Reversible dispersion and precipitation of single-walled carbon nanotubes using a pH-responsive rigid surfactant

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1 Detailed synthetic processes and characterizations of Na-MPA-AZO-Na

1.1 Materials

Gum rosin was purchased from West Tech Chemical Co., Ltd. P-phenylenediamine and phenol were obtained from Aladdin Chemistry Company, Ltd. Maleic anhydride, acetic acid, N, N-Dimethylformamide (DMF), sodium hydroxide (NaOH) and other solvents and reagents were analytical grade and received from Sinopharm Chemical Reagent Company, Ltd. All the materials were used without further purification.

1.2 Synthesis and characterizations

1.2.1 Synthesis of Maleopimaric acid (MPA) The gum rosin (200 g, 0.66 mol), maleic anhydride (70 g, 0.72 mol) and acetic acid (80 g) were added into a 1000 mL three-necked flask. Then, a mechanical stirring device and a reflux condenser were installed. The mixtures were heated to 150 °C and maintained for 5 h. When the temperature of reaction was cooled to room temperature, the reaction was added into acetic acid (250 g). Then the mixture was recrystallized at 25 °C, and white crystals were obtained. Lastly, the crystals were recrystallized twice using acetic acid. The obtained MPA is 126.6 g, yield: 43.9%.

1H NMR (400 MHz, DMSO, Fig. S1b) δ 12.16 (s, 1H, C18-1H), 5.52 (d,1H, C7-1H), 3.23-3.26 (m, 1H, C5-1H), 2.91-2.95 (m, 2H, C6-1H, C4-1H), 2.32 (d, 1H, C1-1H), 2.16 (d, 1H, C13-1H), 1.71 – 1.32 (m, 10H, C17-2H, C13-1H, C15-1H, C11-1H, C12-2H, C16-1H, C9-1H, C8-1H), 1.18-1.08 (t, 2H, C11-1H, C16-1H), 1.03 (s, 3H, C14-3H), 0.93 (dt, 7H, C2-3H, C3-3H, C9-1H), 0.52 (s, 3H, C10-3H); 13C NMR (101 MHz, DMSO, Fig. S1c) δ 179.63 (s, C18),
173.64 (s, C25), 172.01 (s, C26), 147.37 (s, C27), 125.13 (s, C7), 52.77 (s, C15), 52.33 (s, C8),
48.76 (s, C6), 45.93 (s, C5), 45.50 (s, C30), 39.90 (s, C28), 37.66 (s, C11), 37.11 (s, C29), 36.35
(s, C13), 35.22 (s, C4), 34.35 (s, C17), 32.24 (s, C1), 26.71 (s, C14), 21.24 (s, C9), 20.48 (s,
C16), 20.04 (s, C12), 16.71 (s, C2, C3), 15.22 (s, C10); IR (Fig. S1d) 2923.72 (CH$_3$–), 2853.88
(–CH$_2$–), 1828.81, 1757.13 (–COOOC–), 1712.57 (–COOH); MS (Fig. S1e) Calcd for
C$_{24}$H$_{32}$O$_5$ [M - H]-: 399.2. Found: 399.2 and 400.1; Elemental analysis: anal. calcd. for
C$_{24}$H$_{32}$O$_5$: C, 71.97; H, 8.05 (%). Found: C, 71.36; H, 8.37 (%).
1.2.2 Synthesis of (MPA-ph-NH₂) The P-phenylenediamine (8.9 g, 0.0825 mol) and DMF (90 g) were mixed in a 500 mL three-necked flask. Then, a magnetic rotor was added and a reflux condenser was installed. When the reaction was heated to 80 °C, the DMF solution of MPA (30.0 g, 0.075 mol) was dropped in. The reaction was continued to carry out for 1 h at 80 °C after the MPA was completely added in. And then the reaction was refluxed for 5 h at 160 °C. The mixture was added into water after cooling, and brown solids were obtained after filtering. The MPA-ph-NH₂ (29.3 g, 0.060mol) was obtained after recrystallization twice with DMF and water, yield: 79.7%.

