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Smart worm-like micelles responsive to CO₂/N₂ and light dual stimuli

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Abstract. CO₂/N₂ and light dual stimuli-responsive worm-like micelles (WLMs) were obtained by addition of a relatively small amount of a switchable surfactant, 4-butyl-4'-(4-*N,N*-dimethylhexyloxy-amine) azobenzene bicarbonate (AZO-B6), sensitive to the same triggers into a binary aqueous solution of cetyltrimethyl ammonium bromide (CTAB) and sodium salicylate (NaSal).

Worm-like micelles (WLMs) which can be converted to non-fluid hydrogels at high surfactant concentration have received considerable attention over the past several decades because of their peculiar micellar structures and unique rheological response.¹ WLMs have a range of potential applications, including rheology control, heat transfer, drag reduction, in enhanced oil recovery and personal care products.² In recent years, particular interest has been focused on stimuli-responsive or “smart” WLMs,³ that respond reversibly to external environmental stimuli such as pH,^{4,5} CO₂ addition,^{6,7} UV–vis. irradiation,^{8–10} temperature^{11,12} and redox reactions.¹³

Among these studies most WLM systems were designed to respond to a single stimulus, and multi-stimulated systems are much less common.³ It has been recognized that the combination of multiple stimuli can widen the controllable range or improve the degree of precision for a given system. For example, Hu *et al.* reported concentration- and temperature-induced dual-responsive WLMs based on an ionic liquid-type surfactant.¹⁴ Wang *et al.* reported pH and metal ion dual-responsive WLMs using an amphiphilic short peptide.¹⁵ Yan *et al.* reported thermal and pH dual-stimulated WLMs in an aqueous solution of *N*-cetyl-*N*-methylpyrrolidinium

bromide.¹⁶

Although pH has been widely accepted as a cheap, non-toxic and easily employed trigger for WLMs, it is possible that the added chemicals may result in contamination of the system and accumulation of the by-product.³ Simple and environmentally benign triggers are therefore more attractive and highly desirable. As such triggers, CO₂ has been incorporated with surfactants and polymers to constitute switchable or stimuli-responsive chemicals since it can be easily removed from the system on demand, *e.g.* from unstable bicarbonate by bubbling N₂ or air,¹⁷ and light has also been utilized as its wavelength, polarization directions and intensity can be easily controlled.^{6,7,18} By using these two triggers the contamination and by-product accumulation in systems can be avoided.

In this study, we report smart WLM systems which are responsive to both CO₂/N₂ and light irradiation when they are employed individually. By adding a dual-switchable surfactant, 4-butyl-4'-(4-*N,N*-dimethylhexyloxy-amine) azobenzene bicarbonate (AZO-B6) into a binary aqueous solution of cetyltrimethylammonium bromide (CTAB) and sodium salicylate (NaSal), the ternary solution can be gelled or form viscoelastic WLMs with high viscosity at room temperature, which can be converted to a fluid solution of low viscosity by either bubbling N₂ or irradiating with UV light. The systems can thus be reversibly cycled between gel-like and fluid in response to the two triggers.

Results and Discussion

The CO₂/N₂ and UV dual switchable surfactant AZO-B6 has a molecular structure shown in Scheme 1 and a ¹H NMR spectrum shown in Figure S1. The molecule contains both a tertiary amine group and an azobenzene group, which are CO₂/N₂-sensitive and photo-active, respectively. Bubbling N₂ or air into an aqueous solution of AZO-B6, the amphiphilic ammonium bicarbonate (surface-active form) is converted to an uncharged tertiary amine (surface-inactive), whereas bubbling CO₂ enables re-protonation of the tertiary amine

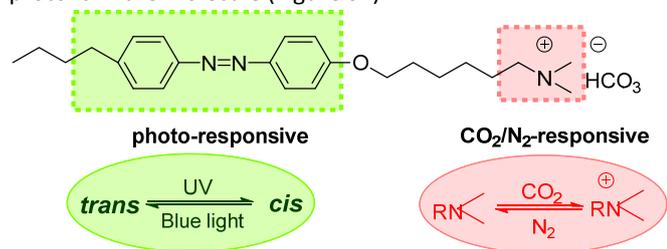
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turning it back to bicarbonate. This inter-conversion can be monitored by measuring the conductivity of the aqueous solution (Figure S2). In addition, the azobenzene group can be easily photo-isomerized between *trans* (cmc 1.44×10^{-4} M, γ_{cmc} 32.9 mN/m) and *cis* (cmc 2.34×10^{-3} M, γ_{cmc} 38.5 mN/m, Figure S3) states upon blue light/UV irradiation, respectively, as confirmed by the different ^1H NMR peaks of the aromatic protons in the molecule (Figure S4).



