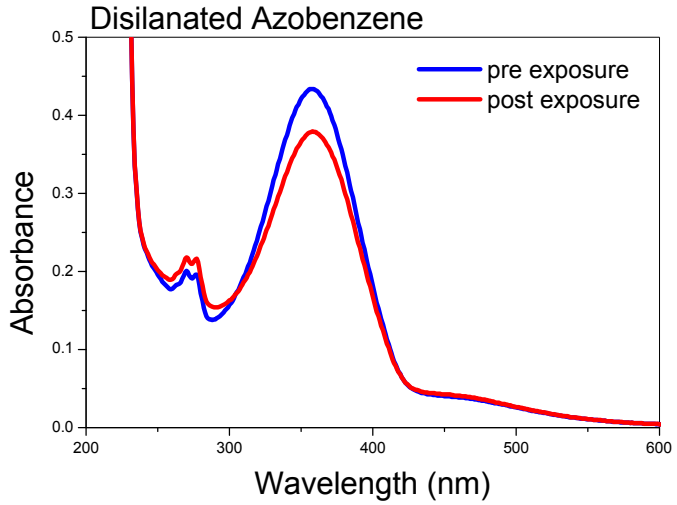
Table: Surface tensions and components of probe liquids (mJ/m^2) suggested by Van Oss

Liquid	γ_L	γ_L^{LW}	γ_L^{-}	γ_L^{+}	γ_L^{AB}
Polar					
DI water	72.8	21.8	25.5	25.5	51.0
ethylene glycol	48.0	29.0	47.0	1.92	19.0
formamide	58.0	39.0	39.6	2.28	19.0
Nonpolar					
diiodomethane	50.8	50.8	--	0	0
1-bromonaphthalene	44.4	44.4	0	0	0

Van Oss Modeling of Azobenzene

Contact Angle Measurements using three fluids of known dispersive, acid and base character.





Solvent Parameters	σL	σD	σ-	σ+
Water	72.8	21.8	25.5	25.5
Diiodomethane	50.8	50.8	0	0
Formamide	58.2	39	39.6	2.28

$$\sqrt{\left(\sigma_l^D\right)\left(\sigma_s^D\right)}+\sqrt{\left(\sigma_l^-\right)\left(\sigma_s^+\right)}+\sqrt{\left(\sigma_l^+\right)\left(\sigma_s^-\right)}=\frac{\sigma_l\left(Cos\Theta+1\right)}{2}$$

Azobenzene (Monosilanated)	σ^D	σ^+	σ^-
Unexposed	40.08	0.10	1.23
Exposed	40.44	0.07	7.89
Azobenzene (Disilanated)			
Unexposed	29.10	0.02	7.24
Exposed	29.44	0.95	7.15

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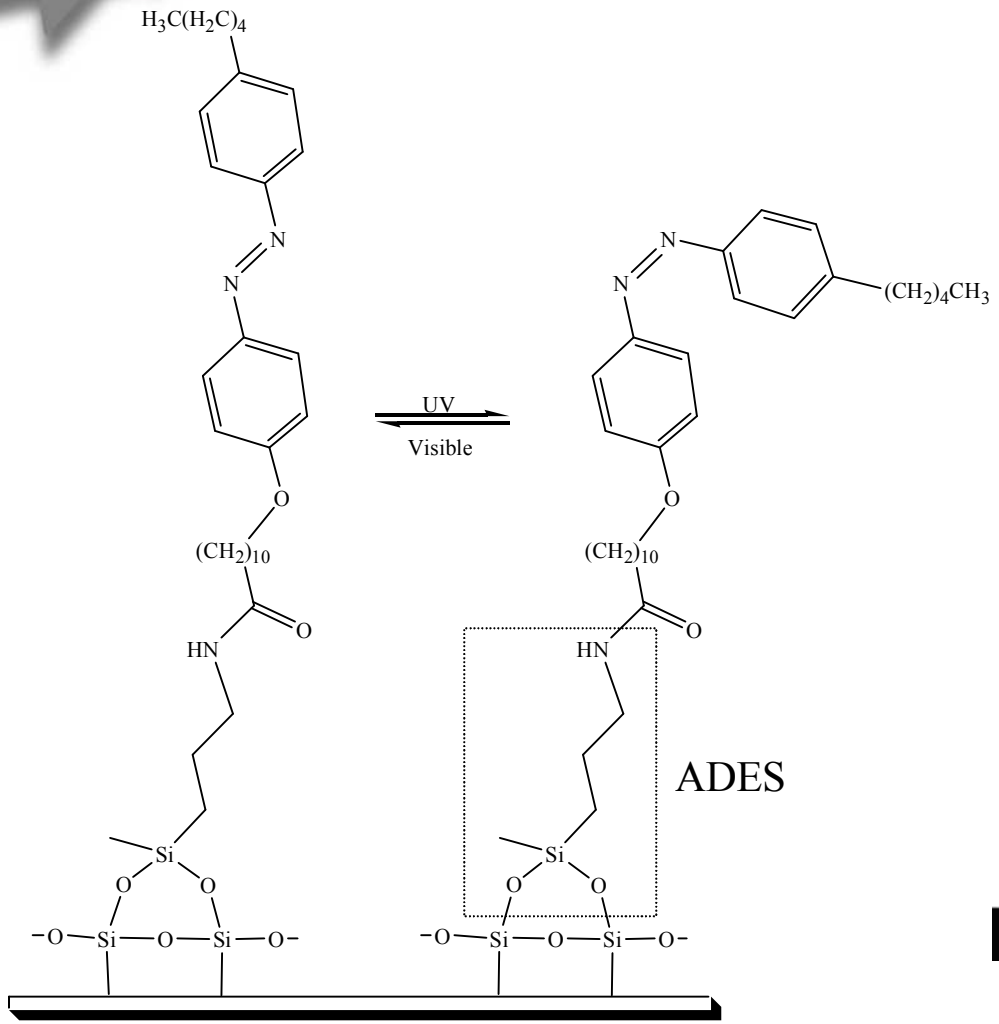
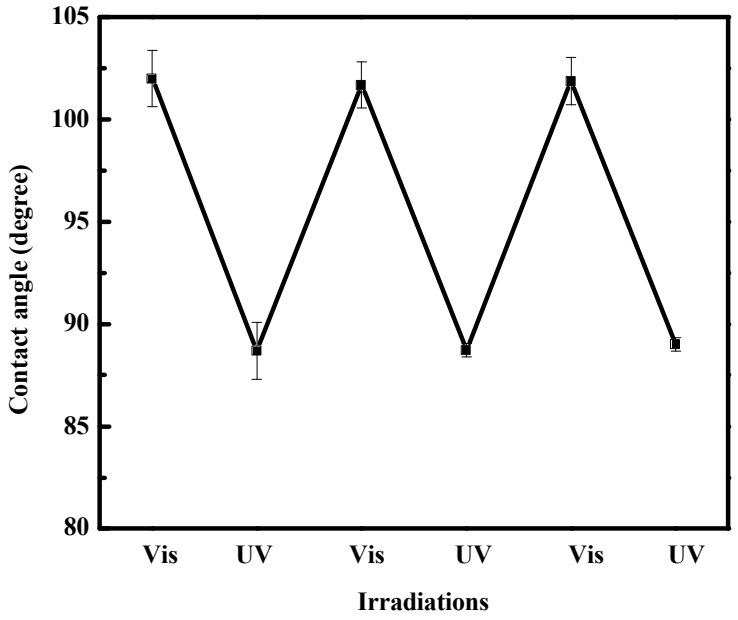
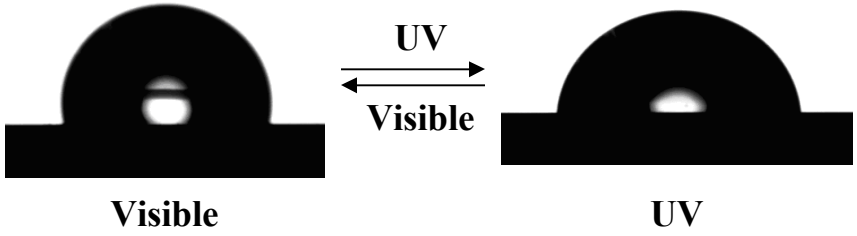
Surface Design with Azobenzene





Wetting angle



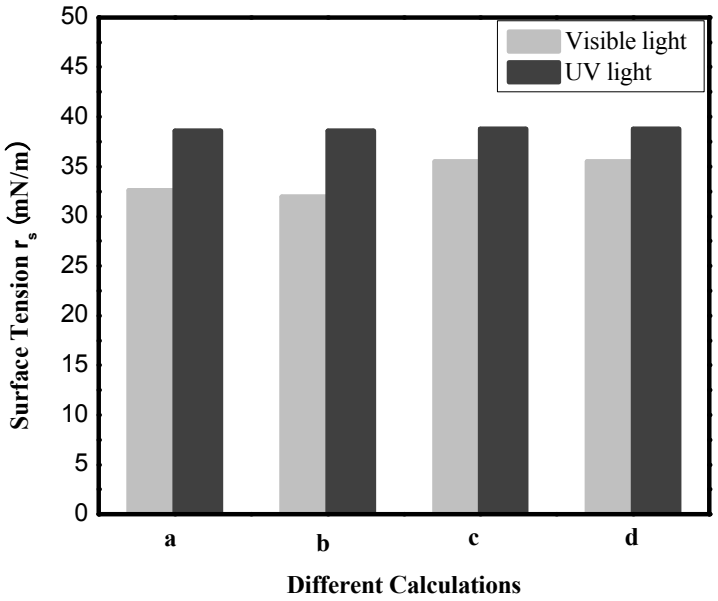
Azobenzene molecules attach to the surface through aminopropylmethyldiethoxysilane(ADES)

Visible	UV
100° -102°	88° -90°

Van Oss Modeling of Azobenzene Surfaces

Azobenzene modified surface energy (mJ/m^2) was calculated by using three fluids of known dispersive, acid and base components.

$$\gamma_L(1+\cos\theta) = 2\left(\sqrt{\gamma_S^{LW}\gamma_L^{LW}} + \sqrt{\gamma_S^{-}\gamma_L^{+}} + \sqrt{\gamma_S^{+}\gamma_L^{-}}\right)$$



Calculations	Illumination	γ_S^{LW}	$(\gamma_S^{+})^2$	γ_S^{-}	γ_S
a)	Visible	32.59	-0.09	0.42	32.59
	UV	38.59	-0.21	3.23	38.59
b)	Visible light	32.02	-0.69	1.99	32.02
	UV	38.59	-0.55	4.57	38.59
c)	Visible	35.49	-0.28	0.36	35.49
	UV	38.83	-0.22	3.22	38.83
d)	Visible	35.49	-1.00	2.07	35.49
	UV	38.83	-0.57	4.58	38.83

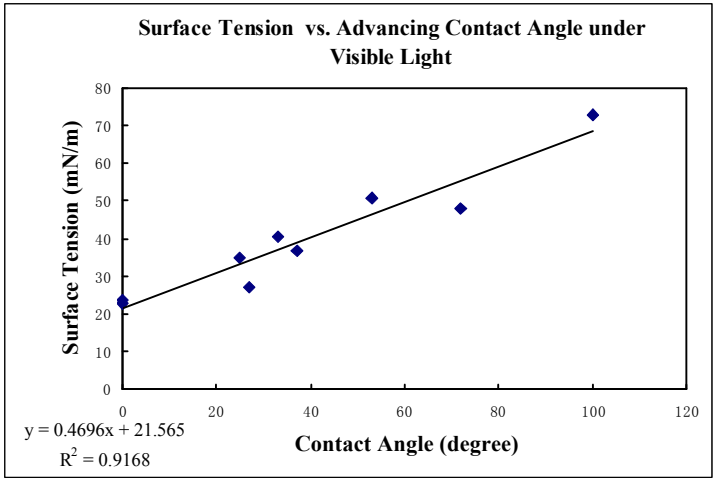
a) Using DI water, Ethylene glycol, diiodomethane to calculate; b) Using DI water, Formamide, diiodomethane to calculate; c) Using DI water, Ethylene glycol, 1-Bromonaphtalene to calculate; d) Using DI water, Formamide, 1-Bromonaphtalene to calculate

Contact Angle Measurement with Liquids

Table: Contact angle on azobenzene modified surface with various solvents (degree).

Solvents	trans	cis	Surface Tension (mN/m)	Switching Angle ($\theta^{trans}_{adv} - \theta^{cis}_{adv}$)
	θ_{adv}	θ_{adv}		
acetonitrile	27.0±0.55	11.5±0.87	27	15.5
benzonitrile	32.0±2.0	17.0±0.76	38.65	15
DI water	101.3±0.98	88.9±0.86	72.8	12.4
diiodomethane	54.1±0.49	42.1±1.08	50.8	12
dimethylformamide	37.5±1.74	26.8±0.53	36.8	10.7
formamide	82.2±1.36	71.5±0.66	58.2	10.7
1-methylnaphthalene	33.3±1.09	23.3±0.34	40.5	10
ethylene Glycol	73.2±0.73	63.9±0.71	48	9.3
1-bromonaphthalene	38.0±0.67	29.7±0.40	44.4	8.3

Linear relationship between surface tension of solvents and its contact angle on azobenzene surface



Droplet Mobility Requirements

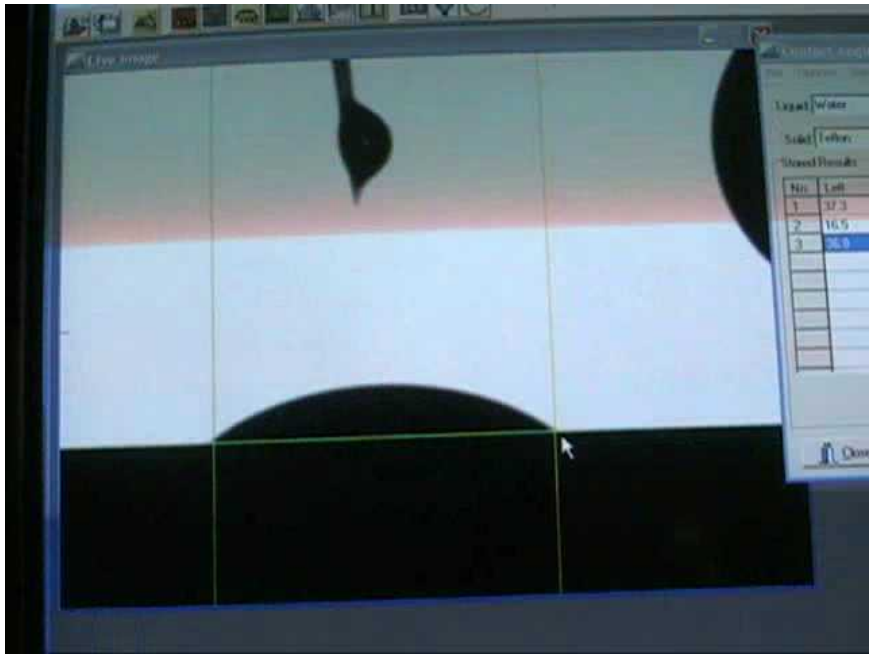
Requirement: The receding contact angle (trans-isomer) under visible light must be larger than advancing contact angle (cis-isomer)

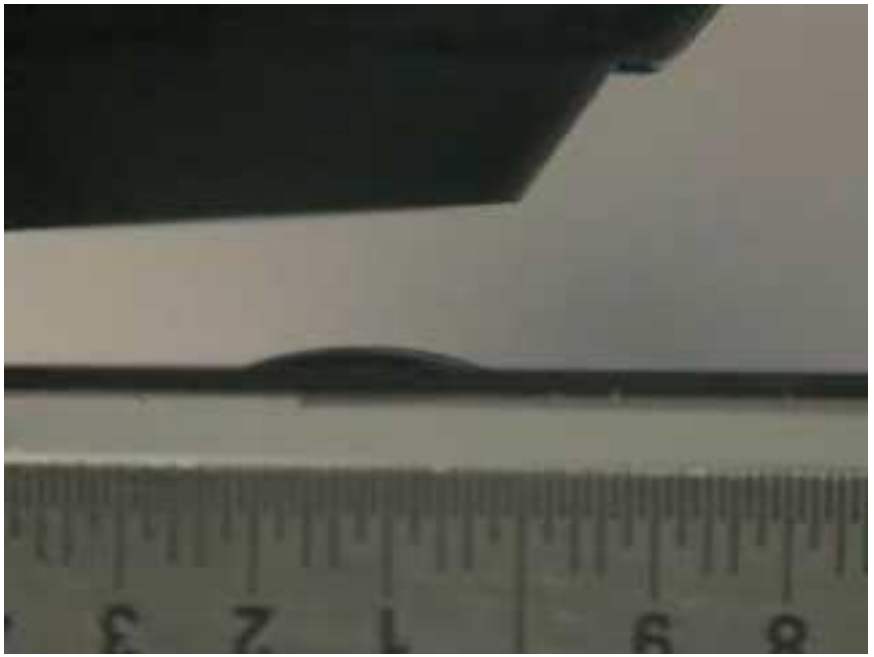
Table: Contact angle measurements (degree) on azobenzene modified surface with various solvents.