1H NMR (400 MHz, DMSO, Fig. S2b) δ 12.16 (s, 1H, C18-1H), 6.63 (d, 2H, C19-2H), 6.54 (d, 2H, C20-2H), 5.49 (d,1H, C7-1H), 5.27 (s, 2H, N21-2H), 3.00 – 2.91 (m, 2H, C5-1H, C6-1H), 2.65 (t, 1H, C4-1H), 2.41 (d, 1H, C1-1H), 2.14 (dt, 1H, C13-1H), 1.78 – 1.30 (m, 10H, C17-2H, C13-1H, C15-1H, C11-1H, C12-2H, C16-1H, C9-1H, C8-1H), 1.13 (t, 2H, C11-1H, C16-1H), 1.05 (s, 3H, C14-3H), 0.91 (dt, 7H, C2-3H, C3-3H, C9-1H), 0.56 (s, 3H, C10-3H);

13C NMR (101 MHz, DMSO, Fig. S2c) δ 179.70 (s, C18), 177.85 (s, C25), 176.87 (s, C26), 148.72 (s, C32), 146.47 (s, C27), 127.40 (s, C7), 124.34 (s, 2C19), 120.35 (s, C31), 113.48 (s, 2C20), 53.41 (s, C15), 51.68 (s, C8), 48.98 (s, C6), 45.98 (s, C5), 44.42 (s, C30), 40.44 (s, C28), 37.87 (s, C11), 37.20 (s, C29), 36.41 (s, C13), 35.59 (s, C4), 35.14 (s, C17), 32.32 (s, C1), 27.18 (s, C14), 21.41 (s, C9), 20.70 (s, C16), 20.12 (s, C12), 16.75 (s, C2, C3), 15.40 (s, C10); IR
(Fig. S2d) 3363.42, 3234.11, 1622.93 (–NH₂), 2939.13 (CH₃–), 2865.78 (–CH₂–), 1764.84, 1720.14 (–CONOC–), 1695.53 (–COOH), 1679.32 (–CON–), 1517.83, 801.01 (–phenyl); MS (Fig. S2e) Calcd for C30H38N2O4 [M - H]-: 489.2. Found: 489.1, 489.2 and 490.1; Elemental analysis: anal. calcd. for C30H38N2O4: C, 73.44; H, 7.81, N, 5.71 (%). Found: C, 73.68; H, 7.93, N, 5.70 (%).
1.2.3 Synthesis of (MPA-AZO-OH) Mixtures of MPA-ph-NH$_2$ (20.0 g, 0.034 mol), distilled water (3.5 g) and concentrated hydrochloric acid (11 g) were added in a 250 mL three-necked flask containing a magnetic rotor. Then, the reaction was maintained in an ice salt bath. The aqueous solution of sodium nitrite (3.1 g, 0.046 mol) was dropped in below 5 °C. When the reaction solution changed in yellow, the solution of diazonium salt was obtained and kept in an ice salt bath. Phenol (3.3 g, 0.036 mol) and distilled water (20 g) were added into a 500 mL three-necked flask containing a magnetic rotor, and the pH value was adjusted to 9-10. The above solution of diazonium salt was slowly added in. The reaction was continued to carry out for 30 min after the solution of diazonium salt was completely added in. The temperature was below 5 °C through the reaction. The mixture was filtered, and Filtered solid was washed twice using deionized water. The yellow solid was purified through silica column chromatography with petroleum ether/ethyl acetate (2:1) as eluent. The obtained brown red solid was MPA-AZO-OH (12.6 g, 0.021 mol) were obtained, yield: 61.8%.