Scheme 1. Chemical structure of AZO-B6 surfactant and its responses to CO_2/N_2 and UV/blue light irradiation.

AZO-B6 cannot form gel-like WLMs alone in water at 50 mM, nor in the presence of sodium salicylate (NaSal) at up to 50 mM (Figure S5). It is well known that CTAB can self-assemble in water to form charged globular micelles at concentrations above the critical micelle concentration (cmc, 0.9 mM), and these globular micelles can continuously grow to long and flexible rod-like micelles upon addition of organic or inorganic salts, such as NaSal.^{19,20}

In an initial experiment, a binary CTAB-NaSal aqueous solution (50 mM each) was found to display Newtonian fluid behaviour at low shear rates (zero-shear viscosity $\eta_0 = 58.6$ Pa s) and shear-thinning behaviour beyond a critical shear rate (0.1 s^{-1}) at room temperature (Figure S6). This phenomenon is generally regarded as evidence of the presence of viscoelastic WLMs.¹

When AZO-B6 in the cationic form was added to the CTAB-NaSal binary solution at concentrations between 5 and 30 mM, the zero-shear viscosity of the ternary solution increases with increasing concentration of AZO-B6, reaching 268 Pa s at 30 mM which is five orders of magnitude higher than the viscosity of water as shown in Figure 1a. This indicates that the addition of AZO-B6 does not destroy the WLMs but improves their entanglements in the ternary solutions. This was also evidenced by oscillatory rheology experiments (Figure 1b). By adding AZO-B6, the storage modulus (G'), which is lower than the loss modulus (G'') in the low-shear-frequency region, predominates at shear frequencies beyond a critical value (ω_c). The solution thus displays an evident viscous behaviour at low-shear frequency and a typical elastic response at high-shear frequencies. In other words, the solution is viscoelastic and its relaxation time τ_r ($\sim 1/\omega_c$) is finite, which typically accounts for the formation of long worms.²¹ Within the range of concentrations of AZO-B6 (5–30 mM), the ternary solution still follows the Maxwell model with a single relaxation time, and G' and G'' fit the Cole–Cole model well in the low- to medium-frequency range. However, deviation from this model occurs at high frequencies because of the strong elastic behaviour (Figure 1c),²² suggesting the formation of WLMs.²³ Both the radius of the semi-circle and the relaxation time τ_r increase

markedly with AZO-B6 concentration. This further suggests that the addition of AZO-B6 increases the average length of the worms and promotes their entanglement.

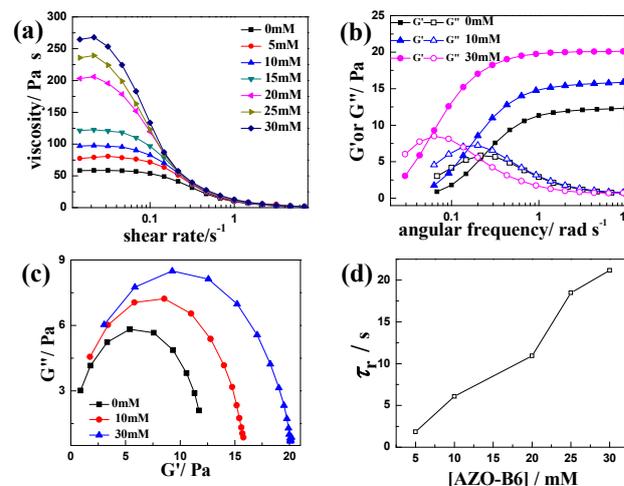


Figure 1. Rheological behaviour of CTAB-NaSal binary aqueous solution (50 mM each) upon addition of AZO-B6 at different concentrations at 25 °C. (a) Steady-state rheology, (b) dynamic rheology, (c) Cole–Cole plot and (d) relaxation time τ_r .