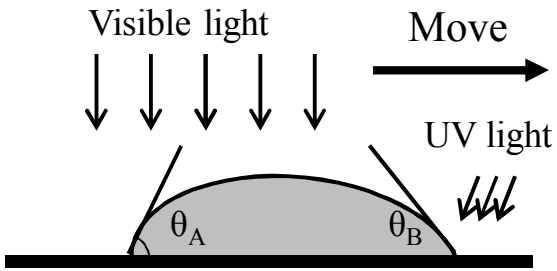
Liquid	trans		cis	
	θ_{adv}	θ_{rec}	θ_{adv}	θ_{rec}
Fulfill motion requirement				
benzonitrile	32.0±2.0	26.2± 1.25	17.0± 0.76	--
diiodomethane	54.1± 0.49	43.7± 0.58	42.1± 1.08	33.1± 1.28
dimethylformamide	37.5± 1.74	29.6± 1.47	26.8± 0.53	19.0± 1.34
1-bromonaphthalene	38.0± 0.67	33.5± 0.5	29.7± 0.40	24.6± 0.51
acetonitrile	27.0± 0.55	19.4± 1.41	11.5± 0.87	--
1-methylnaphthalene	33.3± 1.09	23.5± 0.77	23.3± 0.34	13.5± 0.56
Not fulfill motion requirement				
DI water	101.3± 0.98	76.0± 2.0	88.9± 0.86	69.8± 1.4
formamide	82.2± 1.36	62.8± 1.18	71.5± 0.66	58.2± 1.01
ethylene glycol	73.2± 0.73	49.0± 1.15	63.9± 0.71	41.5± 0.49

Motion of 1-Bromonaphthalene by UV Light

We demonstrate the idea of using light to move liquids on a photoresponsive surface.



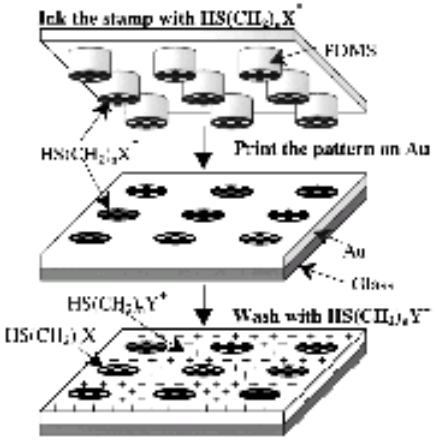


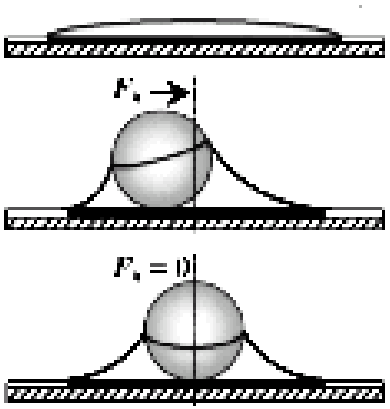


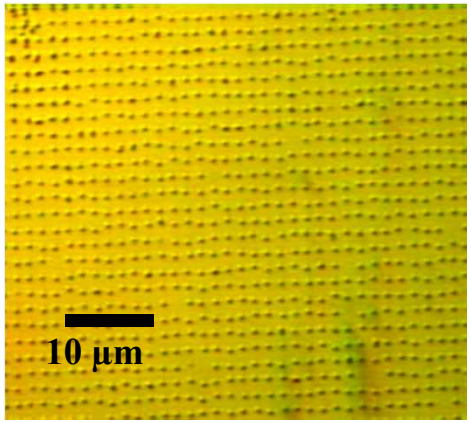
Surface Templating of Particle Assembly

Cationic/anionic functionalization of particles and substrates enables
-single particle spectroscopy
-definition of ABAB composite architectures for energy transfer

Particle templating via microcontact printing (mCP) of heterogeneous monolayers:







Particle templating allows:

- more complicated structures: FCC, BCC, diamond cubic
- introduction of defects (superlattices)
- stamping of waveguides

Clem and Payne, "Monolayer-mediated patterning of electroceramics," J. Electroceram, 3(2), 163 (1999).
Aizenberg, Braun, and Wiltzius, "Patterned colloidal deposition by electrostatic and capillary forces, Phys. Rev. Lett. **84**(13), 29973000 (2000).

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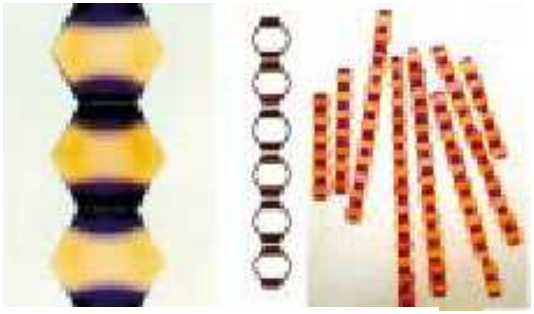
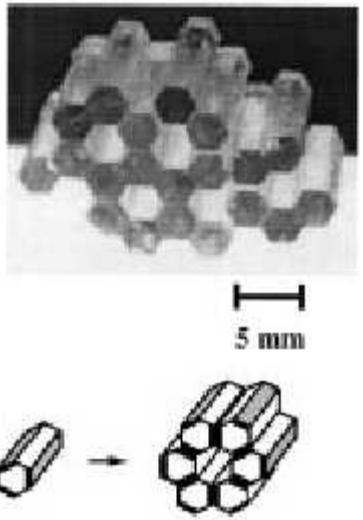
Heterogeneous Particle Assembly

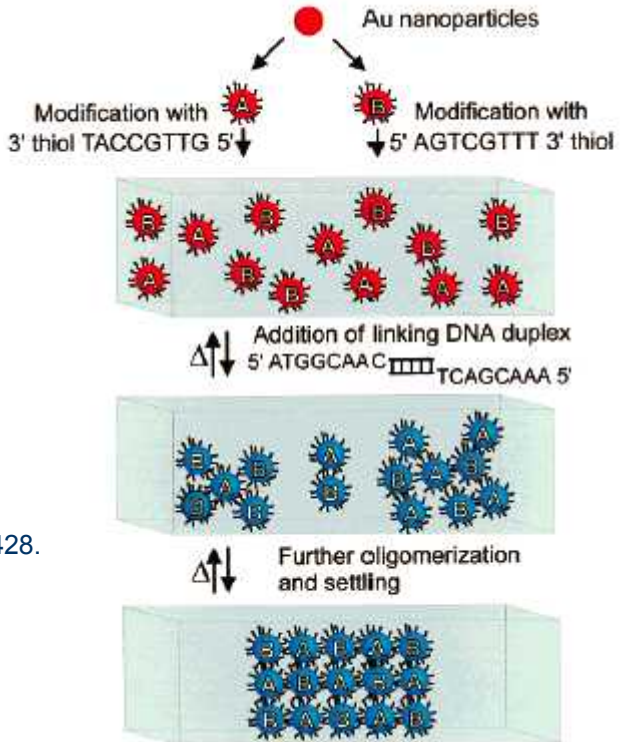
Are there analogs to directed assembly at the colloidal scale?



Ned Bowden, et al. *J. Am. Chem. Soc.* **121** (1999) 5373-5391



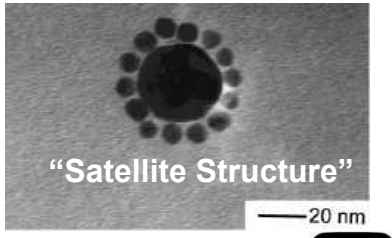
S.R.J. Oliver at al. *J. Colloid Interface Sci.*, **224** (2000) 425-428.



* Chad Mirkin, *Inorg. Chem.* 39 (2000)
Hiddessen et al. *Langmuir*, 16 (2000)

There are <u>novel</u> systems to study with these methods…

- Electrostatic Assembly (i.e. Ionomeric Gel).
- Photosensitive stabilization mechanisms or bonding.
  (Zaitsev SY, et al. Supramolecular Science, **4** (1997) 519-524.)
- Biological Surface Recognition*
  DNA, nucleotides, peptides, lipids …
  Observed structures include colloidal micelles, clusters, rings, and chains.
- Structured layer deposition using non-spherical particles
  (Choi et al. Langmuir (2000) **16**, 2997-2999.)



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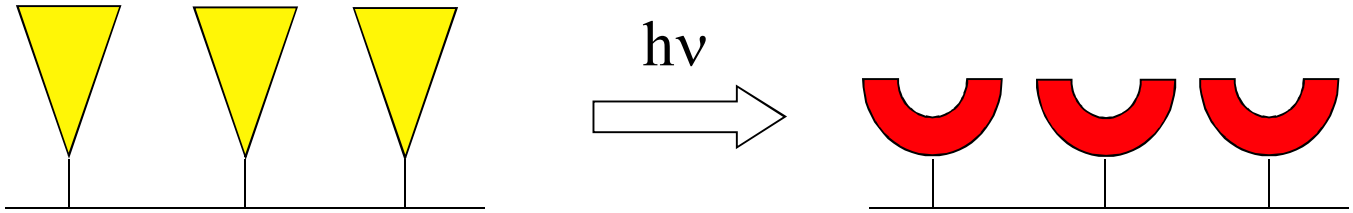
Photochemical Control of Surface Interactions

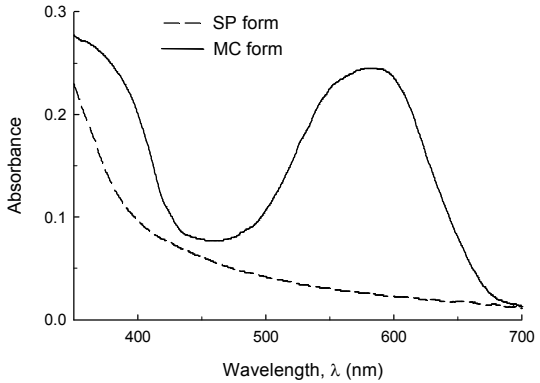
Photochemical alteration of an organic moiety resulting in a change in steric, electrostatic parameters



- Real-time manipulation of surface properties
- Switchable/reversible (new operational modes)
- Non-invasive/remote control over stability

Manifested in:

- changes in optical profile (UV/Vis spectrum)
- alteration of surface energy
- photocontrolled colloidal stability



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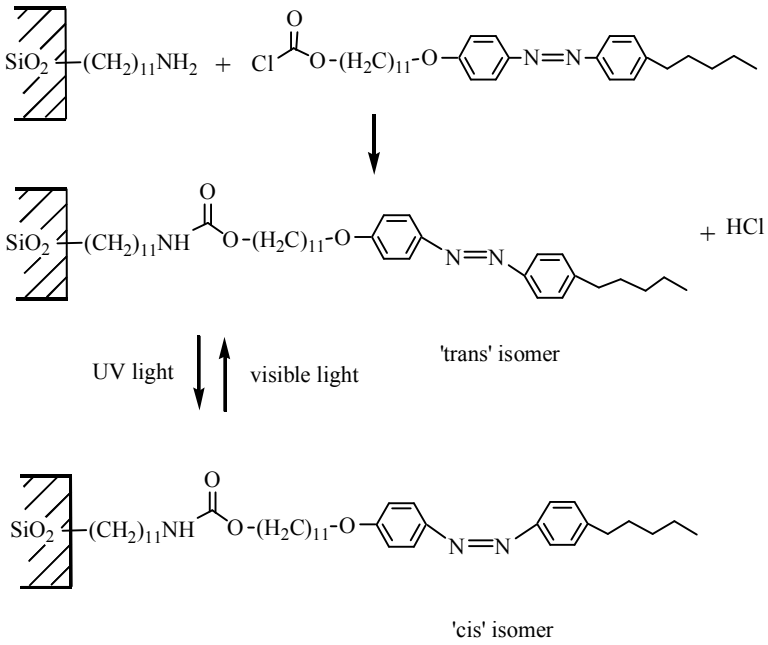
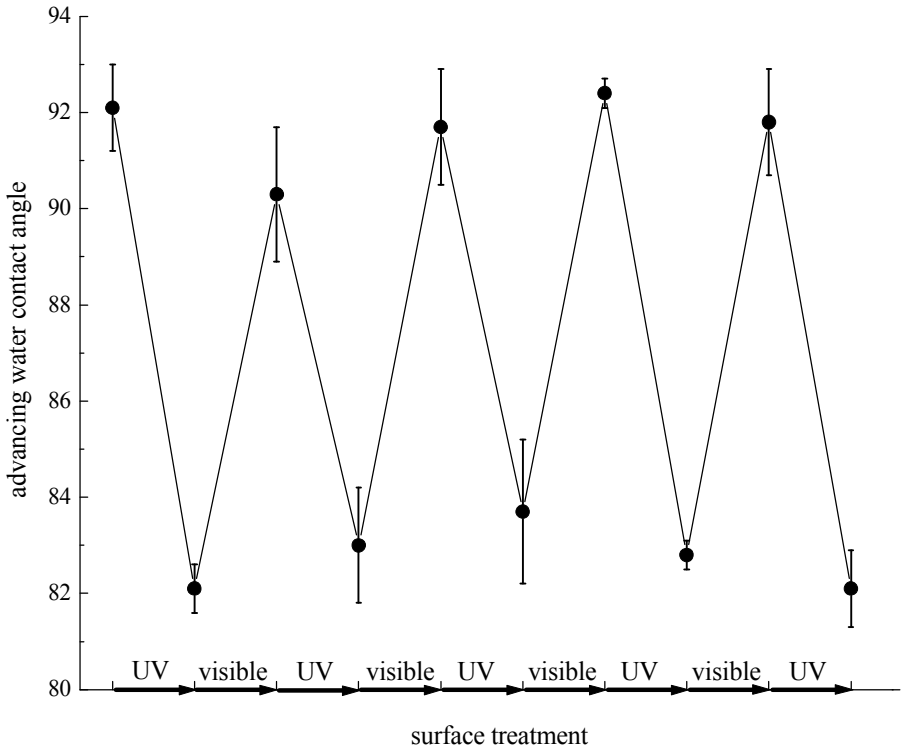
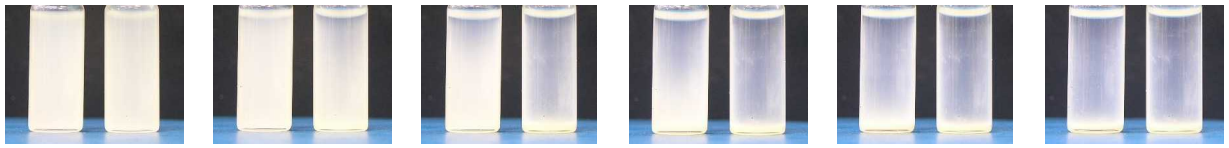
Azobenzene Derivatized Surface and Colloid Aggregation Response





Left sample is visible exposed
Right sample is UV exposed

Sedimentation Rate Difference



12 sec 60 sec 120 sec 180 sec 240 sec 300 sec

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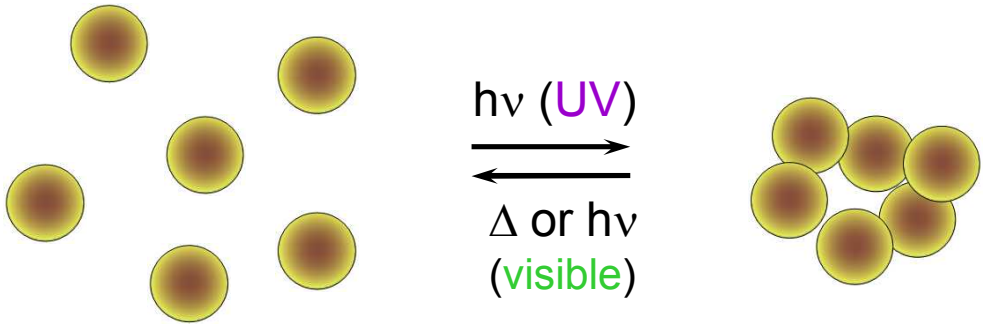
Interfacial Energy Calculation

A transition from negative to positive interfacial energy is desired for photo control of wetting characteristics.

