



# Tunable Fluorescence via Self-Assembled Switching of AIE-Active Micelle-like Nanoaggregates

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## METHODS

**1-bromo-4-ethoxybenzene(2a):** Add 4-bromophenol (0.52 g, 3.0 mmol) and  $K_2CO_3$  (1.2 g, 9.0 mmol) into a 100 mL double-necked round bottom flask and use 20 mL of acetone to dissolve the compound. Then slowly add 1-iodoethane (360  $\mu$ L, 4.5 mmol) and react under reflux for 12 hours. The solvent was removed under reduced pressure. Extracted twice with  $CH_2Cl_2/H_2O$ , and the organic layer was dried over anhydrous magnesium sulfate ( $MgSO_4$ ). Purify via silica column chromatography (the eluent was n-hexane) to obtain a transparent liquid with a yield of 80 %.  $^1H$  NMR (400 Hz,  $CDCl_3$ ):  $\delta$  = 7.36 (d, J = 9.2 Hz, 2H), 6.77 (d, J = 9.2 Hz, 2H), 3.99 (q, J = 7.0 Hz, 2H), 1.40 (t, J = 7.0 Hz, 3H) ppm.

**1-bromo-4-butoxybenzene(4a):** Add 4-bromophenol (0.52 g, 3.0 mmol) and  $K_2CO_3$  (1.2 g, 9.0 mmol) into a 100 mL double-necked round bottom flask and use 20 mL of acetone to dissolve the compound. Then slowly add 1-iodobutane (340  $\mu$ L, 4.5 mmol) and react under reflux for 12 hours. The solvent was removed under reduced pressure. Extracted twice with  $CH_2Cl_2/H_2O$ , and the organic layer was dried over anhydrous magnesium sulfate ( $MgSO_4$ ). Purify via silica column chromatography (the eluent was n-hexane) to obtain a transparent liquid with a yield of 80 %.  $^1H$  NMR (400 Hz,  $CDCl_3$ ):  $\delta$  = 7.36 (d, J = 9.2 Hz, 2H), 6.77 (d, J = 9.2 Hz, 2H), 3.92 (t, J = 7.0 Hz, 2H), 1.75 (m, 2H), 1.48 (m, 2H), 0.97 (t, J = 7.0 Hz, 3H) ppm.

**1-bromo-4-hexyloxybenzene(6a):** Add 4-bromophenol (0.52 g, 3.0 mmol) and  $K_2CO_3$  (1.2 g, 9.0 mmol) into a 100 mL double-necked round bottom flask and use 20 mL of acetone to dissolve the compound. Then slowly add 1-bromohexane (630  $\mu$ L, 4.5 mmol) and react under reflux for 12 hours. The solvent was removed under reduced pressure. Extracted twice with  $CH_2Cl_2/H_2O$ , and the organic layer was dried over anhydrous magnesium sulfate ( $MgSO_4$ ). Purify via silica column chromatography (the eluent was n-hexane) to obtain a transparent liquid with a yield of 80 %.  $^1H$  NMR (400 Hz,  $CDCl_3$ ):  $\delta$  = 7.36 (d, J = 9.2 Hz, 2H), 6.77 (d, J = 9.2 Hz, 2H), 4.06 (t, J = 7.0 Hz, 2H), 1.85 (m, 2H), 1.53 (m, 2H), 1.38 (m, 4H), 0.93 (t, J = 7.0 Hz, 3H) ppm.