In both the binary and ternary solutions, the strong electrostatic repulsion between the positively charged head-groups of the cationic surfactants is greatly reduced by the insertion of NaSal molecules into the aggregates. The geometry of the aggregates is thus adjusted to be suitable for formation of WLMs. Since the *trans*-AZO-B6 molecules have a linear hydrophobic alkyl chain similar to CTAB molecules, when they are added into the CTAB-NaSal binary solution they tend to insert themselves into the aggregates in mixed micelles, equivalent to increasing the concentration of CTAB. In fact similar WLMs can be obtained by simply increasing the concentration of CTAB in the binary solution to 80 mM, as shown in Figure S7. The average length of the worms thus increases promoting the entanglement of long worms. This finding is consistent with the results of rheology. Such behaviour was also observed in the CTAB-NaSal-AZTMA (4-butylazobenzene-4'-(oxyethyl)-trimethylammonium bromide) system⁸ in which the addition of *trans*-AZTMA to a CTAB/NaSal aqueous solution causes an increase in η_0 from 60 to 100 Pa s.

It was found that the WLMs in CTAB-NaSal-AZO-B6 ternary systems can be reversibly converted between gel-like and fluid states triggered by either CO_2/N_2 or light irradiation, *i.e.* the WLMs are “smart”. When 30 mM of cationic *trans*-AZO-B6 was added into the CTAB-NaSal (50 mM–50 mM) binary solution, the viscosity of the ternary solution dramatically increased ($\eta_0 = 265$ Pa s) such that the system was gelled as shown in Figure 2a (1). However, after bubbling N_2 at 65 °C for 2 h, the gel-like system was changed to be highly flowable with $\eta_0 = 0.02$ Pa s, which is much lower than that of CTAB/NaSal binary system ($\eta_0 = 58$ Pa s) and close to that of water as shown in Figure 2a (2). On the other hand the fluid solution can be turned back to gel-like by streaming CO_2 at ambient temperature for 30 min, as shown in Figure 2a (3). This

conversion between gel-like and fluid states can be reversibly cycled many times.

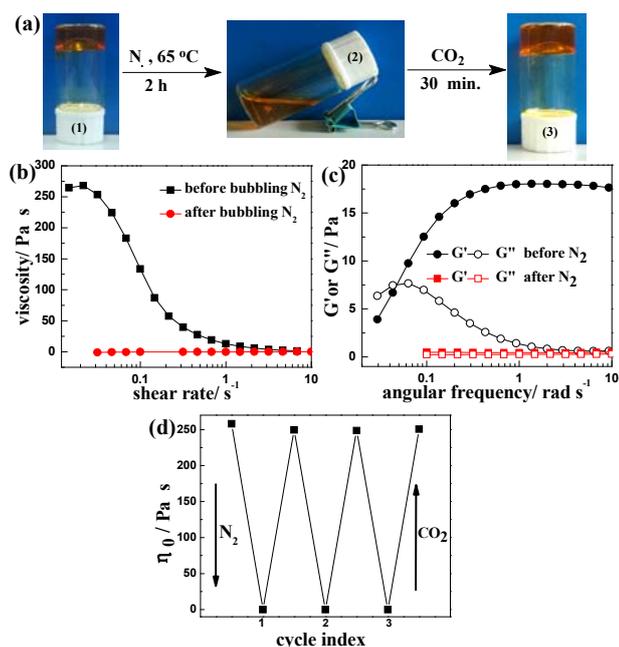


Figure 2. (a) Digital photographs of the CTAB-NaSal-AZO-B6 ternary solution (50/50/30 mM) at 25 °C. (1) Viscoelastic WLMs (gel-like), (2) after bubbling N₂ at 65 °C for 2 h (fluid), (3) after bubbling CO₂ at room temperature for 30 min (gel-like). (b) Steady-state rheology of the CTAB-NaSal-AZO-B6 ternary solution with or without bubbling N₂. (c) Dynamic rheology of the CTAB-NaSal-AZO-B6 ternary solution. (d) Reversible change of the zero-shear viscosity of the CTAB-NaSal-AZO-B6 ternary solution by bubbling CO₂ and N₂ alternately. All the viscosities were measured at 25 °C.