$^1$H NMR (400 MHz, DMSO, Fig. S3b) δ 12.14 (s, 1H, C18-1H), 10.35 (s, 1H, C24-1H), 7.84 (dd, 4H, C20-2H, C22-2H), 7.27 (d, 2H, C19-2H), 6.95 (d, 2H, C23-2H), 5.54 (s, 1H, C7-1H), 3.07 (dd, 1H, C5-1H), 2.99 (s, 1H, C6-1H), 2.79–2.71 (m, 1H, C4-1H), 2.45 (d, 1H, C1-1H), 2.19 (dt, 1H, C13-1H), 1.79–1.32 (m, 10H, C17-2H, C13-1H, C15-1H, C11-1H, C12-2H, C16-1H, C9-1H, C8-1H), 1.23–1.13 (m, 2H, C11-1H, C16-1H), 1.06 (s, 3H, C14-3H), 0.94
(dd, 7H, C2-3H, C3-3H, C9-1H), 0.58 (s, 3H, C10-3H); $^{13}$C NMR (101 MHz, DMSO, Fig. S3c) δ 179.71 (s, C18), 177.32 (s, C25), 176.31 (s, C26), 161.32 (s, C34), 151.36 (s, C32), 146.66 (s, C27), 145.27 (s, C33), 133.87 (s, C31), 127.60 (s, C7), 125.09 (s, 2C19), 124.52 (s, 2C20), 122.50 (s, 2C22), 116.05 (s, 2C23), 53.30 (s, C15), 52.01 (s, C8), 48.94 (s, C6), 45.98 (s, C5), 44.79 (s, C30), 40.55 (s, C28), 37.85 (s, C11), 37.23 (s, C29), 36.42 (s, C13), 35.70 (s, C4), 35.06 (s, C17), 32.37 (s, C1), 27.16 (s, C14), 21.41 (s, C9), 20.74 (s, C16), 20.20 (s, C12), 16.76 (s, C2, C3), 15.39 (s, C10); IR (Fig. S3d) 3304.57, 1381.06 (–OH), 2917.43 (CH$_3$–), 2857.99 (–CH$_2$–), 1771.88 (–CONOC–), 1696.39 (–COOH), 1677.06 (–CON–), 1599.68, 841.34 (–phenyl), 1278.79 (–CN=NC–); MS (Fig. S3e) Calcd for C$_{36}$H$_{41}$N$_3$O$_5$ [M - H]-: 594.2. Found: 594.2 and 595.2; Elemental analysis: anal. calcd. for C$_{36}$H$_{41}$N$_3$O$_5$: C, 72.58; H, 6.94, N, 7.05 (%). Found: C, 72.24; H, 7.09. N, 7.05 (%).
1.2.4 Synthesis of (Na-MPA-AZO-Na)  
MPA-AZO-OH (10.0 g, 0.017 mol), sodium hydroxide (0.74 g, 0.018 mol) and ethanol (100 mL) were added into a 250 mL eggplant-type flask. A magnetic rotor was added and a reflux condenser was installed. The reaction was carried out for 6 h at 55 °C. After cooling, the solvent was removed and the resultant solid was recrystallized twice with ethanol and acetone. Yellow solid of Na-MPA-AZO-Na (8.6 g) was obtained, yield: 79.1%.

$^1$H NMR (400 MHz, CD3OD, Fig. S4b) δ 7.76 (dt, 4H, C20-2H, C22-2H), 7.23 (d, 2H, C19-2H), 6.72 (t, 2H, C23-2H), 5.61 (s, 1H), 3.19–3.00 (m, 2H, C5-1H, C6-1H), 2.81 (t, 1H, C4-1H), 2.54 (d, 1H, C1-1H), 2.27 (dd, 1H, C13-1H), 2.00–1.68 (m, 4H, C17-1H, C13-1H, C15-1H, C11-1H), 1.64–1.28 (m, 8H, C17-1H, C13-1H, C15-1H, C11-1H), 1.16 – 0.94 (m, 10H, C14-3H, C2-3H, C3-3H, C9-1H), 0.69 (s, 3H, C10-3H); $^{13}$C NMR (101 MHz, CD3OD, Fig. S4c) δ 187.31 (s, C18), 179.79 (s, C25), 178.26 (s, C26), 153.77 (s, C34), 148.08 (s, C32), 144.93 (s, C27), 133.89 (s, C33), 128.27 (s, C31), 126.53 (s, C7), 126.20 (s, C21), 123.07 (s, C22), 118.81 (s, C23), 55.34 (s, C15), 53.79 (s, C8), 50.95 (s, C6), 46.43 (s, C5), 42.24 (s, C30), 39.62 (s, C28), 38.78 (s, C11), 38.50 (s, C29), 37.49 (s, C13), 36.59 (s, C4), 33.93 (s, C17), 28.59 (s, C1), 22.74 (s, C14), 21.13 (s, C9), 20.52 (s, C16), 18.59 (s, C12), 18.38 (s, C2, C3), 16.17 (s, C10); IR (Fig. S4d) 2929.11 (–CH$_3$–), 2866.57 (–CH$_2$–), 1773.47 (–CONOC–), 1586.66, 1446.55 (–COO$^-$Na$^+$), 1544.83, 842.49 (–phenyl), 1381.14 (–
O\textsuperscript{−}Na\textsuperscript{+}), 1273.55 (−CN=NC−); MS (Fig. S4e) Calcd for C\textsubscript{36}H\textsubscript{39}N\textsubscript{3}O\textsubscript{5}Na\textsubscript{2} [M - 2Na]−: 594.27. Found: 594.3 and 595.3. Elemental analysis: anal. calcd. for C\textsubscript{36}H\textsubscript{39}N\textsubscript{3}O\textsubscript{5}Na\textsubscript{2}: C, 67.59; H, 6.15, N, 6.57 (%). Found: C, 67.26; H, 6.32. N, 6.53 (%). The purity of Na-MPA-AZO-Na is 98.78% (HPLC, Fig. S4f).
2 Method