**1-bromo-4-dodecyloxybenzene(12a):** Add 4-bromophenol (0.52 g, 3.0 mmol) and  $K_2CO_3$  (1.2 g, 9.0 mmol) into a 100 mL double-necked round bottom flask and use 20 mL of acetone to dissolve the compound. Then slowly add 1-bromododecane (1080  $\mu$ L, 4.5 mmol) and react under reflux for 12 hours. The solvent was removed under reduced pressure. Extracted twice with  $CH_2Cl_2/H_2O$ , and the organic layer was dried over anhydrous magnesium sulfate ( $MgSO_4$ ). Purify via silica column chromatography (the eluent was n-hexane) to obtain a transparent liquid with a yield of 80 %.  $^1H$ NMR(400 Hz,  $CDCl_3$ ):  $\delta$  = 7.36 (d, J = 9.2 Hz, 2H), 6.77 (d, J = 9.2 Hz, 2H), 3.91 (t, J = 7.0 Hz, 2H), 1.76 (m, 2H), 1.44(m, 2H), 1.26(m, 16H), 0.88(t, J = 7.0 Hz, 3H) ppm

**2-(4-methoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane(1b):** Purge the high-pressure bottle by nitrogen for about 1 minute, then put  $Pd(PPh_3)_2Cl_2$  (30 mg) in the high-pressure bottle, and then mix 4-bromoanisole (0.47 g, 2.5 mmol) and the mixed solvent  $Et_3N/1,4$ -dioxane = 1/3 (v/v) of 12 mL into it and stir evenly with a magnet. Finally, add pinacoborane (630  $\mu$ L, 3.75 mmol) and bubble with nitrogen for about 2 minutes. After refluxing for 1 day, the mixture was cooled down to room temperature. Extracted twice with  $CH_2Cl_2/H_2O$ , and the organic layer was dried over anhydrous magnesium sulfate ( $MgSO_4$ ). Purify via silica column chromatography (the eluent was n-hexane) to obtain transparent liquid. The yield is about 70%.  $^1H$ NMR (400 Hz,  $CDCl_3$ ):  $\delta$  = 7.75 (d, J = 9.2 Hz, 2H), 6.89 (d, J = 9.2 Hz, 2H), 3.82 (s, 3H), 1.33(s, 12H) ppm.

**2-(4-ethoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane(2b):** Purge the high-pressure bottle by nitrogen for about 1 minute, then put  $Pd(PPh_3)_2Cl_2$  (30 mg) in the high-pressure bottle, and then mix **2a** (0.50 g, 2.5 mmol) and the mixed solvent  $Et_3N/1,4$ -dioxane = 1/3 (v/v) of 12 mL into it and stir evenly with a magnet. Finally, add pinacoborane (630  $\mu$ L, 3.75 mmol) and bubble with nitrogen for about 2 minutes. After refluxing for 1 day, the mixture was cooled down to room temperature. Extracted twice with  $CH_2Cl_2/H_2O$ , and the organic layer was dried over anhydrous magnesium sulfate ( $MgSO_4$ ). Purify via silica column chromatography (the eluent was n-hexane) to obtain transparent liquid. The