To quantitatively characterize the CO₂-responsive behaviour of the CTAB-NaSal-AZO-B6 ternary system, steady-state and dynamic rheological measurements were made. As depicted in Figure 2(b), the CTAB-NaSal-AZO-B6 ternary solution displays a steady rheological behaviour of a viscoelastic fluid before treatment with N₂. The ternary solution gives a Newtonian platform at low shear rates, but displays shear-thinning behaviour with increasing shear rate due to the presence of viscoelastic WLMs ($\eta_0 = 268$ Pa s). However, after bubbling N₂ at 65 °C for 2 h, the viscosity of the ternary solution (at 25 °C) decreases dramatically and remains at 0.02 Pa s, comparable to that of pure water, with little change regardless of the shear rate, *i.e.* typical Newtonian fluid behaviour. Furthermore, the values of G' and G'' after N₂ bubbling are very small, and the curves do not cross each other throughout the range of shear frequency (Figure 2(c)). This does not conform to the linear viscoelastic-fluid behaviour of the Maxwell model, indicating that the worm-like micelles are destroyed after treatment with N₂. Therefore, it can be concluded that the ternary system of CTAB-NaSal-AZO-B6 is CO₂-responsive, and the high viscosity of the ternary solution can be repeatedly and reversibly switched on/off following bubbling with CO₂/N₂. As can be seen in Figure 2(d), η_0 can return to its original value without any deterioration after 3 cycles.

In order to investigate the structures of the micelles formed in the ternary system, the WLMs at “on” and “off” status were observed by cryo-TEM. Before N₂ treatment, flexible and elongated WLMs that entangle each other were evidenced (Figure 3(a)), which have a diameter of several nanometers and a length in the micrometer range. After N₂ treatment (removing CO₂) only small spherical micelles were found (Figure 3(b)), which have diameters agreeing with the average value of 2.7 nm obtained from dynamic light scattering (DLS) (Figure S8). Therefore, the significant variation in viscosity of the CTAB-NaSal-AZO-B6 ternary solution can be unambiguously attributed to the transformation between the entangled networks of worm-like micelles and discrete spherical micelles.

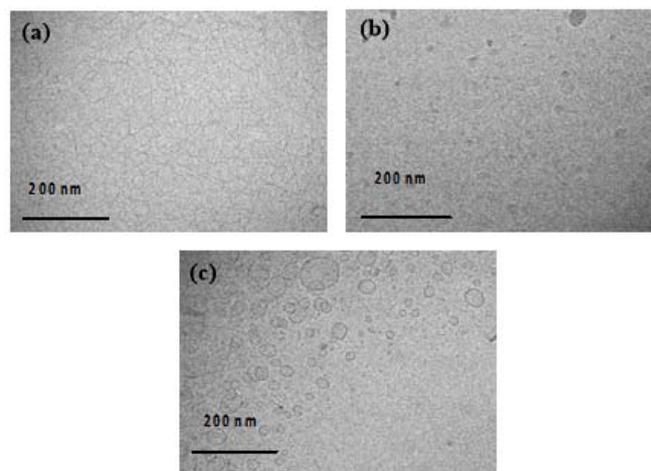


Figure 3. Cryo-TEM images of the CTAB-NaSal-AZO-B6 ternary solution (50/50/30 mM) (a) without bubbling N₂ or UV irradiation, (b) solution (a) after bubbling N₂, (c) solution (a) after UV irradiation.

When the CTAB-NaSal binary solution was heated to 65 °C with magnetic stirring (3 h) and then cooled (without stirring) to room temperature, the viscosity of the solution showed a small increase ($\eta_0 = 67$ Pa s) at low shear rates and the shear-thinning behaviour was similar. However the viscosity of the binary solution did not change by stirring without heating at room temperature (Figure S6). On the other hand, for the same binary solution after UV irradiation (30 min) at room temperature, only a small change in η_0 was observed. It is worth noting that η_0 also remains constant when CO₂ was streamed into the binary solution at room temperature (20 min), which implies that CO₂ cannot induce an obvious rheological response in the CTAB-NaSal binary solution²⁴ or that neither CTAB nor NaSal is CO₂-sensitive.