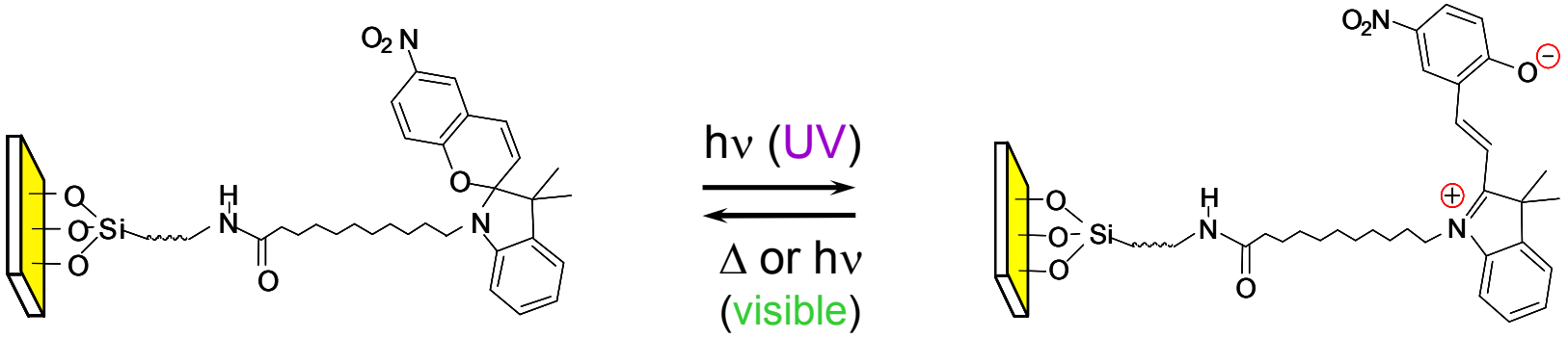
$$\Delta G_{131} = -2\left(\sqrt{\gamma_1^D} - \sqrt{\gamma_3^D}\right)^2 - 4\left(\sqrt{\gamma_1^+\gamma_1^-} + \sqrt{\gamma_3^+\gamma_3^-} - \sqrt{\gamma_1^+\gamma_3^-} - \sqrt{\gamma_3^+\gamma_1^-}\right)$$

	Pre exposure	Post Exposure	Change in Surface Energy
Cyclohexane	-4.69	-7.03	-2.34
Chloroform	2.72	13.51	10.78
Tetrahydrofuran	-12.29	-16.31	-4.02
Diiodomethane	-2.67	-3.83	-1.16
Water	-80.14	-49.41	30.73
Formamide	-24.77	-17.46	7.31
Glycerol	-43.54	-33.54	10.00
Ethylene Glycol	-26.37	-20.52	5.85

Reversible Light-regulated Colloidal Interactions



Approach: Utilize reversible photochemical reaction



closed form

Spirobenzopyran (SP)

zwitterionic form

Merocyanine (ME)

- Reversible aggregation of SP-coated colloidal particles in non-polar solvents
  K. Ichimura, et al. *J. Mat. Chem.* **4**, 883 (1994)

Strategic Approach to forming a Photosensitive Colloidal System

Demonstrate Reversible aggregation and dispersion of colloidal particles by light based methods.

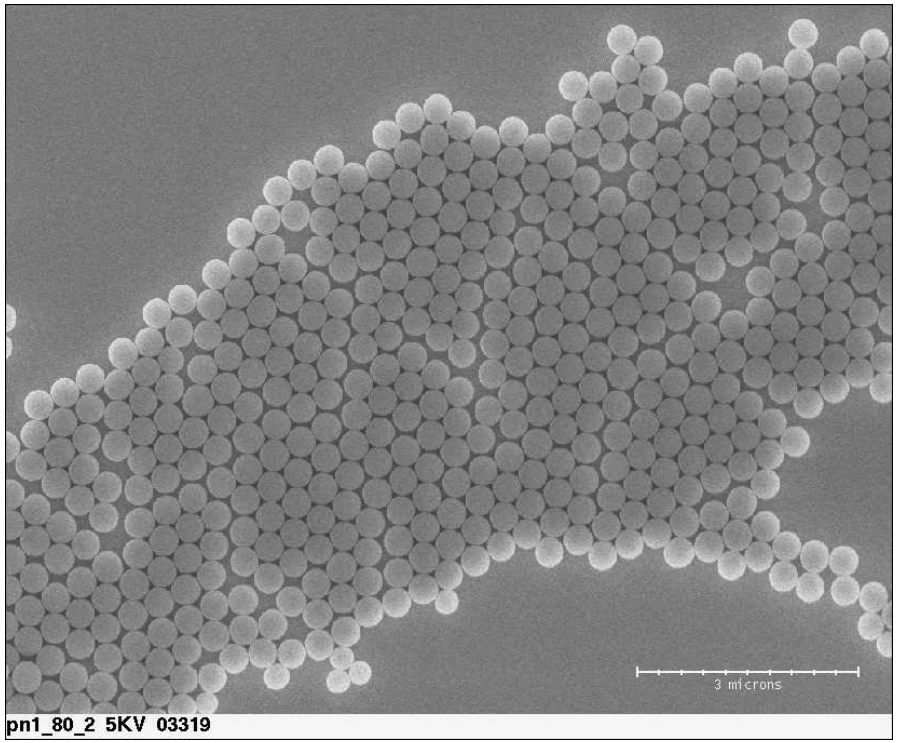
Particle system: Silica

- monodisperse,
- spherical
- common model system
- surface chemistry can be modified using silanes

Organic component: spiropyran

- large dipole moment change
- forms a zwitterionic state
- literature states solubility changes of SPMMA polymers in nonaqueous solvents

Approach: derivatize particles with layers of photo-switchable polymer.



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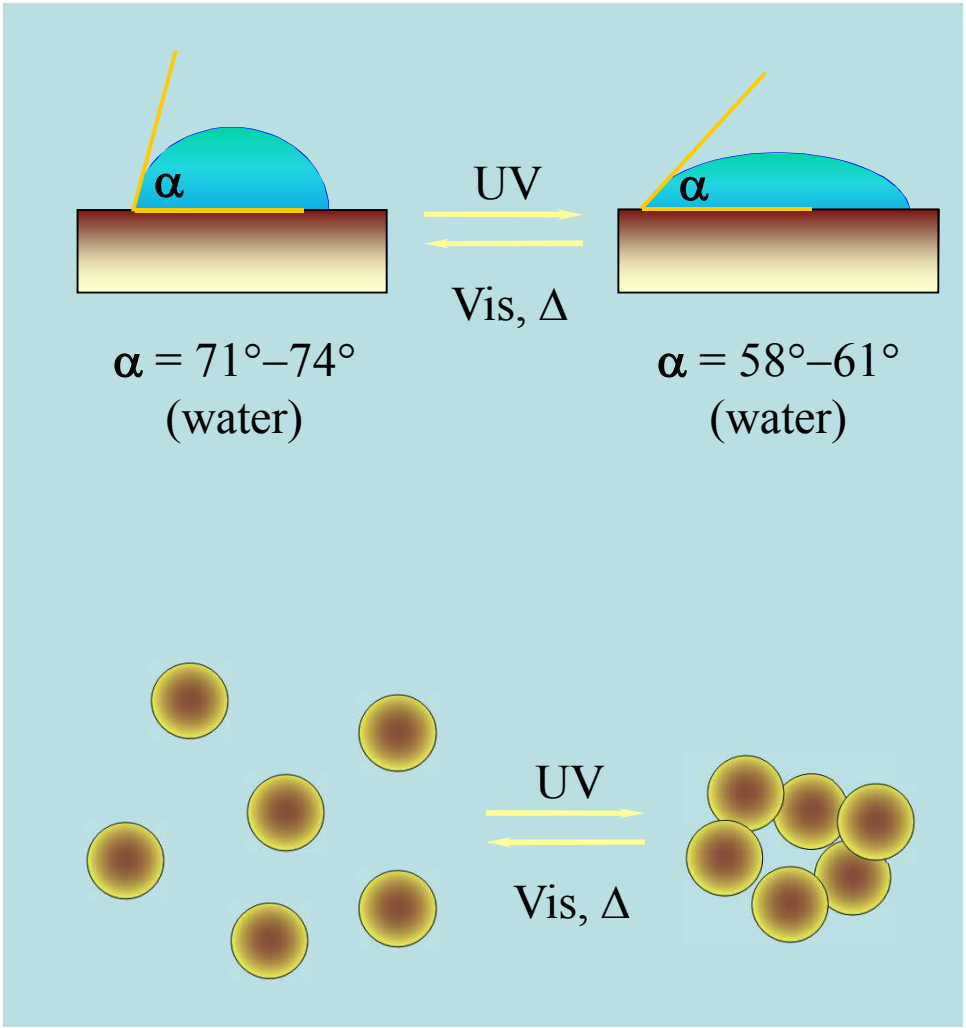
Effects of Photo-Isomerization on Spiropyran Monolayers

- Spyrobenzopyran monolayers on flat surfaces demonstrate reversible wettability change
- Contact angle altered by as much as 13° (Garcia, *et al.*)
- Colloidal particles covered with SP monolayers and dispersed in non-polar solvents undergo reversible aggregation (Ichimura, *et al.*)
- Change brought about by marked polarity difference between closed SP and open ME forms



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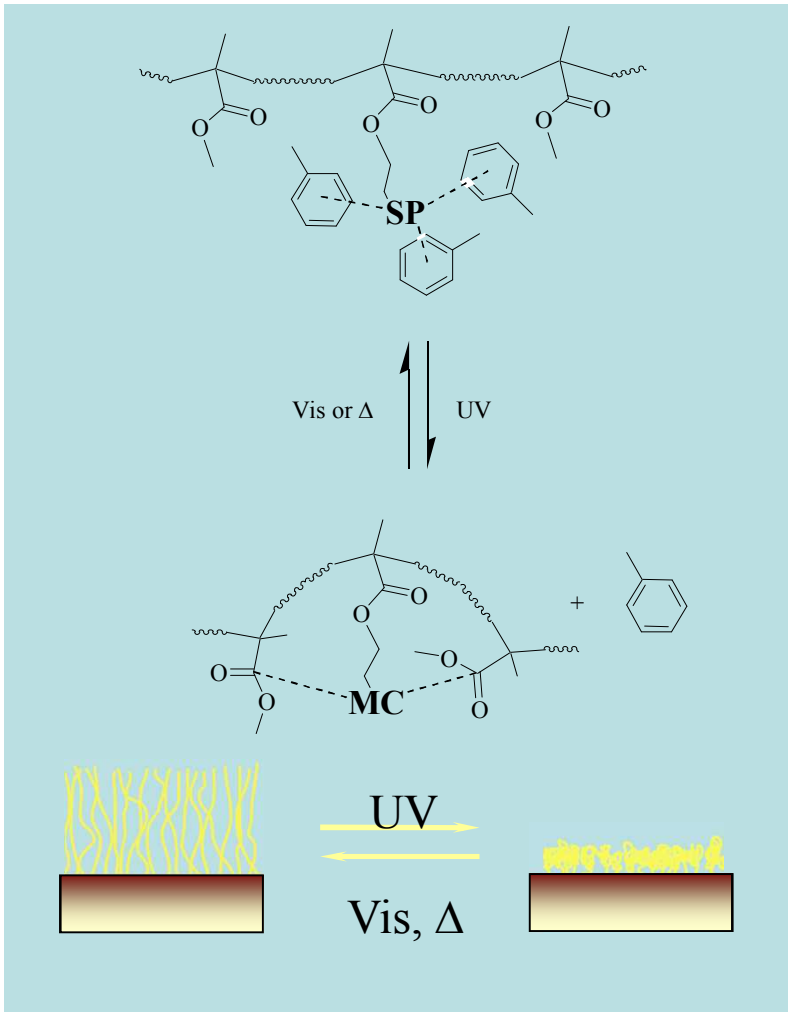
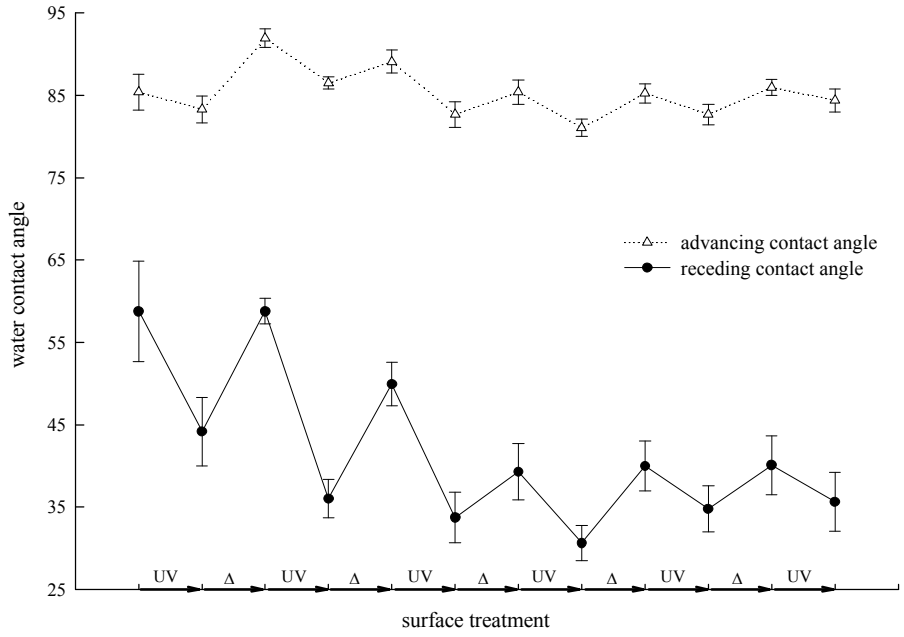
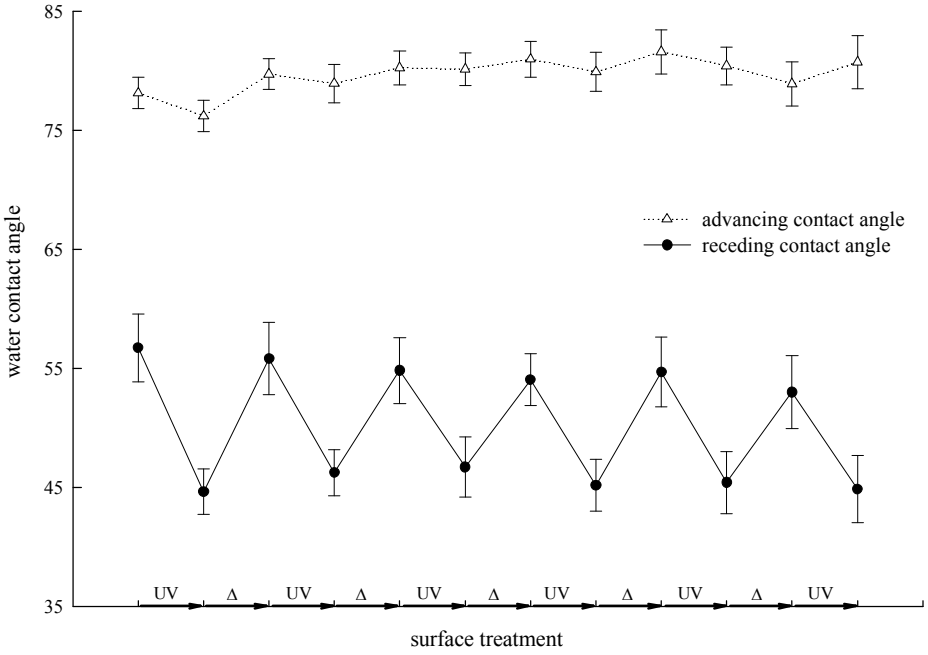
Effects of photo-isomerization on SP/MMA polymers

- Free SP/MMA polymers reduce solution viscosity upon UV irradiation due to polymer chain shrinkage (Irie *et al.*, Golburt *et al.*)
- Chain shrinkage is most pronounced in non-polar solvents (*e.g.*, toluene) and disappears in polar media (*e.g.*, dichloroethane)
- Process caused by specific intramolecular solvation of polar merocyanine form by MMA ester side groups in competition with solvation by solvent
- Polymer shrinkage also demonstrated on flat surfaces by as much as 30 nm for 45 nm thick films in non-polar solvents (Ito, *et al.*)



Wetting of SPMMA monolayers versus SPMMA-*co*-MMA (20% SP) polymers





✓ Loss of switching ability is prevented by using polymeric layers instead of organic monolayers.