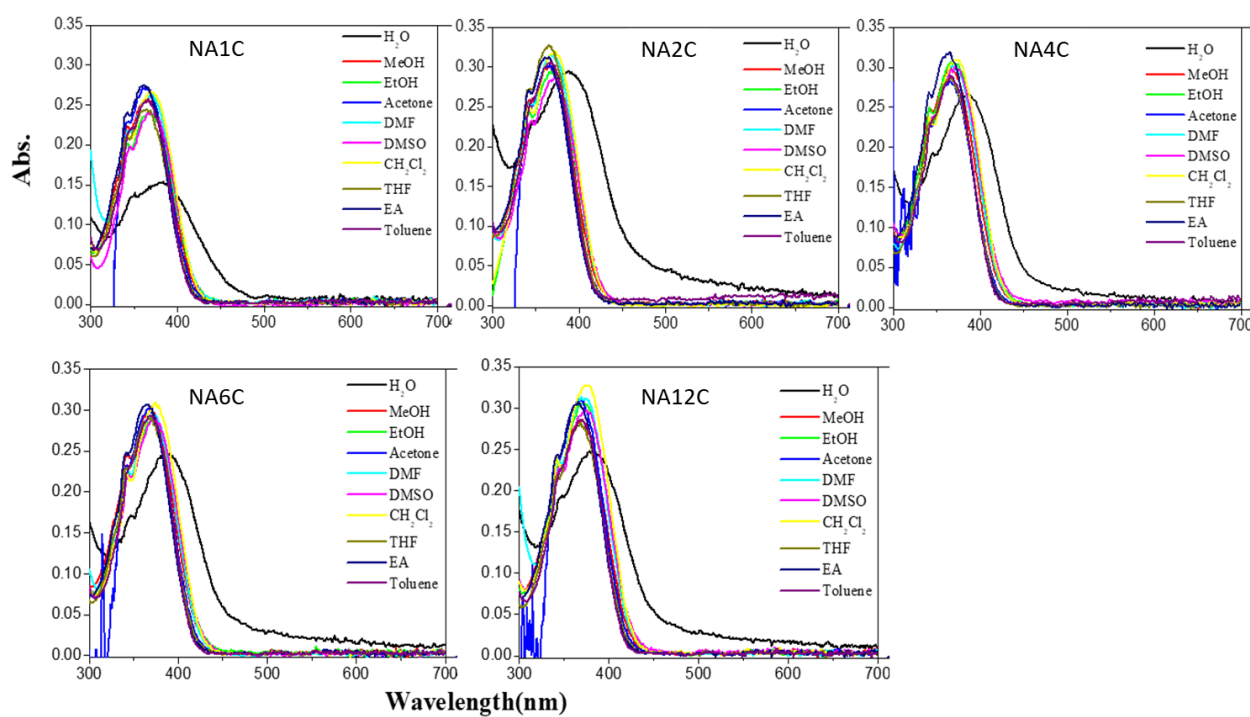
yield is about 70%.  $^1\text{H}$ NMR (400 Hz,  $\text{CDCl}_3$ ):  $\delta$ = 7.74 (d,  $J$  = 9.2 Hz, 2H), 6.88 (d,  $J$  = 9.2 Hz, 2H), 4.06 (q,  $J$  = 7.0 Hz, 2H), 1.41 (t,  $J$  = 7.0 Hz, 3H), 1.35(s, 12H) ppm.

**2-(4-butoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane(4b):** Purge the high-pressure bottle by nitrogen for about 1 minute, then put  $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$  (30 mg) in the high-pressure bottle, and then mix **4a** (0.50 g, 2.5 mmol) and the mixed solvent  $\text{Et}_3\text{N}/1,4\text{-dioxane}$  = 1/3 (v/v) of 12 mL into it and stir evenly with a magnet. Finally, add pinacoborane (630  $\mu\text{L}$ , 3.75 mmol) and bubble with nitrogen for about 2 minutes. After refluxing for 1 day, the mixture was cooled down to room temperature. Extracted twice with  $\text{CH}_2\text{Cl}_2/\text{H}_2\text{O}$ , and the organic layer was dried over anhydrous magnesium sulfate ( $\text{MgSO}_4$ ). Purify via silica column chromatography (the eluent was n-hexane) to obtain transparent liquid. The yield is about 70%.  $^1\text{H}$ NMR (400 Hz,  $\text{CDCl}_3$ ):  $\delta$ = 7.74 (d,  $J$  = 9.2 Hz, 2H), 6.88 (d,  $J$  = 9.2 Hz, 2H), 3.98 (t,  $J$  = 7.0 Hz, 2H), 1.75 (m, 2H), 1.48(m, 2H), 0.97 (t,  $J$  = 7.0 Hz, 3H) ,1.33 (s, 12H) ppm.