After removal of CO₂, AZO-B6 in the CTAB-NaSal-AZO-B6 ternary solution is deprotonated to a neutral and surface-inactive hydrophobic tertiary amine, which becomes solubilized within the micelles of CTAB-NaSal, as reflected by a transparent solution. This results in a decrease of the total surfactant concentration to below that required to form gel-like WLMs. It seems that the solubilization effect is dominant for the collapse of the WLMs. When the neutral AZO-B6 (tertiary amine) was added into a CTAB-NaSal (50/50 mM)

binary solution, η_0 of the solution decreases dramatically from 58 Pa s to 8 Pa s by adding only 2 mM of the tertiary amine, which further decreases to 0.07 Pa s at 15 mM of the tertiary amine as shown in Figure S9. All the solutions are transparent indicating solubilization, which favours the transition of the micelle structure from worm-like to spherical leading to a decrease of viscosity.

When CO₂ is bubbled into the mixed solution, the AZO-B6 molecules in surface-inactive form are protonated to surface active form again, recovering the viscoelasticity of the WLMs. Similar results have been reported by Zhang *et al.*²⁴, where a CTAB-NaSal worm-like micellar system was observed to become CO₂-responsive when CO₂-responsive triethylamine (TEA) is added, which allows a transformation of micelles from spheres to worms.

The WLMs of the CTAB-NaSal-AZO-B6 ternary system with AZO-B6 in the cationic form were also found to be responsive to UV and visible light irradiation. Firstly, the highly viscoelastic gel-like ternary solution in which AZO-B6 is in the cationic form (Figure 4(a) (1)) can be transformed to a runny fluid with considerably low viscosity (Figure 4(a) (2)) after UV irradiation for 30 min. Secondly, the viscoelasticity of the solution can be recovered by blue light irradiation for 2 h (Figure 4(a) (3)) and the zero-shear viscosity returns to the original value, as shown in Figure 4(e) where the system was cycled for three times. The curves of G' and G'' after UV irradiation do not correspond to Maxwell-model behaviour in the measured frequency range (Figure 4d), which demonstrates destruction of the WLMs. This can also be confirmed by cryo-TEM (Figure 3(c)), where the typical network structure of worm-like micelles disappeared and vesicles appeared with diameters between *ca.* 10 and 100 nm after UV irradiation. Due to the transformation from WLMs to vesicles, the η_0 of the ternary solution undergoes a significant decrease from 268 Pa s to 0.03 Pa s following UV irradiation (Figure 4(c)).

The light response of the systems can be attributed to the *trans*- to *cis*- state transition of the AZO-B6 surfactant (Scheme 1). This transition can be confirmed by UV-Vis absorption spectra (Figure 4(b)) and the ¹H NMR spectrum of the aromatic protons. Based on the integral calculations of ¹H NMR, 95% of the molecules are in the *cis* state after UV irradiation (Figure S4). Before UV irradiation the maximum absorption peak (at 350 nm) in the UV-Vis spectra of the AZO-B6 solution is due to the $\pi \rightarrow \pi^*$ transition of *trans*-AZO-B6.^{25,26} But UV irradiation results in a remarkable decrease in absorption intensity and a blue-shift of the absorption peak, and a photostationary state can be attained after 1 min of UV irradiation. The changes of the absorption spectrum confirm the photo-isomerization of *trans*-AZO-B6 to *cis*-AZO-B6. It is worth noticing that the light isomerization can be reversed when the sample is irradiated with blue light (436 nm), as shown in Figure 4(b), where the characteristic absorption peak at 350 nm appears again.