2.1 Characterization

$^1$H NMR and $^{13}$C-NMR measurements were performed on an Advance III 400 MHz spectrometer (Bruker, Germany). FT-IR spectra were obtained using a Thermo Scientific Nicolet iS10 FT-IR spectrometer (Thermo Fisher Scientific, USA) in the 400-4000 cm$^{-1}$ wavenumber range at 4 cm$^{-1}$ resolution and averaged over 16 scans per sample. Mass spectrometric detection was conducted on an Agilent-5973 spectrometer (Agilent Technologies, USA). Elemental analysis measurements were performed on a PE 2400 series II elemental analyzer.

2.2 Transmittance for Na-MPA-AZO-Na aqueous solutions

The measurement of transmittance was performed on a UV2450 UV–Vis spectrometer (Shimadzu, Japan) using a quartz cell with 1 cm beam path length at 25 °C. The background correction was performed with ultrapure water (18.2 MΩ cm). The concentration of Na-MPA-AZO-Na for all samples is 4 mM.

2.3 Preparation of SWNT dispersions

Single-walled carbon nanotubes (SWNTs) were purchased from Aladdin Chemistry
Company, Ltd, and the diameter of SWNTs is less than 2 nm and the length is between 5 and 30 μm. SWNTs (8 mg) and the aqueous solutions of Na-MPA-AZO-Na at a concentration of 4 mM and a pH of 10.40 (20 mL) were added to a reagent bottle. The mixtures were treated by sonication for 20 min using an Ultrasonic Cell disrupter System at 0.25 kW. And then the resultant suspensions were handled by centrifugation for 20 min at 8000 rpm. And the pH of the SWNT dispersions was adjusted to 10.40±0.05. The water used in all samples is ultrapure water with a resistivity of 18.2 MΩ cm.

2.4 UV-vis-NIR spectrum

The UV-vis-NIR measurements were carried on a PerkinElmer L-750 UV-vis-NIR spectrometer using a quartz cell with 1 cm beam path length at 25 °C. A background correction was performed with the Na-MPA-AZO-Na aqueous solutions. The SWNT dispersions used in the test was diluted 10 times.

2.5 Zeta potential

The zeta potential was obtained using a Malvern zeta potentialmeter (Malvern Instruments Ltd., Worcestershire, UK) equipped with 1 cm path length quartz electrode cuvettes. The SWNT dispersions used in the test was diluted 10 times.

2.6 Transmission electron microscopy (TEM)

2 μL of samples were loaded on a copper grid using a micropipette, and then the sample was dried in vacuum drying oven at 60 °C for 4h. The dried sample was rinsed with ultrapure water for 6 times and was dried naturally. The images were obtained using a JEOL JEM-1400 TEM (120kV) at 25 °C.

2.7 Cryogenic-transmission electron microscopy (Cryo-TEM)

2 μL of SWNT dispersions were loaded on a copper grid using a micropipette, and then two pieces of filter were used to blot the solution at the same time. Then the thin films could be formed on the mesh holes. And the copper grid was then rapidly thrown into liquid propane. The vitrified sample was reserved in liquid nitrogen before it was diverted into a cryogenic sample holder (Gatan 626). The sample was detected using a JEOL JEM-1400 TEM (120kV) at approximate −174 °C. The Cryo-TEM images were recorded by a Gatan multiCCD containing a digital micrograph.