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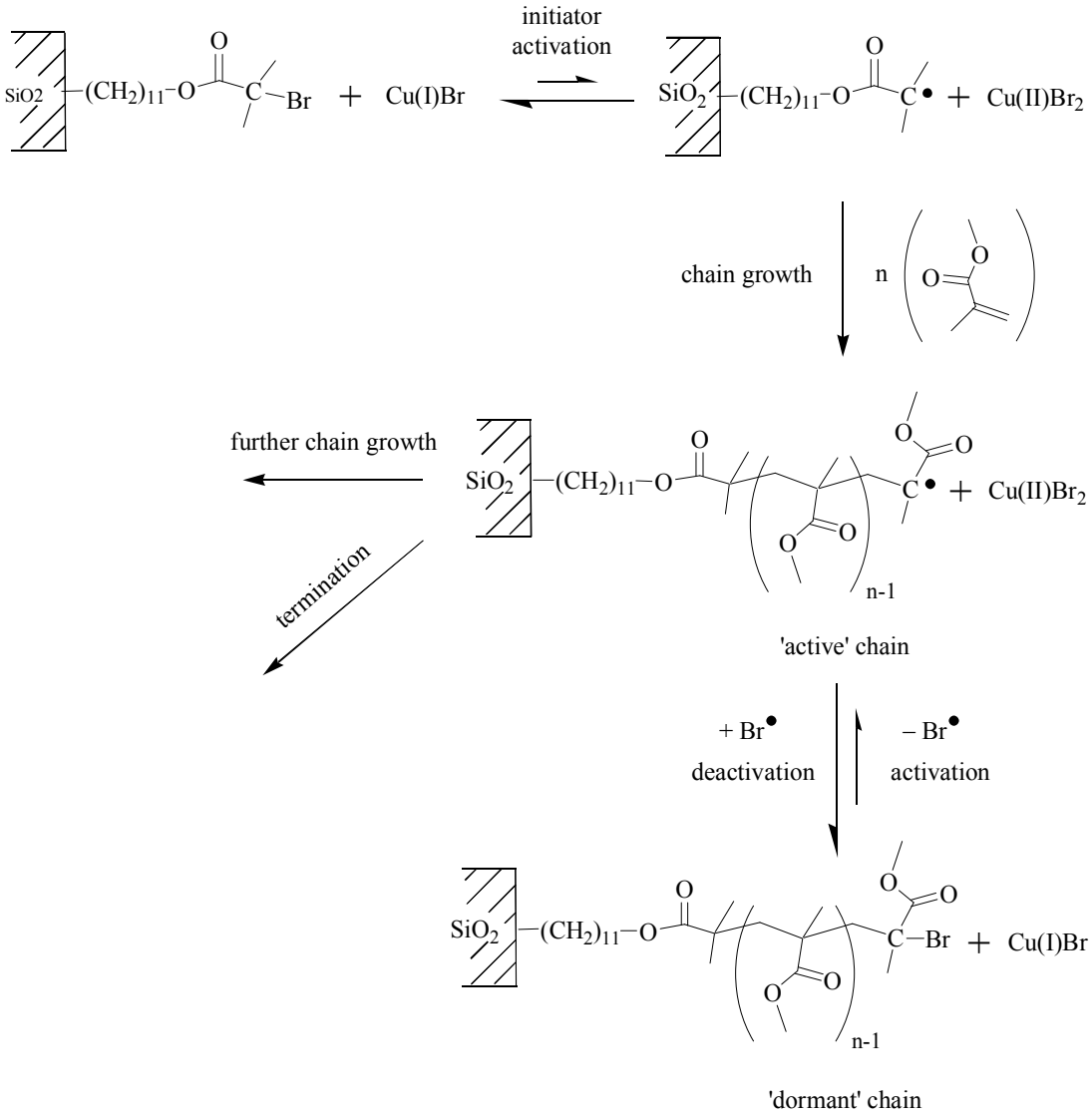
Atom Transfer Radical Polymerization (ATRP) Reaction Mechanism



Properties:

- Controlled growth of uniform layers
- Reaction kinetics allow for thickness control
- Requires oxygen free environment or techniques
- Large number of catalyst systems to test

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ATRP Reaction Kinetics for SP/MMA Colloids

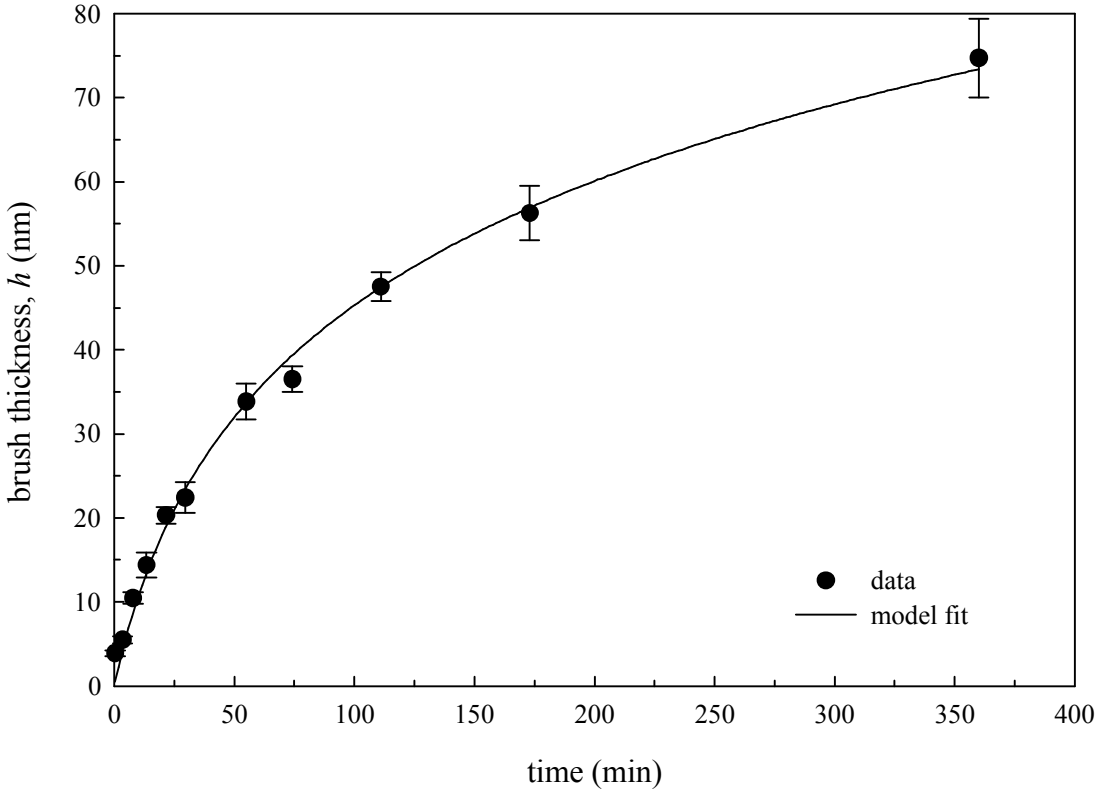
Model

$$\frac{d[A]}{dt} = -k_1[A]^2$$

$$\frac{dh}{dt} = k_2[A]$$

$$h = (k_2/k_1)\ln(1 + k_1[A]_0 t)$$

- Fit to the model of Kim *et al.*† indicates the presence of termination reactions



† J. –B. Kim, W. Huang, M. L. Bruening, G. L. Baker, "Synthesis of Triblock Copolymer Brushes by Surface-Initiated Atom Transfer Radical Polymerization," *Macromolecules* **35** (2002) 5410-5416.

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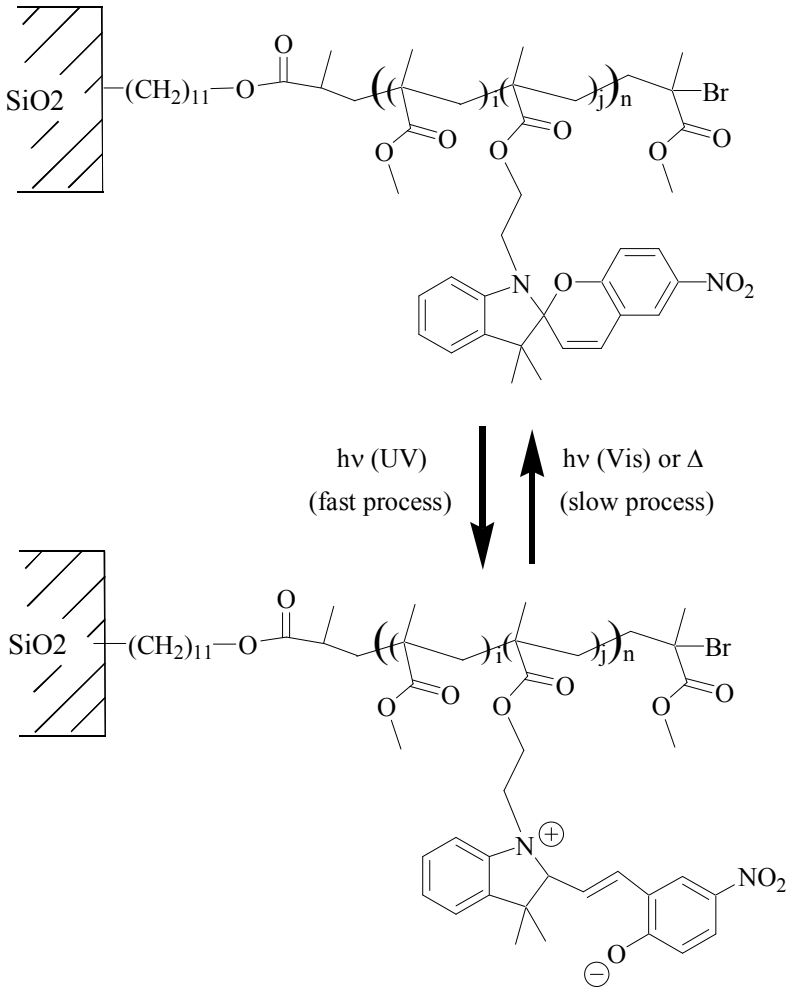
Photo-induced switching



- In non-polar solvents spyrobenzopyran (SP) molecule exists in closed form
- UV excitation causes ring-opening photoisomerization
- After UV treatment molecule exists in zwitterionic merocyanine (ME) form
- Corresponding polarity change alters polymer conformation and polymer-solvent interactions
- Process is reversible (albeit slower) upon exposure to visible light or heat

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Behavior of core-shell silica/polymer particles in toluene

(data shown for 284 nm silica core and 20 nm SP/MMA polymer shell containing 20 mol % of SP molecules)

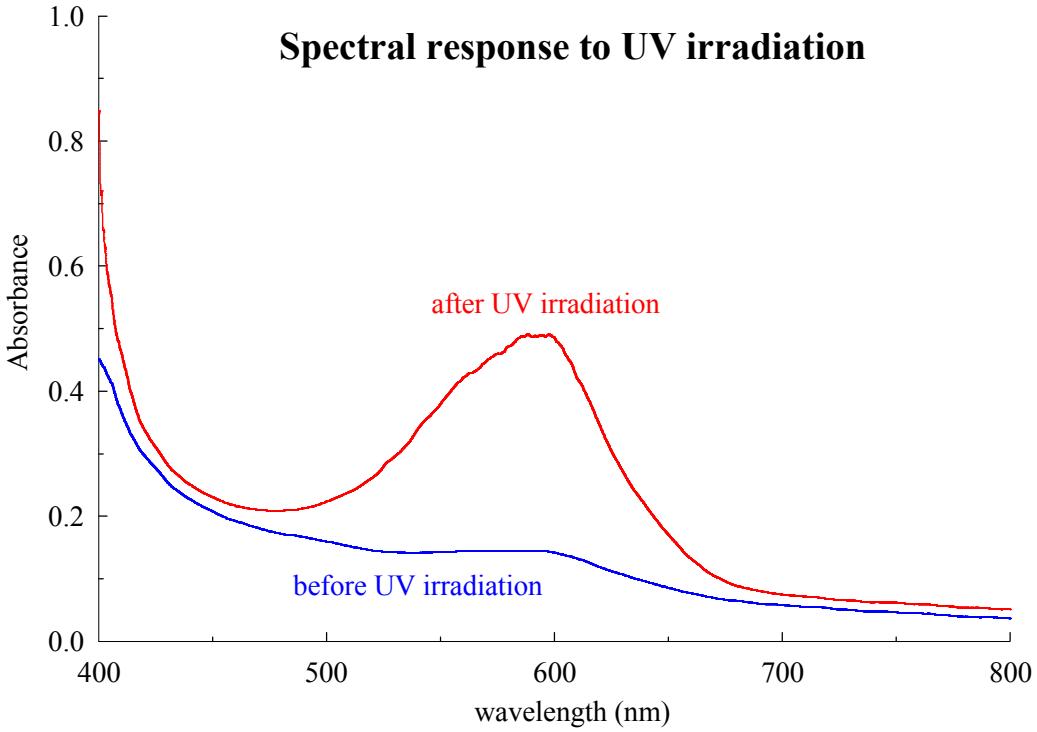
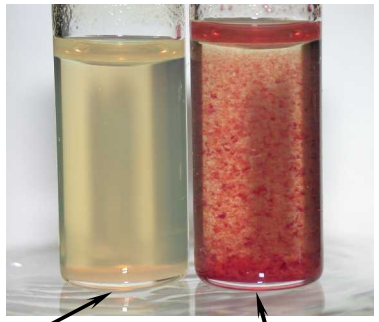


Photo-induced aggregation



before UV
particles well dispersed with SP molecules in closed, non-polar form

after UV
particles aggregate and sediment with SP molecules in more polar, open form

- aggregation induced in < 1min by λ=366 nm light from a hand-held lamp
- particles easily re-disperse upon shaking after visible light irradiation (λ>420 nm) or heating

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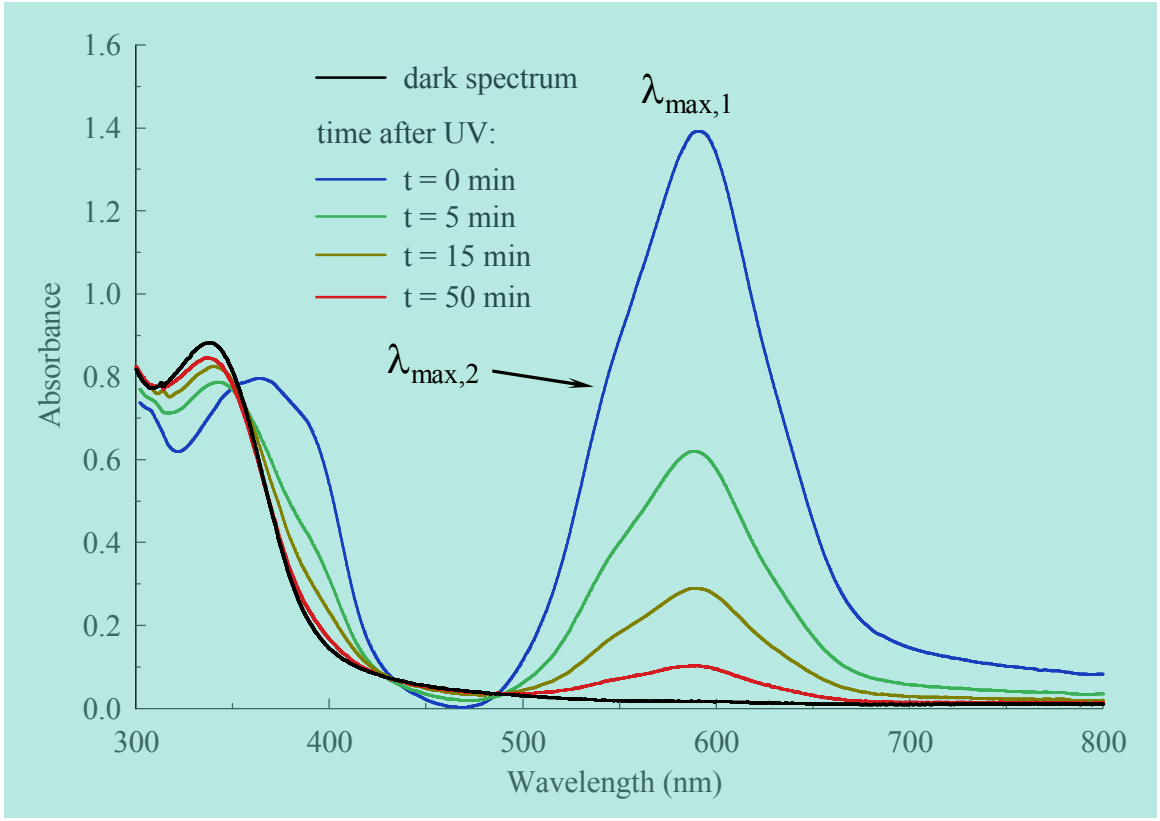
UV-Vis Characterization

Spectra in Tetrahydrofuran Solvent



Spectrum of 20% SP / 80% MMA polymer attached to 1 μm SiO_2 colloids at concentration of 0.079% wt/wt in THF

- $\lambda_{max,1}$ = 589 nm due to ME
- $\lambda_{max,2} \approx$ 550 nm due to ME-SP aggregates
- identical spectra obtained for attached and free polymers
- SP monomer shows only $\lambda_{max,1}$ = 587 nm due ME
- SP micro-environment within SP/MMA polymers favors ME-SP aggregation