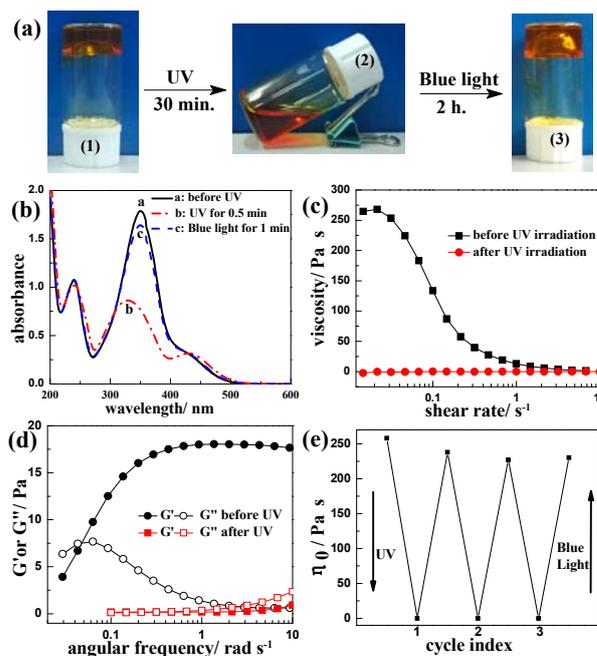


Figure 4. Change of appearance and properties of CTAB-NaSal-AZO-B6 ternary solution (50/50/30mM) following light irradiation at 25 °C. (a) Photographs of ternary solution at 25 °C, (1) viscoelastic worm-like micelles, (2) after UV irradiation for 30 min, (3) after blue light irradiation for 2 h; (b) UV-Vis spectra of AZO-B6 solution (10^{-4} M) after UV and blue light irradiation for different times; (c) steady-state rheology of the CTAB-NaSal-AZO-B6 ternary solution before and after UV irradiation; (d) dynamic rheology of the ternary solution before and after UV irradiation; (e) reversible cycling of zero-shear viscosity of the ternary solution following UV/blue light irradiation.

For the CTAB-NaSal-AZO-B6 ternary system, the decrease in viscosity after UV irradiation may be explained as a change of the micelle structure from worms to vesicles (Figure 3a, c). Compared to *trans*-AZO-B6, the *cis*-AZO-B6 molecule has a larger effective volume of the hydrophobic chain. The insertion of the *cis*-AZO-B6 molecules into the micelles thus changes the packing parameter P (below), resulting in transformation of the micelle structure from worm-like to vesicular and a drastic viscosity decrease. This explanation is well confirmed by the rheology measurements and cryo-TEM image (Figure 3c). Dynamic light scattering shows that the average diameter of the vesicles is about 40 nm (Figure S10). Therefore, the reversible transformation of the system between gel-like WLMs and fluid vesicular solution following UV/blue light irradiation can be attributed to the reversible *trans*-*cis* photoisomerization of AZO-B6.

Based on the above rheological behaviour and cryo-TEM microstructure of the system, it can be seen that the addition of AZO-B6 to the CTAB-NaSal system not only increases the bulk viscosity but also endows the system with dual CO₂ and light stimuli-responsive properties. In general, the morphology of surfactant micelles can be empirically predicted by the critical packing parameter,^{19,27}

$$P = v / (l \cdot a) \quad (1)$$

where v is the volume of the hydrophobic surfactant chain, l is the effective length of the alkyl chain and a is the effective area of the surfactant headgroup at the surfactant-water

interface. This packing parameter has been employed to explain the mechanism of the phase transition. For $P < 1/3$, it is usually forecasted that the surfactant molecules assemble into globular micelles. For $1/3 < P < 1/2$, rod-like or worm-like micelles are obtained. Further when $1/2 < P < 1$, vesicles are obtained. When $P = 1$, planar bilayers are obtained (lamellar phase) and if $P > 1$ inverted micelles are formed.

The cationic surfactant *trans*-AZO-B6, whose hydrophobic alkyl chain is linear, can insert itself into the palisade layer of the aggregates of CTAB forming mixed micelles, but UV irradiation will induce an increase in v without much change in a . The packing parameter of the mixed micelles will increase and provided the value of P remains between $1/3$ and $1/2$, the mixed system will still prefer worm-like micelles. On the one hand, after removal of CO_2 , AZO-B6 can be deprotonated into a neutral form. The inactive form of AZO-B6 may be solubilized inside the micelles and result in swelling of the WLMs, reflecting a transition of micelle structure from worms to spheres and a decrease in viscosity. Thus, P will decrease resulting in a microstructure transformation from worm micelles to globular micelles. When CO_2 is bubbled into this mixed solution, the inactive form of AZO-B6 can be protonated again, reverting the system to the viscoelastic worm-like micelles, as illustrated in Figure 5.