2.8 High performance liquid chromatography
Chromatographic separation was performed on a high performance liquid chromatography (Agilent Technology, 1260 Infinity, USA) equipped with a C18 column (Agilent, Zorbax Eclipse Plus C18). The mobile phase is consisted of methanol (80%) and water (20%), the flow rate was constant at 1 mL·min⁻¹, the column temperature was set to 35 °C, and the absorbance wavelength was also set to 354 nm.

2.9 Recovery of SWNTs after the precipitation

When the pH of SWNT dispersion is lower than 5.35, the SWNTs precipitate from the dispersion. The SWNT dispersion at pH 5.35 is treated by centrifugation at 8000 rpm for 10 min. After removing the supernatant, desired amounts of N, N-dimethylformamide (DMF) are added into the centrifuge tube. Then the complex is treated by sonication for 2 min and centrifugation at 8000 rpm for 10 min. The supernatant turns into yellow, and the phenomenon demonstrates that the acid form of Na-MPA-AZO-Na dissolve in DMF. The supernatant after centrifugation is removed. The above process is cycled until the supernatant becomes colorless. Surfactants attached on SWNTs are removed, and clean and non-polluted SWNTs are obtained.

3 Results

3.1 Transmittance of the Na-MPA-AZO-Na aqueous solutions

The transmittance was measured to detect the solubility of Na-MPA-AZO-Na (4 mM) at different pH, and the results are shown in Fig. S5. When the pH is larger than 9.97, the transmittance of Na-MPA-AZO-Na aqueous is large than 99.2%, which demonstrates that Na-MPA-AZO-Na can completely dissolve in water. And the transmittance then decreases with decreasing pH when the pH is lower than 9.97. As the pH drops to 6.52, the transmittance is 3.81%, which demonstrates lots of Na-MPA-AZO-Na participates from its aqueous solutions. Therefore, the pH for Na-MPA-AZO-Na aqueous solutions is fixed at 10.40±0.05 in the dispersion of SWNTs.
3.2 The SWNT dispersion at pH 6.56

According to the results of transmittance for the aqueous solutions at a concentration of 4 mM, when the pH decreases to 6.56, lots of Na-MPA-AZO-Na participates from its aqueous solutions. Decreasing the pH of the SWNT dispersion to 6.56, the appearance of SWNT dispersion at pH 6.56 is shown in Fig. S6a. The supernatant is light black, and its UV-vis-NIR spectrum is shown in Fig. S6b. The absorption intensity of the UV-vis-NIR spectrum decreases, but the characteristic peaks still exist. So a bit of SWNTs are in individuals in the Na-MPA-AZO-Na aqueous solutions at pH 6.56.

3.3 TEM of the SWNT dispersion at pH 10.36

The TEM of SWNT dispersion at pH 10.36 as a complementary to Cryo-TEM is exhibited in Fig. S7, the individual SWNTs shown in the image confirms the result of Cryo-TEM.
3.4 UV-vis-NIR

To detect the efficiency of re-dispersion without sonication, the SWNT dispersion after a pH-switchable cycle treated by sonication for 20 min was studied using UV-vis-NIR. As Fig. S8 shows, the absorption intensity and characteristic peaks of the UV-vis-NIR spectrums for SWNT dispersion with and without sonication are quite similar. Thus, the precipitated SWNTs can be completely re-dispersed without sonication.

![Fig. S8](image)

**Fig. S8** UV-vis-NIR spectrums for the SWNT dispersion after a pH-switchable cycle treated by sonication and shaking (the original liquid were diluted 20 times)

3.5 Acid-base titration

In order to detect the molecular structure of Na-MPA-AZO-Na in its aqueous solutions at different pH, the concentration of hydrogen ions was measured by acid-base titration. In the light of the results of transmittance (Fig. S5), when the pH is larger than 9.97, the Na-MPA-AZO-Na at a concentration of 4 mM can completely dissolve in water. So the end point of titration is set to pH 9.97. The concentration of hydrogen ions in the Na-MPA-AZO-Na aqueous solutions at different pH is exhibited in Fig. S9a. As Fig. S9a shows the concentration of
hydrogen ions monotonically decreases with increasing pH value. When the pH is lower than 5.96, the concentration of hydrogen ions is over 8 mM. All sodium phenolates and sodium carboxylates convert to their corresponding acid form. So the SWNTs absolutely precipitate from the dispersions when the pH decreases to 5.35.