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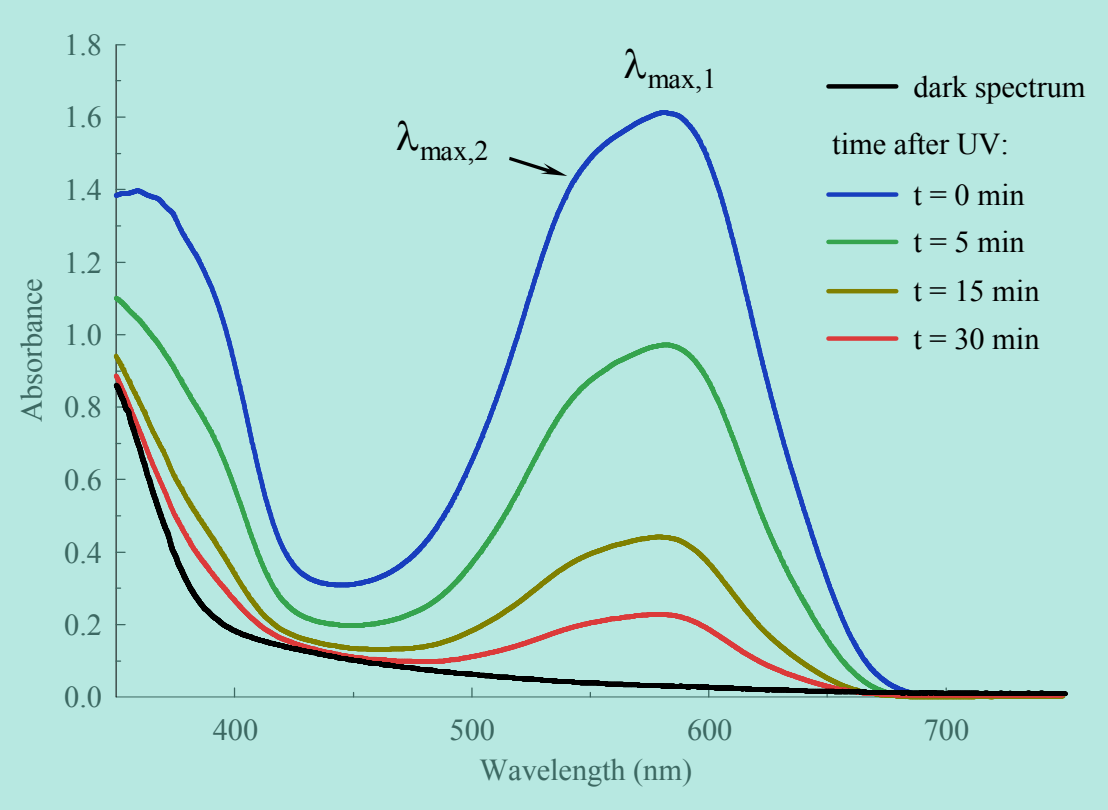
UV-Vis Characterization

Spectra in Toluene Solvent



Spectrum of 20% SP / 80% MMA polymer attached to 1 μm SiO_2 colloids at concentration of 0.20% wt/wt in toluene

- $\lambda_{max,1}$ = 581 nm due to ME
- $\lambda_{max,2}$ ≈ 545 nm due to ME-SP aggregates
- $\lambda_{max,1}$ diminished relative to $\lambda_{max,2}$ in toluene compared to THF
- This points to better ME solvation by THF vs. toluene
- $\lambda_{max,1}$ and $\lambda_{max,2}$ blue shifted in toluene compared to THF
- This points to more polar ME micro-environment in toluene vs. THF

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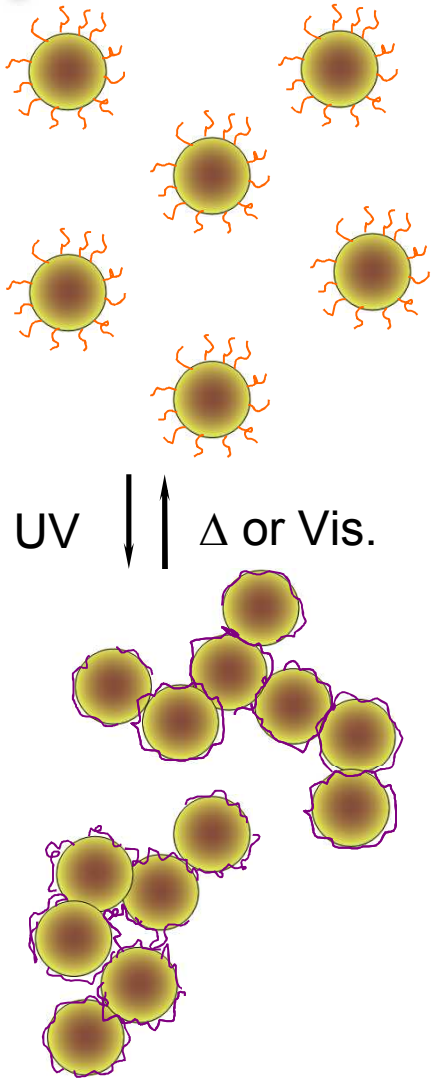
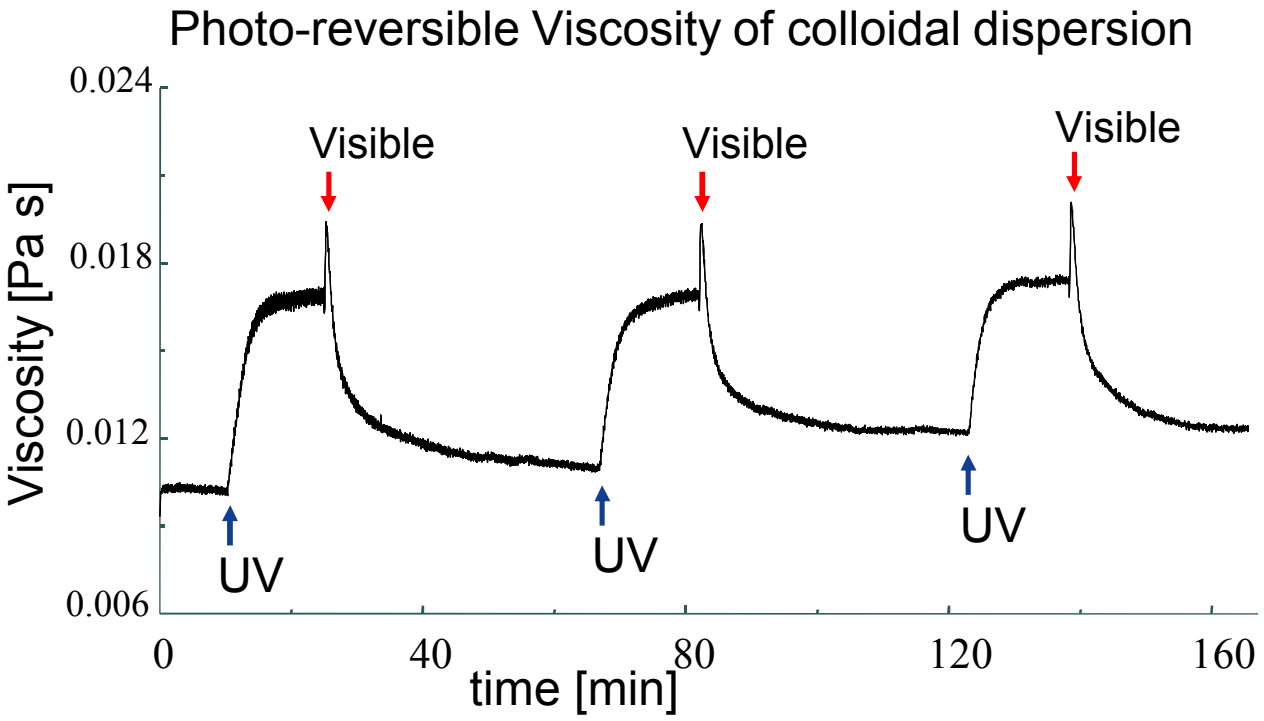
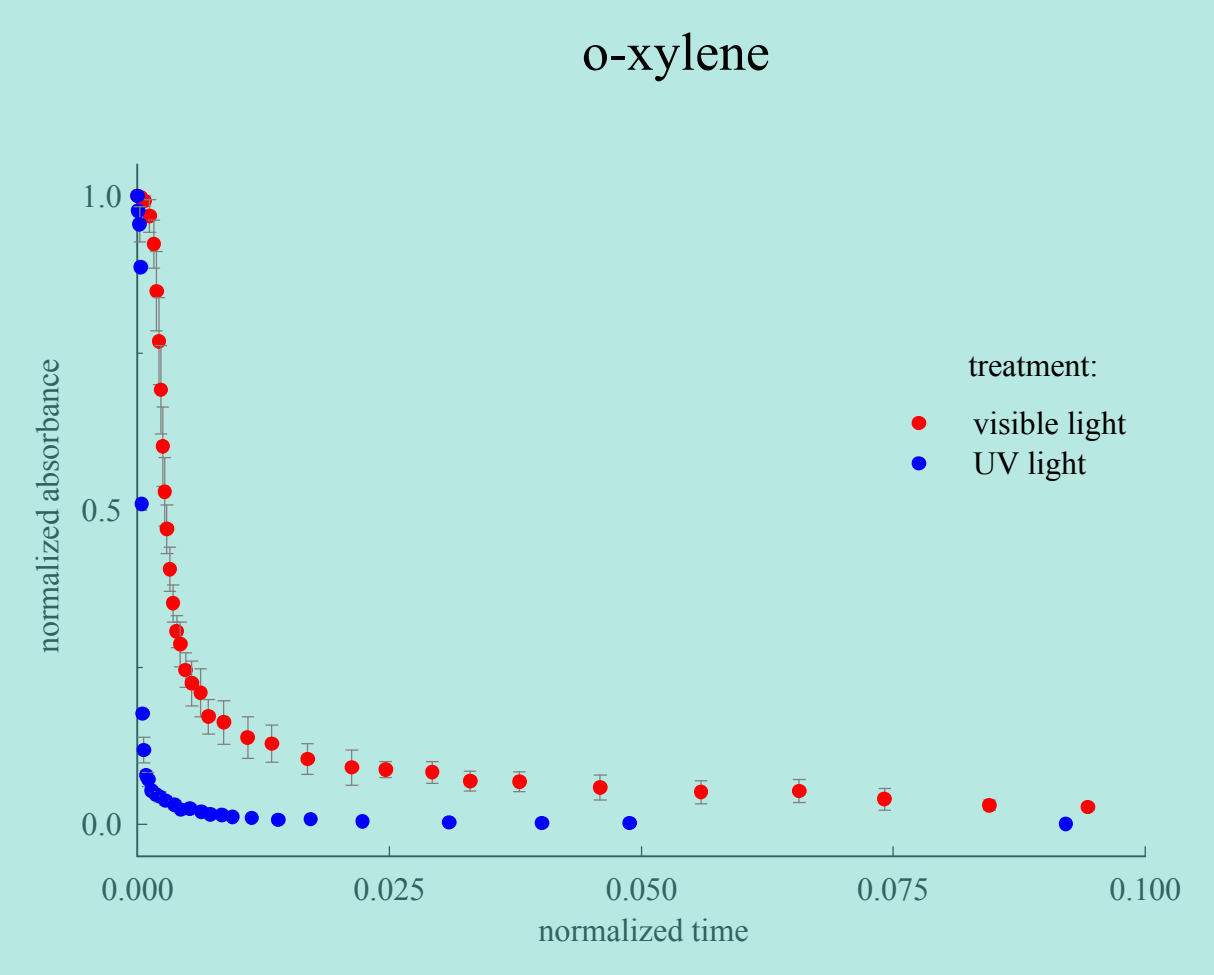
Photo-control of viscosity in colloidal systems





1 μm SP/MMA modified colloids (20% SP) in toluene at ~30 vol.%

Sedimentation in different solvents



- Surface Layers contain 20 mol% spirobenzopyran molecules in PMMA shell.
- Time axis normalized by theoretical sedimentation time (Stokes equation)

$$t = \frac{18\eta_L H}{a^2\left(D_p - D_L\right)g}$$

- Decrease in solvent polarity causes stronger aggregation
- UV irradiation precipitates stronger aggregation in in border-line solvents

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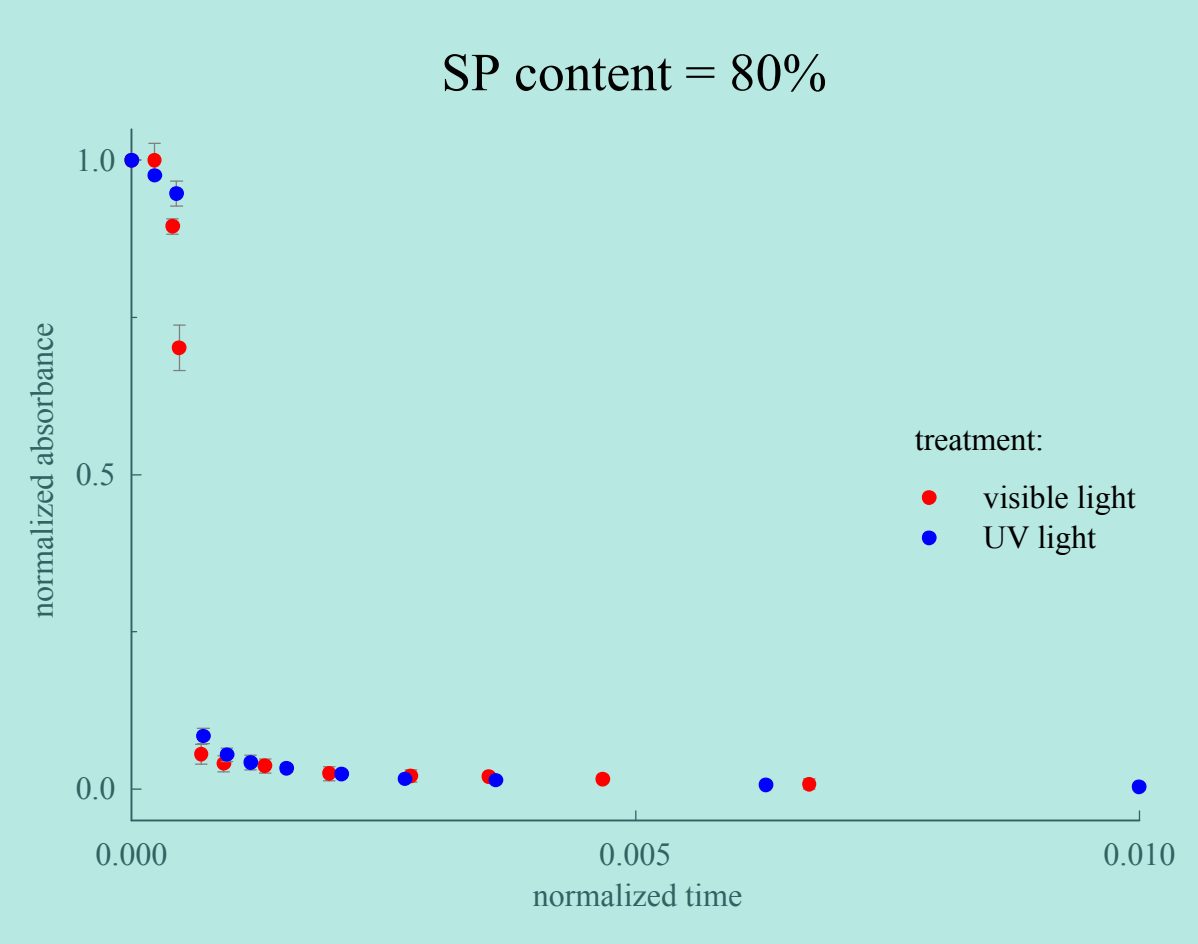
Sedimentation with different spiropyran content particles



- Sedimentation studied in toluene solvent for each SP concentration
- Increase in SP content causes stronger aggregation
- UV irradiation precipitates stronger aggregation in in border-line solvents
- Photo-aggregation is sensitive to solvent and %SP; polymer solubility factors can be adjusted for different systems.