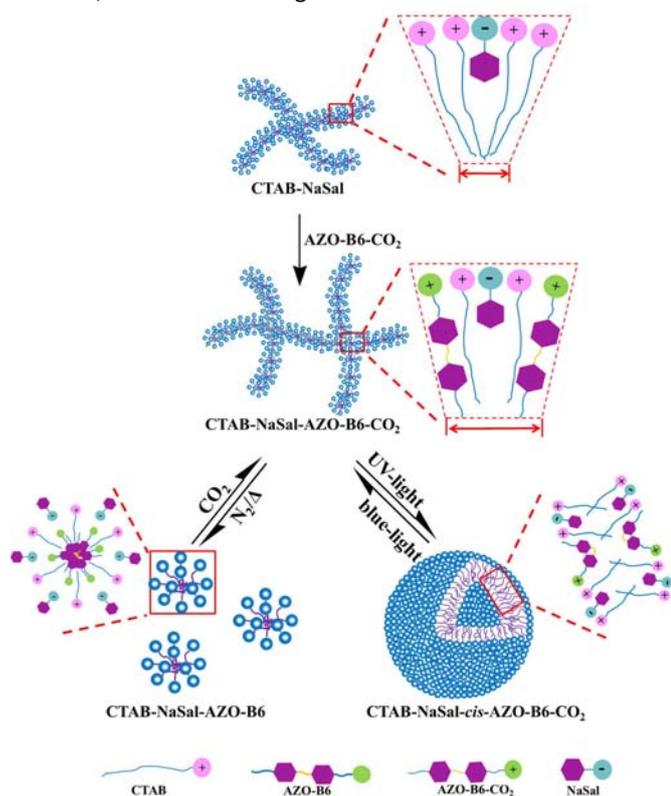


Figure 5. Possible molecular configuration changes in dual-stimuli responsive CTAB-NaSal-AZO-B6- CO_2 mixed micelle systems.

For the CTAB-NaSal-AZO-B6 ternary system, the viscosity is decreased after UV irradiation and the microstructure changes from worm-like micelles to vesicles. The reversible *trans-cis* photoisomerization of AZO-B6 causes changes in the geometrical structure and interfacial properties of the

surfactant, thus leading to the reversible formation and disruption of molecular assemblies. Compared to the *trans* form, *cis*-AZO-B6 has a short effective length of its hydrophobic chain and a large volume. This increases the value of P to between $1/2$ and 1 , which leads to the transformation from worm-like micelles to vesicle structures. Such a prediction can be confirmed by the results of rheology and cryo-TEM. Therefore, the reversible formation and disruption of molecular assemblies can be attributed to the reversible *trans-cis* photoisomerization of AZO-B6.

Conclusions

A dual stimulus-responsive WLM system with CO_2 and light triggers can be prepared by addition of a relatively small amount (5 mM) of the dual-switchable surfactant AZO-B6 into a classic CTAB-NaSal binary WLM system. The high viscosity of the CTAB-NaSal-AZO-B6 ternary solution can be reversibly switched on/off by either bubbling CO_2/N_2 or by UV/blue light irradiation. The CO_2/N_2 trigger controls the change of the AZO-B6 molecules between surface-active and surface-inactive forms. The surface-active form increases the total concentration of the cationic surfactants to the concentration required for forming gel-like WLMs, and the surface-inactive form is solubilized inside the micelles to change the geometry of the micelle favouring formation of spherical micelles. The light trigger induces a reversible *trans-cis* photoisomerization of AZO-B6, where the *trans*-isomer favours the formation of viscoelastic systems but the *cis*-isomer does not. This dual responsive viscoelastic system is expected to have potential applications in biomedicine, microfluidics, analytical chemistry and in tertiary oil recovery.

Acknowledgements

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