In addition, the deprotonation or protonation of Na-MPA-ZAO-Na have been investigated by potentiometric titration. Na-MPA-ZAO-Na (0.05g), sodium hydroxide (NaOH, 0.1 M, 10 mL) aqueous solution and H₂O (40 mL) were added into a conical flask. When Na-MPA-ZAO-Na was completely dissolved, pH was measured at different volume of added hydrochloric acid (HCl), and the results are shown in Fig. S9b. When the pH decreases to 9.95, the sodium phenolates begin to convert into their corresponding phenol. Thus, when the pH is above 9.95, the presence of Na-MPA-ZAO-Na is shown as Fig. S9c. Further decreasing pH to 5.74, the sodium phenolates and sodium carboxylates completely convert into their corresponding acid form (Fig. S9b). And the acid form of Na-MPA-ZAO-Na is shown in Fig. S9c.

Fig. S9 (a) the concentration of the hydrogen ions in the aqueous solutions of Na-MPA-ZAO-Na (4 mM) at different pH, (b) variation of pH with the volume of HCl in the aqueous solutions of Na-MPA-ZAO-Na (1 g/L)/NaOH, (c) schematic representation of Na-MPA-AZO-Na structure induced by the changes in pH.

3.6 TEM of the SWNTs precipitates at pH 5.35 and in original sample

The enlarged TEM image of the SWNT dispersion at pH 5.35 is shown in Fig. S10a. As
Fig. S10a shows, SWNTs are still isolated in the bundles. The result demonstrates that the large rigid group hampers the contact between SWNTs. But the SWNTs are closely aggregated within the bundles in original sample shown in Fig. S10b. There is no gap between the SWNTs.

**Fig. S10** TEM images of (a) reformed precipitated SWNTs at pH 5.35 and (b) the SWNTs in original sample

### 3.7. Reversibility of the pH-responsive SWNT dispersion

The TEM image for the SWNT dispersion at pH 10.36 in cycle 3 is shown in Fig. S11, the individual SWNTs distribute in Fig. S11. The result verifies that the dispersion and precipitation of SWNTs can be switched for at least 3 times by the changes in pH without sonication.

**Fig. S11** TEM image of the SWNT dispersions at pH 10.36 in cycle 3

Reversibility of the pH-responsive SWNT dispersion has been further investigated. As Fig. S12a shows, the homogenous and stable SWNT dispersion at pH 10.36 in Cycle 6 indicates that the dispersion and precipitation states of SWNTs can be further switched after three times. To detect influences of accumulated salts from neutralization of acid and alkali on the further cycle of pH-responsive SWNT dispersion, desired amounts of sodium chloride (NaCl) were
added into the SWNT dispersion at pH 10.36. The concentration of NaCl was adjusted to 0.1 M. The photograph of SWNT dispersion at pH 10.36 with 0.1 M NaCl taken after two weeks is shown in Fig. S12b. The homogenous SWNT dispersion reveals the salts have no obvious influences on the dispersion of SWNTs. The SWNT dispersions before and after adding NaCl were then investigated using UV-vis-NIR spectroscopy to assess the influence of salts. The results are shown in Fig. S12c. The intensity of the absorption spectrum and the characteristic peaks are quite similar for the SWNT dispersion before and after adding NaCl. The above results demonstrate the accumulation of salts has little influence on the dispersion of SWNTs. The pH-responsive SWNT dispersion using Na-MPA-AZO-Na shows excellent reversibility.

**Fig. S12** photographs of (a) SWNT dispersion at pH 10.36 in Cycle 6 and (b) SWNT dispersion at pH 10.36 with 0.1 M NaCl, (c) UV-vis-NIR spectra of SWNT dispersion before and after adding NaCl