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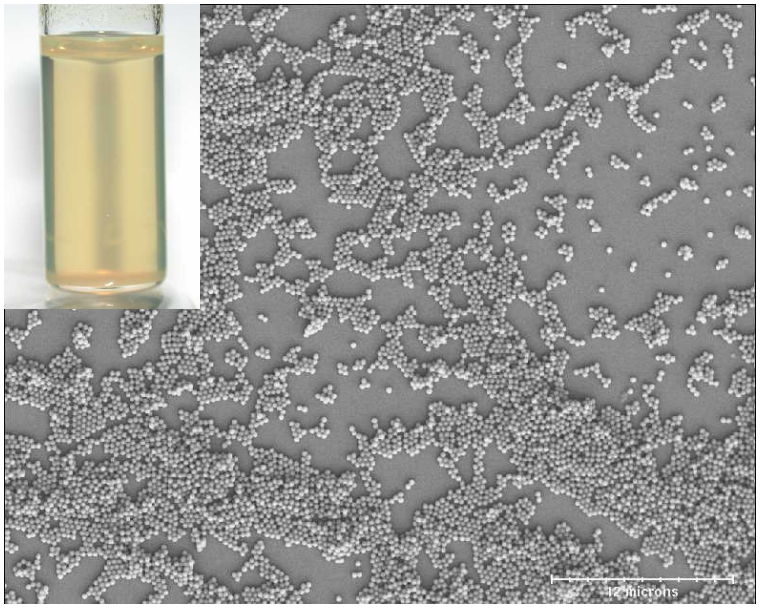
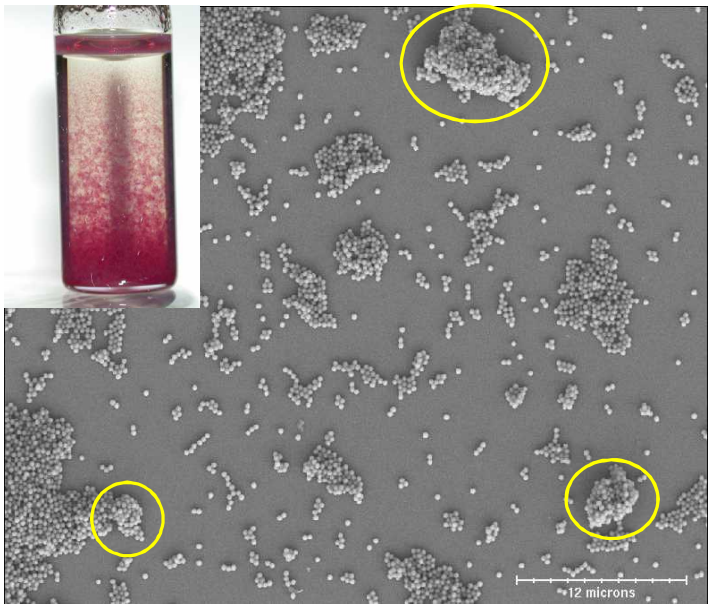
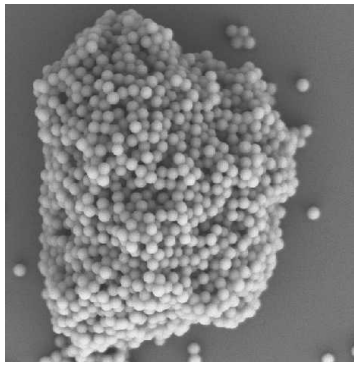
Sediment Comparison via SEM and Confocal Microscopy

No UV exposure

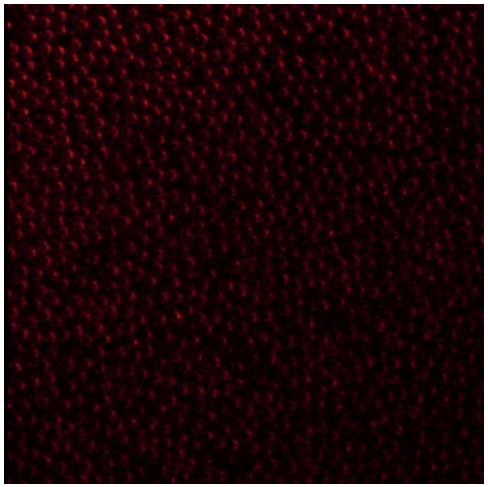


After 1 min UV irradiation (λ=366 nm)

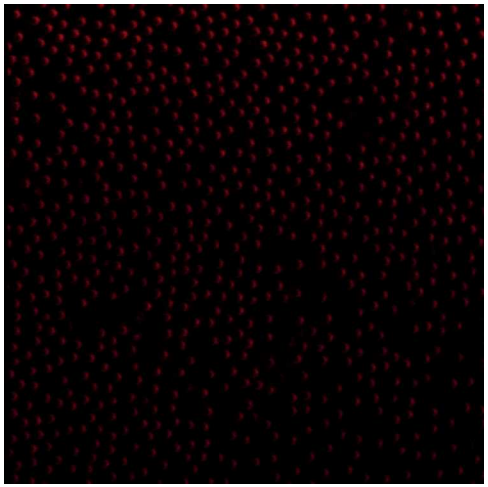




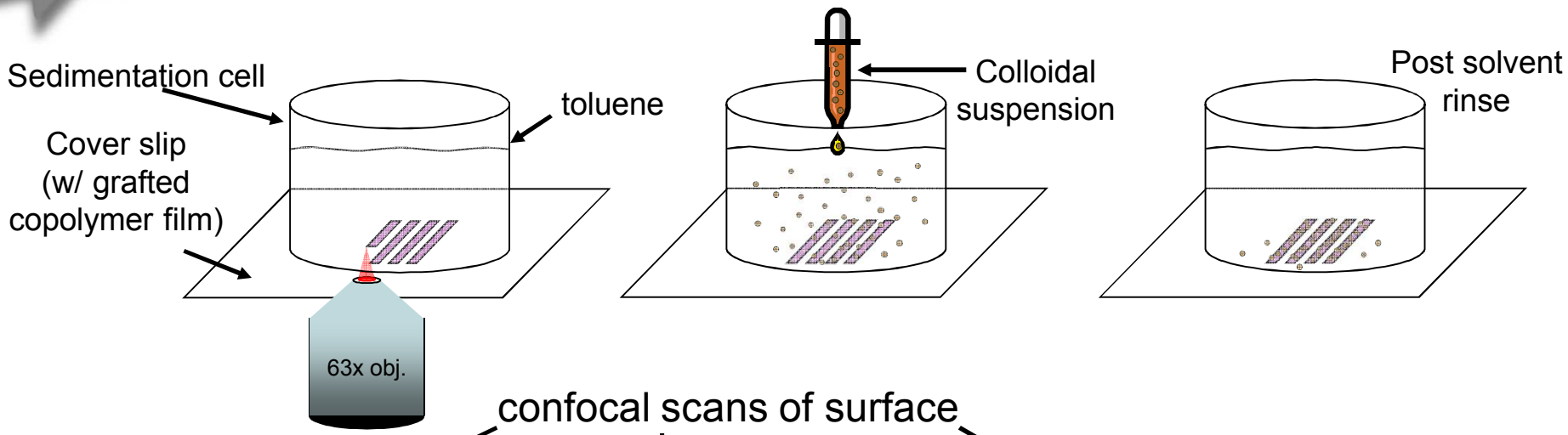
- particles form well spread mono- and multi-layers
- predominant hexagonally packed domains



- larger aggregates present in addition to mono- and multi-layers
- aggregates characterized by random particle packing

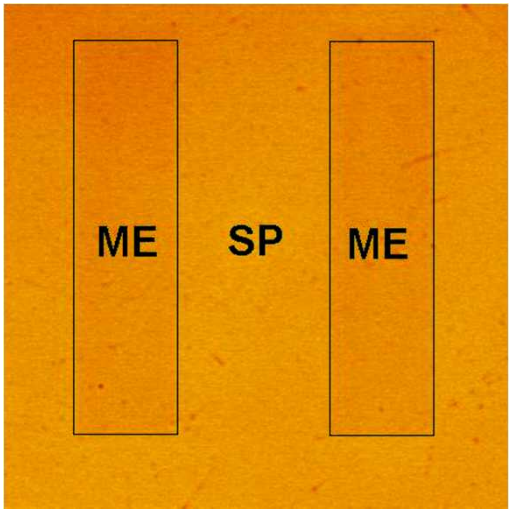


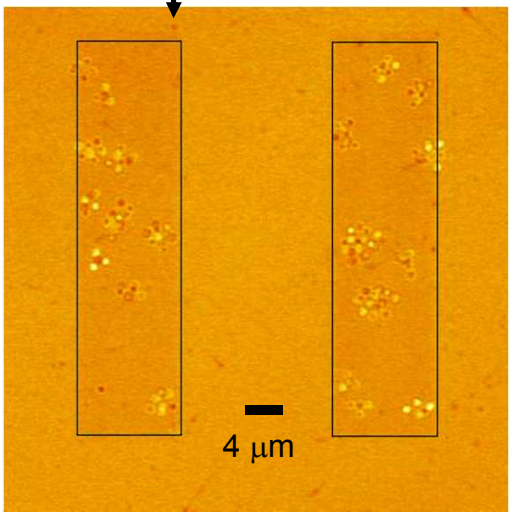
2D Photo-defined Colloidal Adsorption

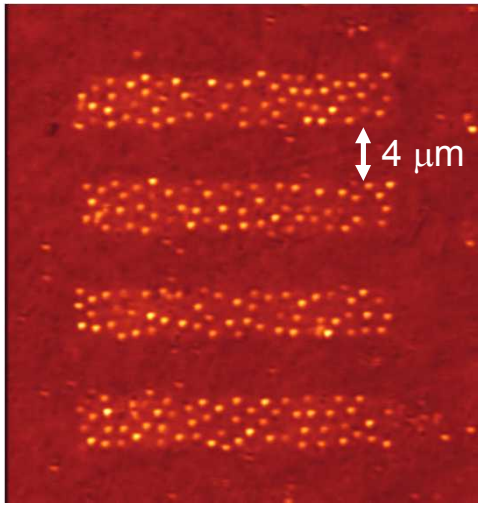


confocal scans of surface

2D patterning example







exp. start, t = 0

clusters, t = 660 s

M. Piech, M. C. George, N. S. Bell and P. V. Braun:, *Langmuir*, **22**, 1379-1382 (2006)

Photo-Actuated Deposition in Toluene

SPMMA-*co*-MMA coated glass slide in toluene

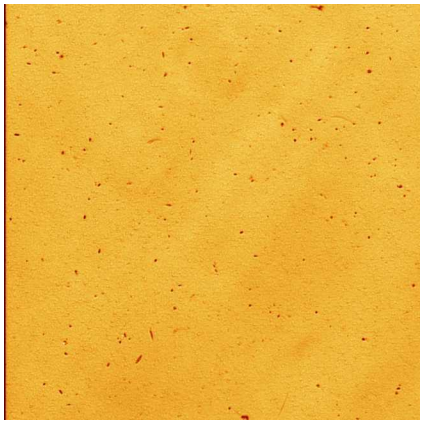
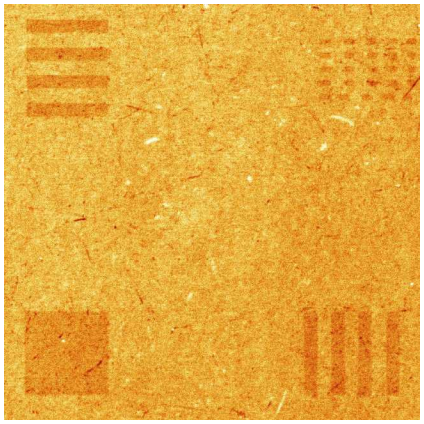
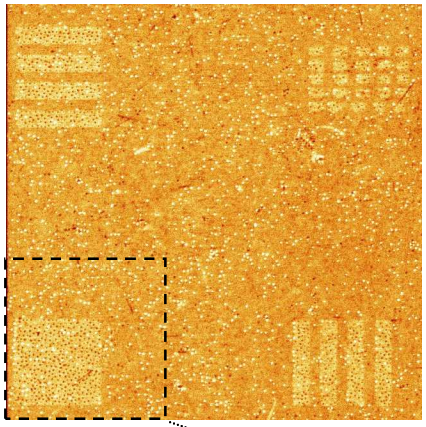


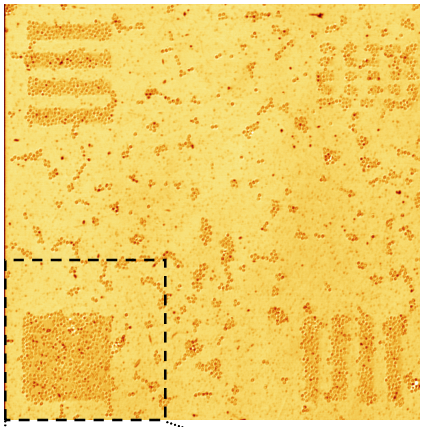
Photo-isomerizing SP in dark regions

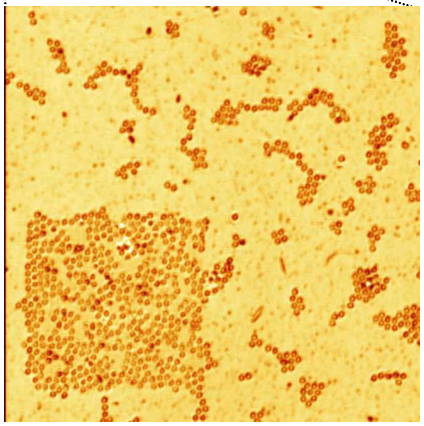


After settling dilute colloidal suspension

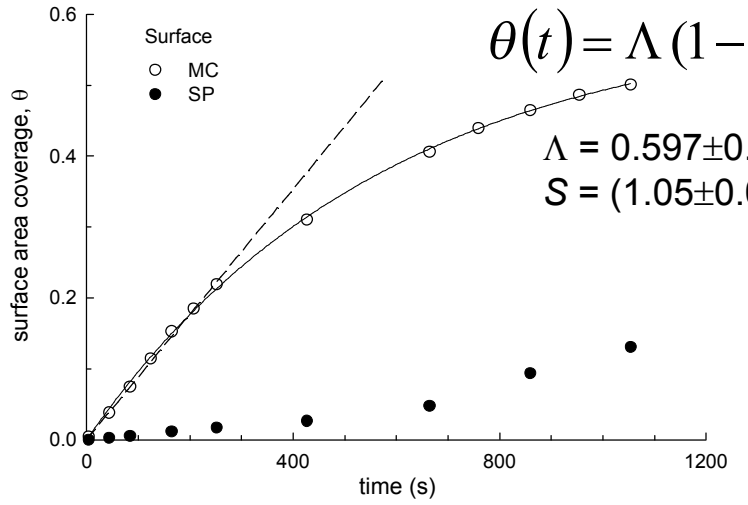


After drying





particle adsorption kinetics



$$\theta(t) = \Lambda\left(1 - e^{-S\,t/\Lambda}\right)$$

Λ = 0.597±0.007,
S = (1.05±0.02)×10^{-3} s^{-1}

The deposition behavior falls between random sequential adsorption and ballistic deposition models, with a time constant near the sedimentation rate.

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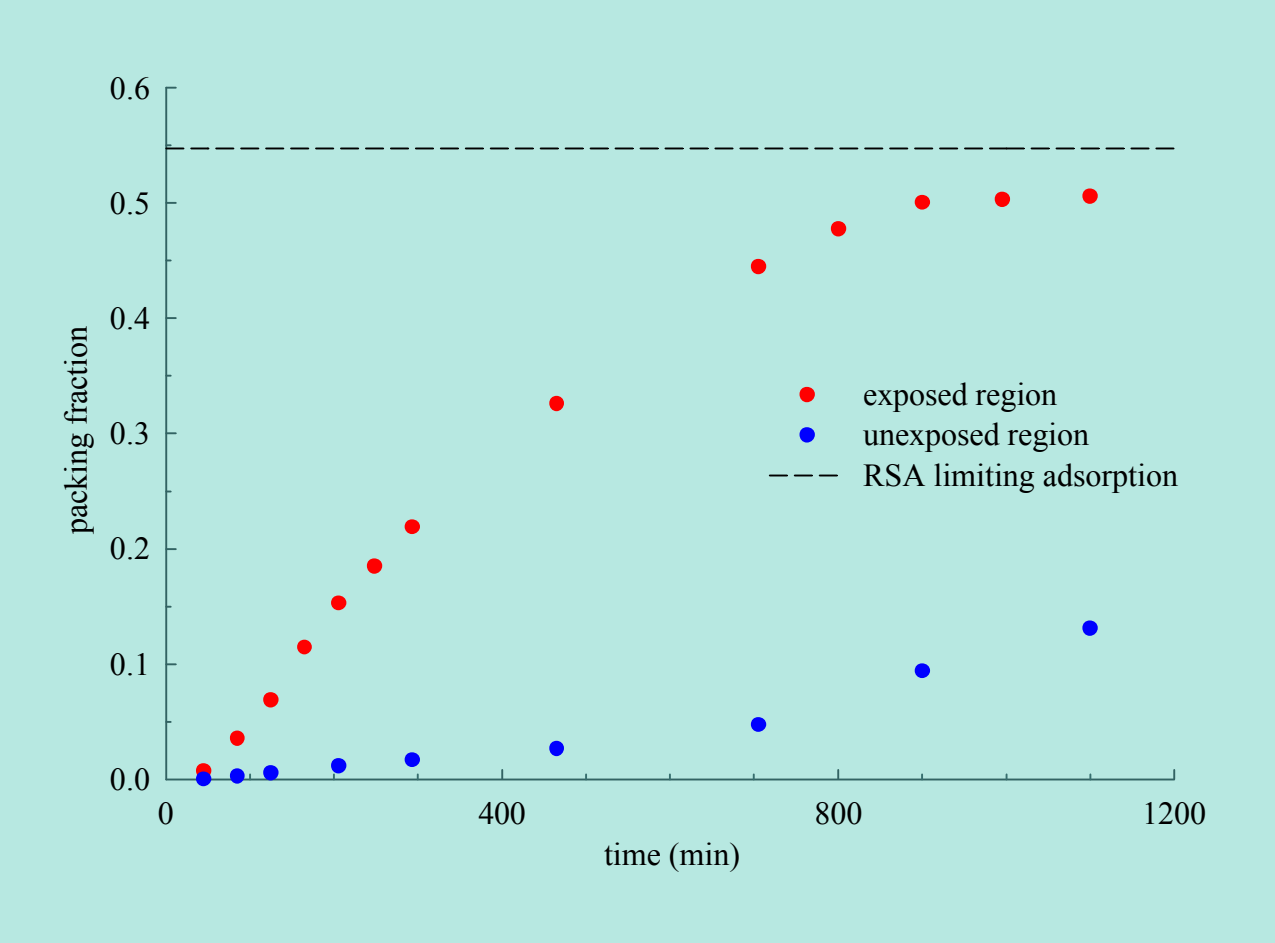
Kinetics of photo-actuated deposition



- Initially particles adsorb 14× more selectively to the exposed regions containing ME

- Following rinsing and drying particle density is 10× higher in exposed regions

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3D laser gel-writing in colloidal sediments

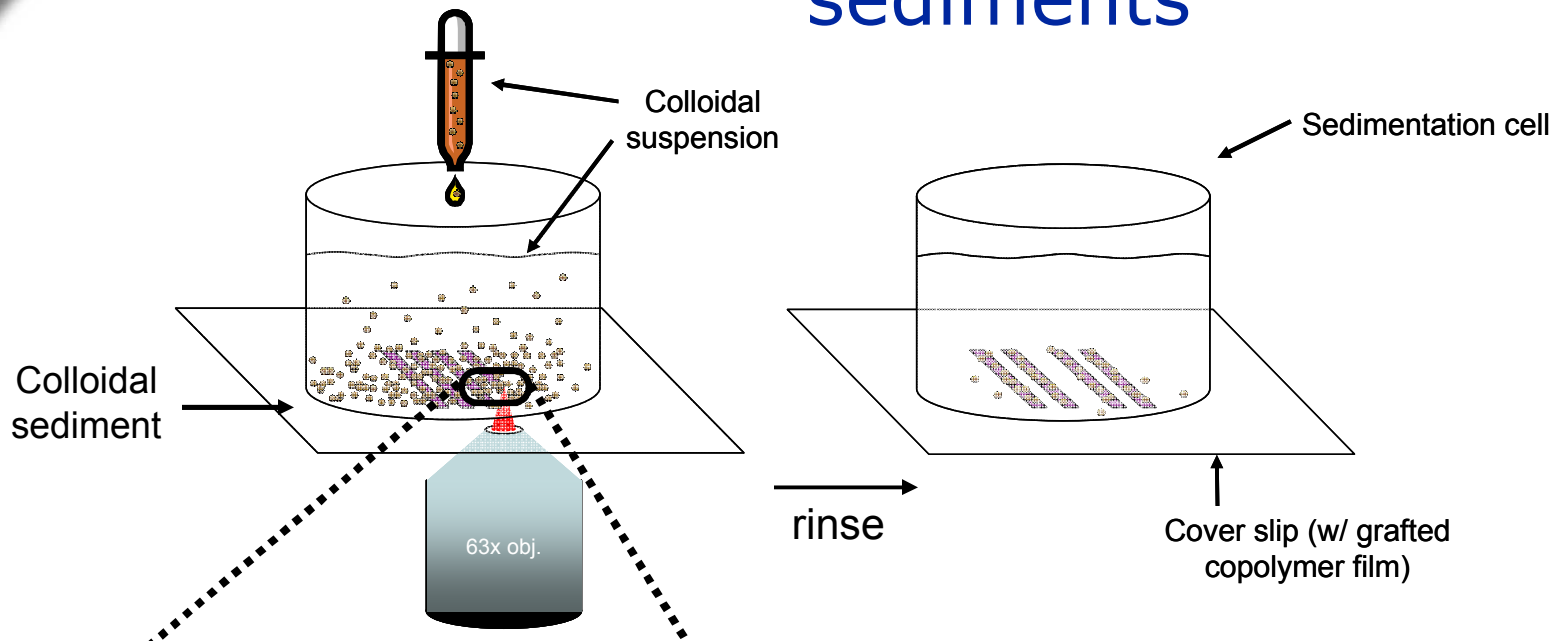
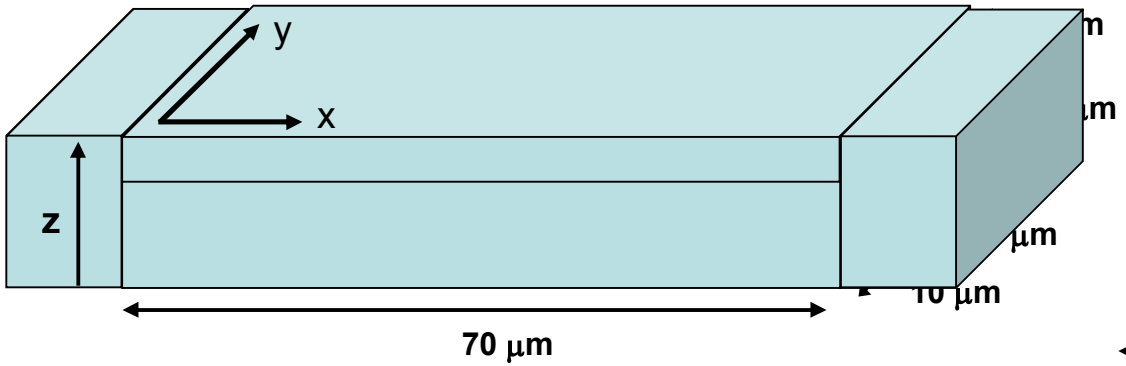
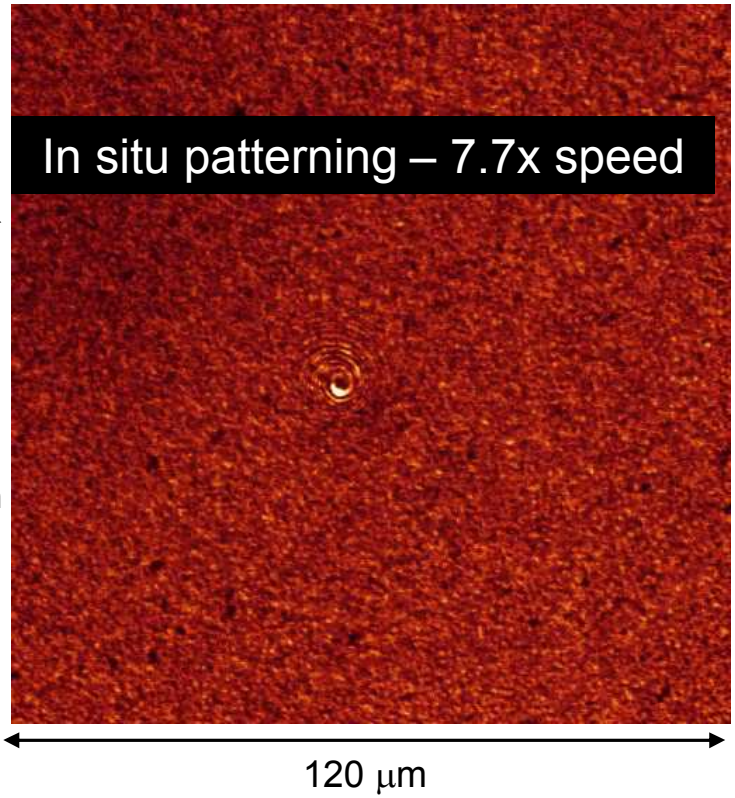


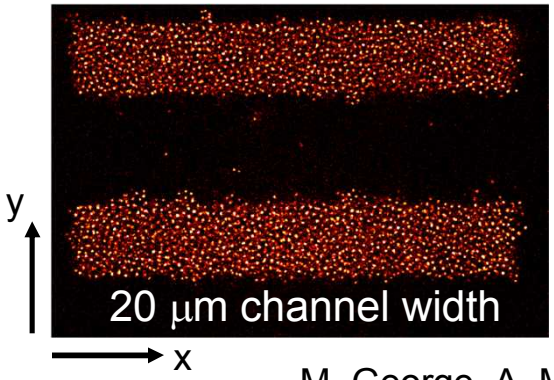
Photo-Patterned Colloidal Gel Micro-channels

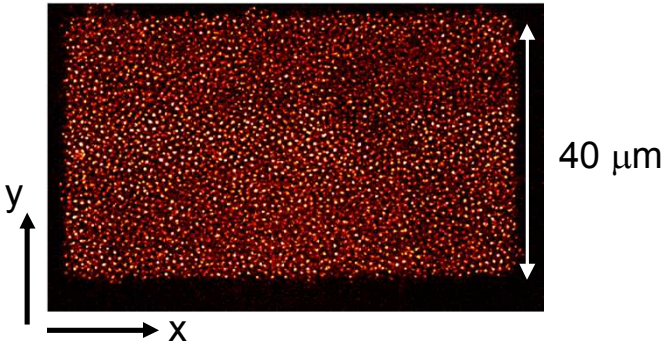
Processing Steps

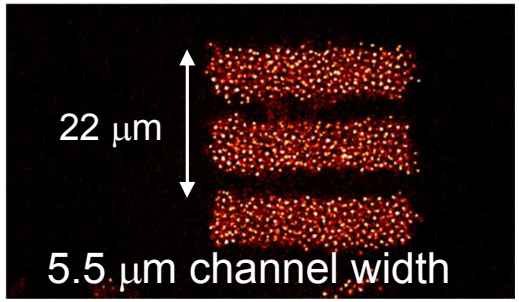
- Introduce colloids, form sediment
- Write walls (via 2PA)
- Write channel top
- Rinse out un-gelled colloids







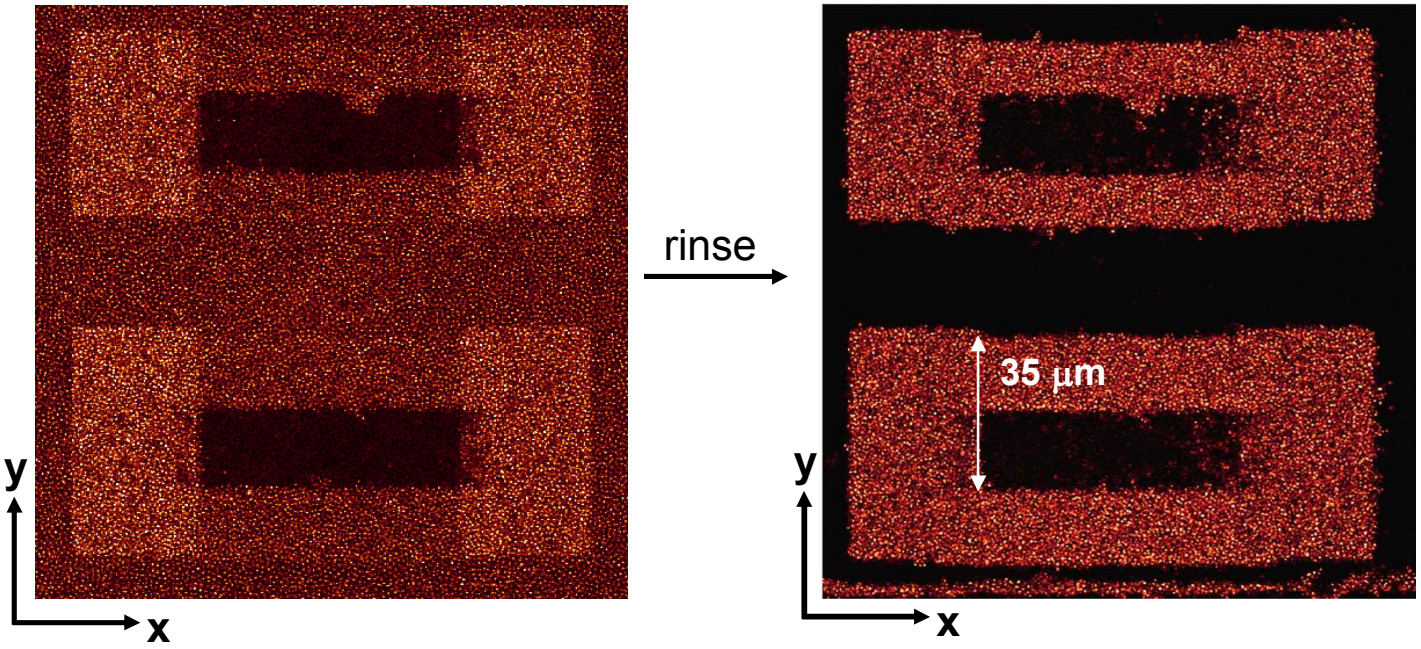




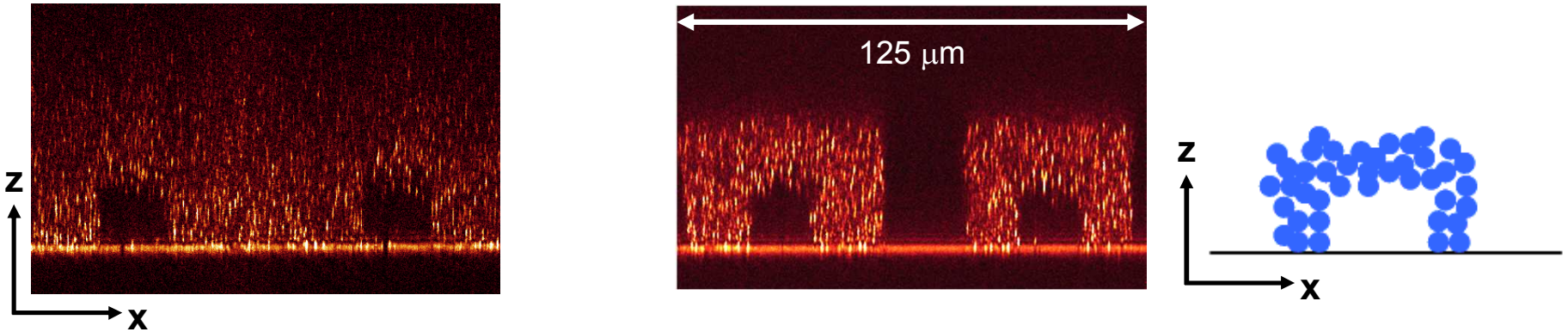
M. George, A. Mohraz, M. Piech, N. Bell, J. Lewis, and P. Braun (unpublished work)

Photo-Patterned Colloidal Gel Micro-cavity

confocal xy reflectance sections



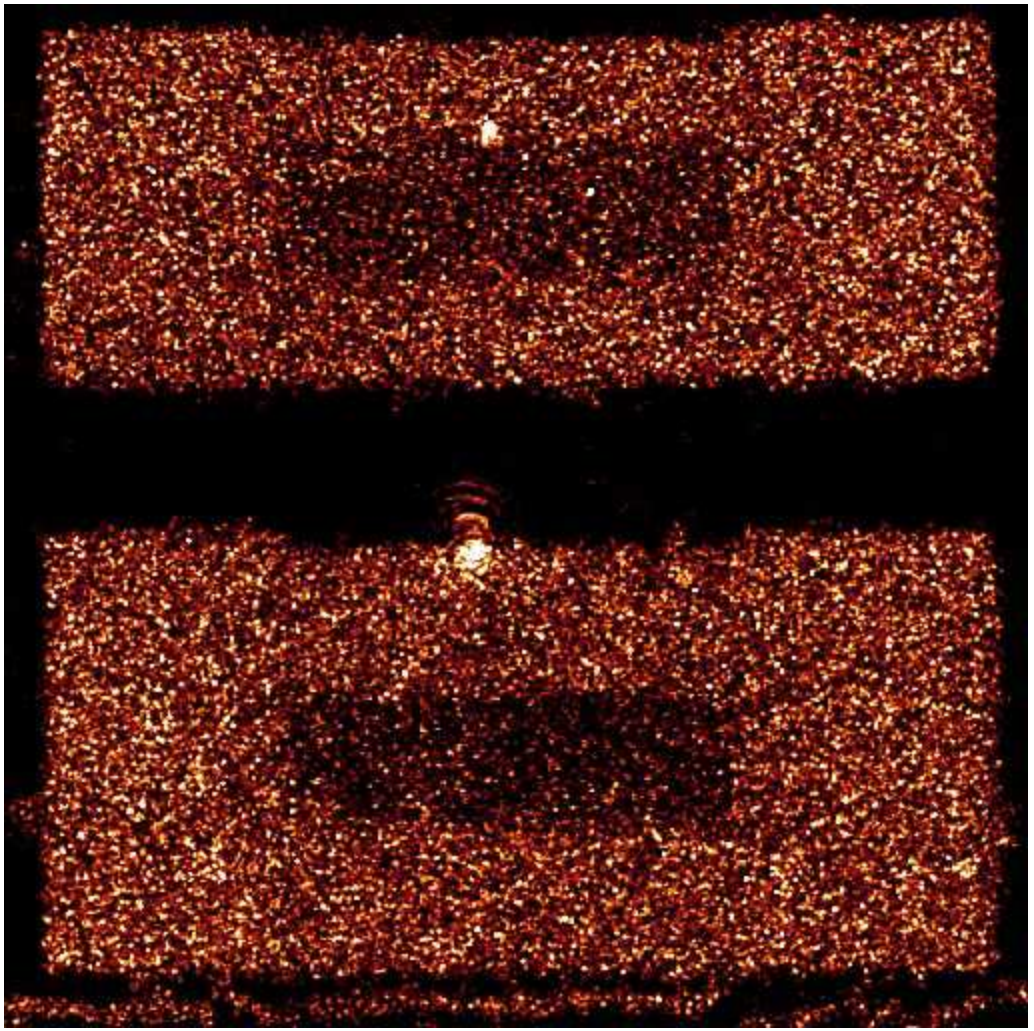
confocal reflectance cross-sections (xz)



Trapped Colloidal Particles

<u>Processing Steps</u>

- form a colloidal sediment
- Write walls (via 2PA)
- Write channel top
- Rinse out un-gelled colloids
- Observe Brownian motion of contained colloids

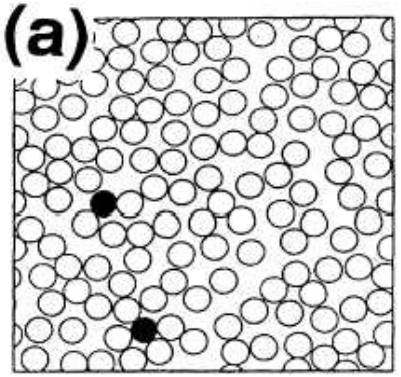


Conclusions

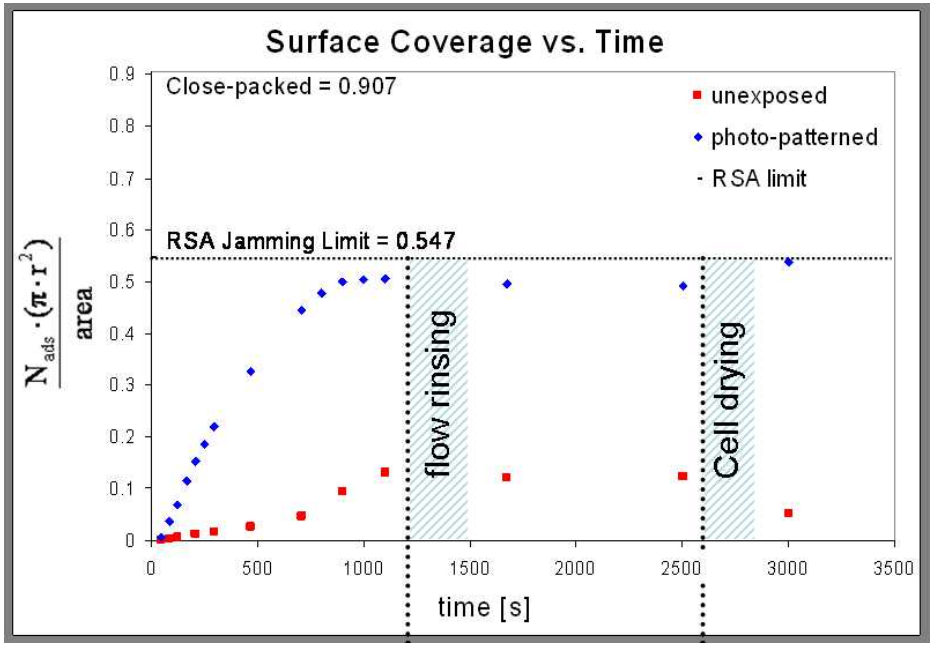
1. Photochromic molecules show the capability to influence macroscopic phenomena
2. Changes in optical and surface energy of modified surfaces have been detected by UV/VIS and contact angle measurements, respectively.
3. Modeling of Surface Energy Parameters gives greater insight into the mechanisms of surface energy control and allows screening of systems to generate desired photowetting effects.
4. A colloidal system has demonstrated reversible aggregation and dispersion phenomena.
5. Polymer layers show greater cycle lifetime than monolayers.
6. Chromophoric molecules act as dopants in modifying polymer behavior and show promise for more application.

Adsorption kinetics

RSA of Disks $\Rightarrow$ Jamming coverage
0.547 +/- 0.0002



Evans, J.W. *Rev. Mod. Phys.*, **65**, No. 4, (1993)



same

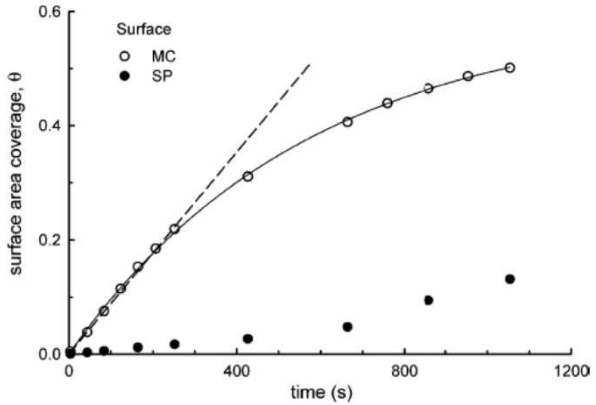
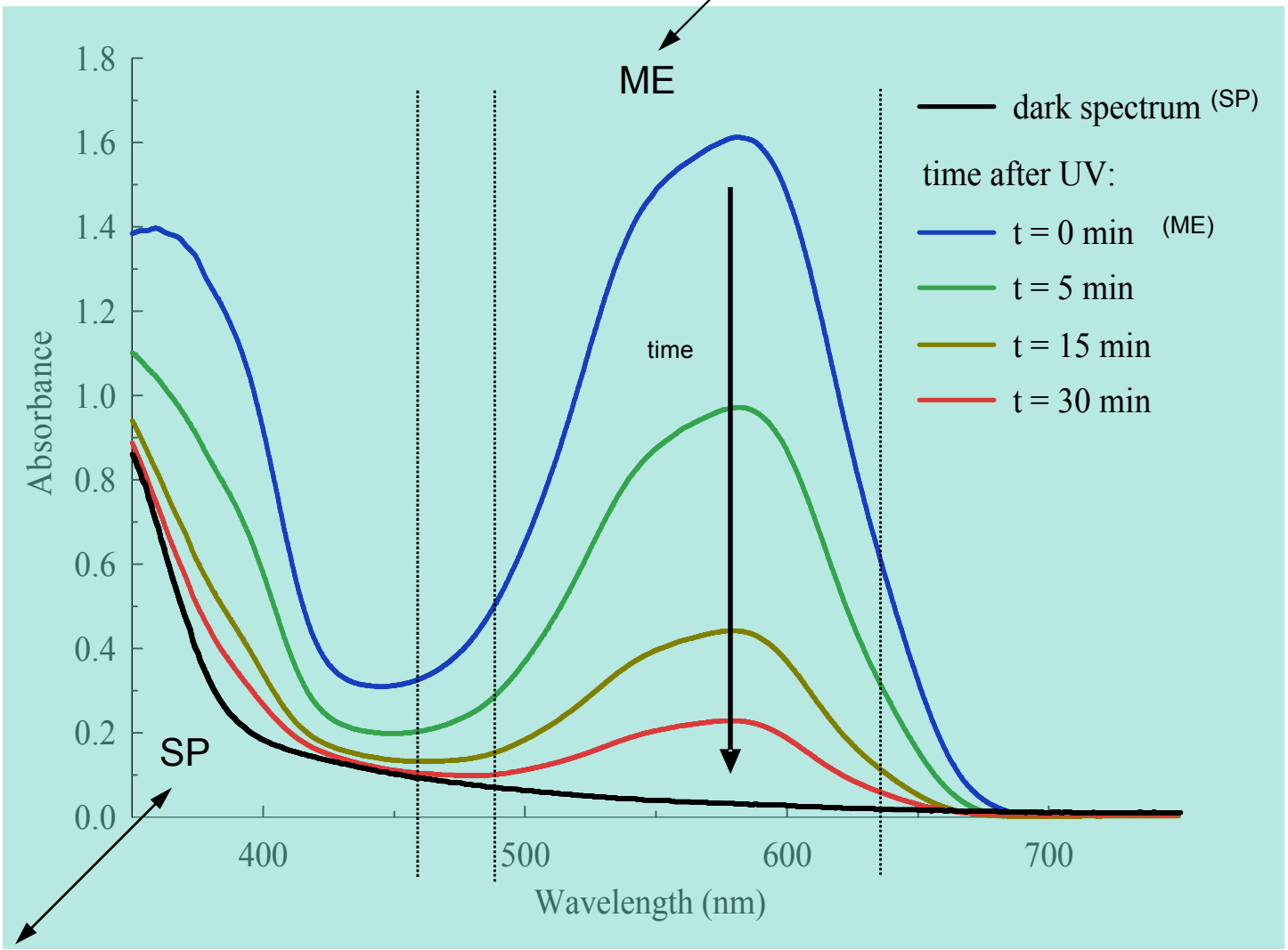
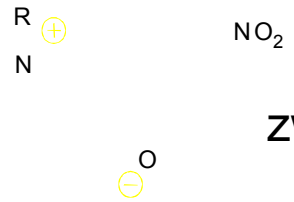


Figure 3. Plot of fractional surface coverage as a function of time for colloids depositing onto the photoswitched (MC) and unexposed (SP) regions of the substrate modified with an SP-*co*-MMA

M. Piech, M. C. George, N. S. Bell and P. V. Braun:, *Langmuir*, **22**, 1379-1382 (2006).

Absorbance spectrum of a complex fluid, containing SP/MMA copolymer

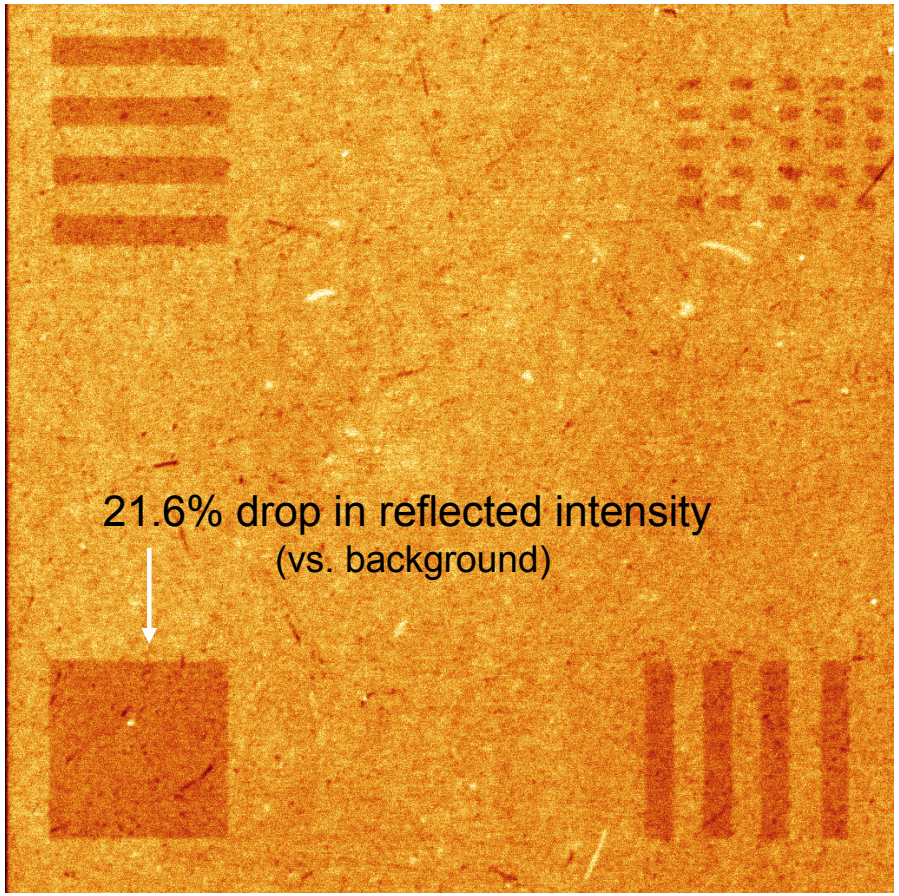




zwitterionic form

closed form

Confocal Reflectance Images (458nm)



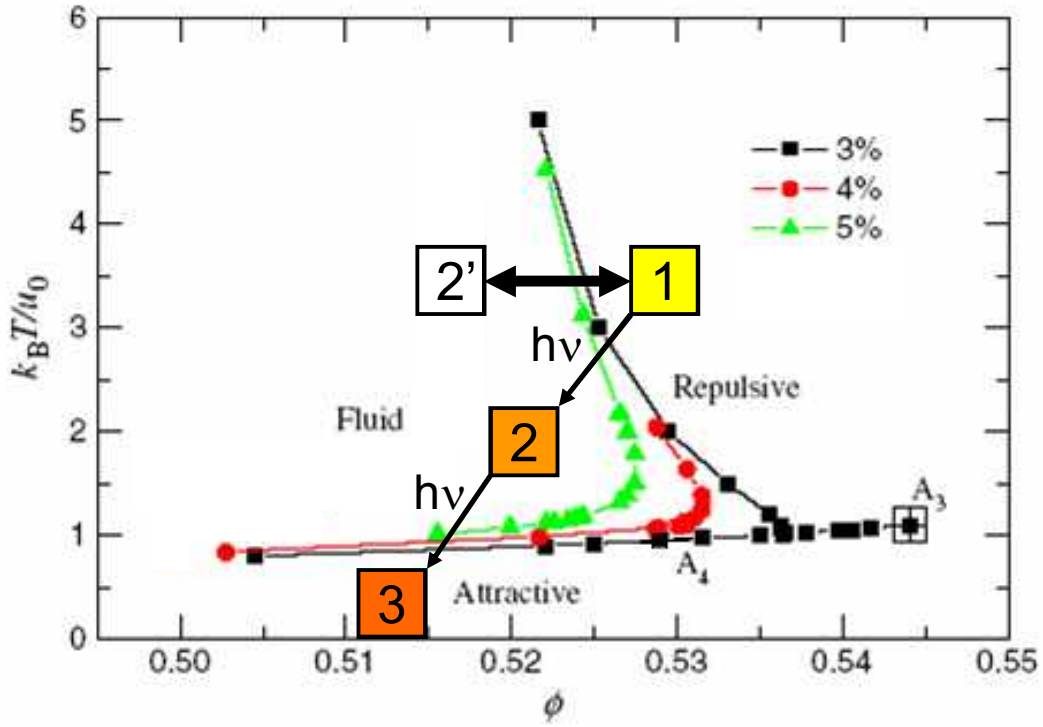
Initial image taken after 2-photon exposure

time

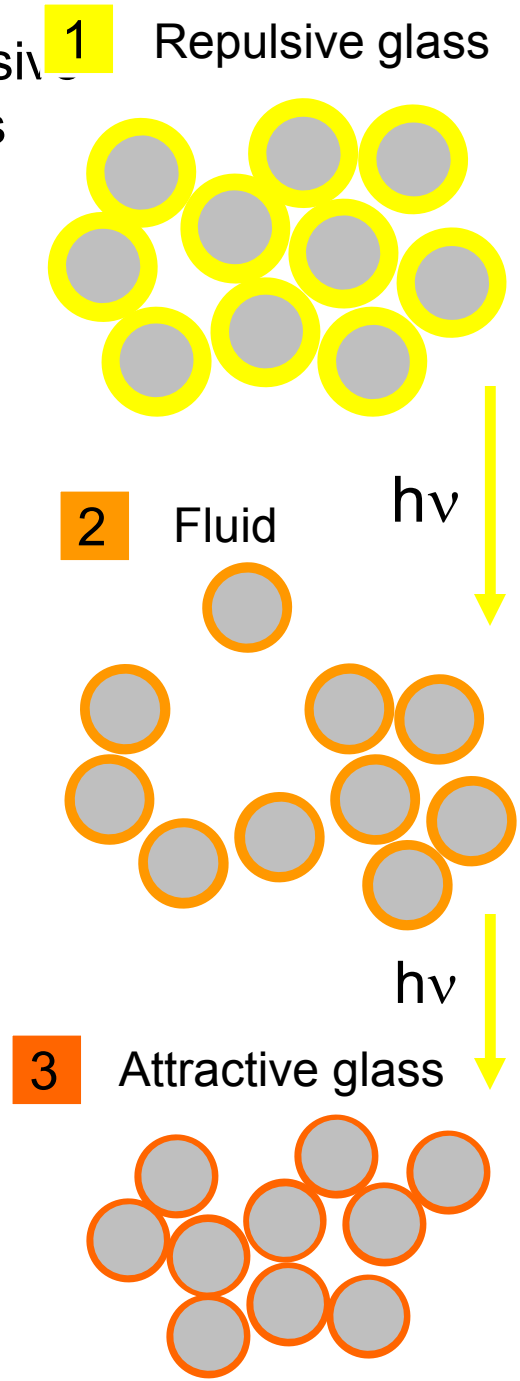
After several minutes (& additional exposure)

- Initial drop in reflected intensity… followed by brightening with time !?!?!

Phase diagram for colloids with hard core repulsive and short range attractive square well potentials



Solution: SP-co-MMA-block-PS (colloids do not aggregate upon UV irradiation)



Luca Cipelletti and Laurence Ramos, "Slow dynamics in glassy soft matter", *J. Phys.: Condens. Matter* **17** (2005) R253-R285

Synthesis and Light-induced Assembly of Photo-responsive Colloids

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