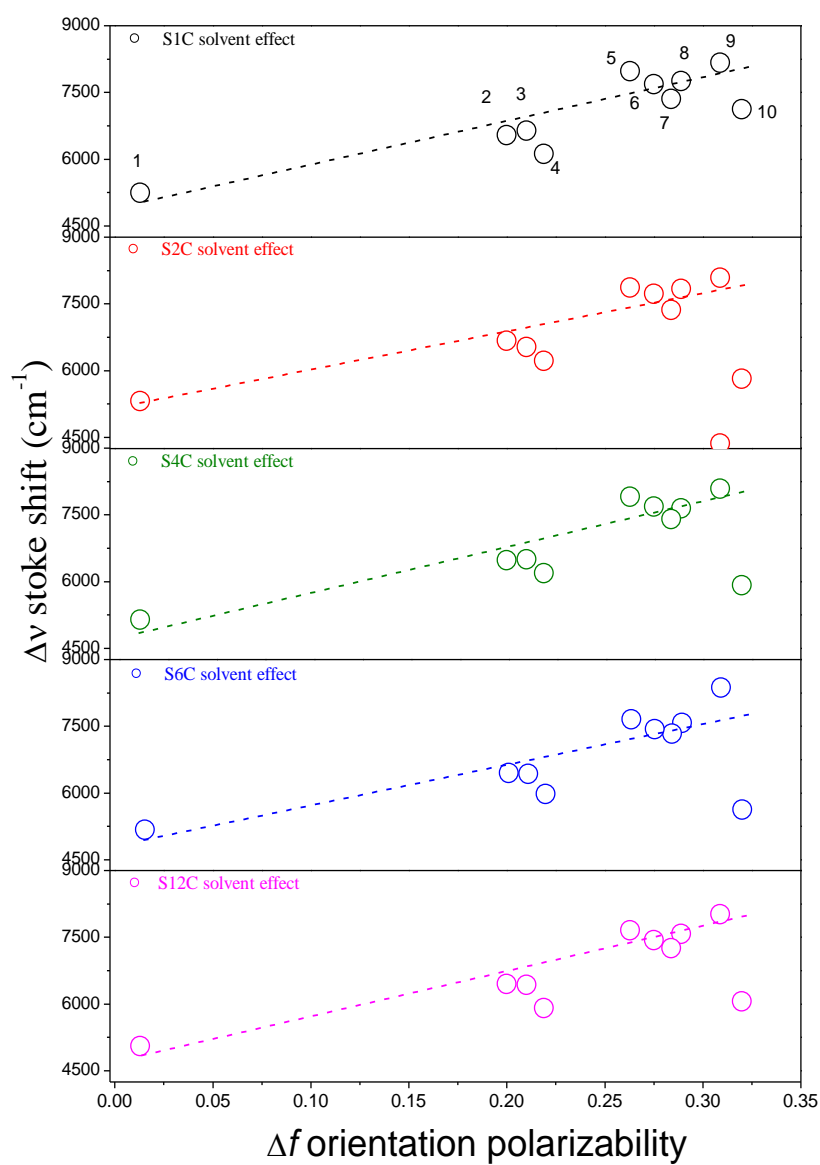
**2-(4-(hexyloxy)phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane(6b):** Purge the high-pressure bottle by nitrogen for about 1 minute, then put  $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$  (30 mg) in the high-pressure bottle, and then mix **6a** (0.50 g, 2.5 mmol) and the mixed solvent  $\text{Et}_3\text{N}/1,4\text{-dioxane}$  = 1/3 (v/v) of 12 mL into it and stir evenly with a magnet. Finally, add pinacoborane (630  $\mu\text{L}$ , 3.75 mmol) and bubble with nitrogen for about 2 minutes. After refluxing for 1 day, the mixture was cooled down to room temperature. Extracted twice with  $\text{CH}_2\text{Cl}_2/\text{H}_2\text{O}$ , and the organic layer was dried over anhydrous magnesium sulfate ( $\text{MgSO}_4$ ). Purify via silica column chromatography (the eluent was n-hexane) to obtain transparent liquid. The yield is about 70%.  $^1\text{H}$ NMR (400 Hz,  $\text{CDCl}_3$ ):  $\delta$ = 7.74 (d,  $J$  = 9.2 Hz, 2H), 6.89 (d,  $J$  = 9.2 Hz, 2H), 3.97 (t,  $J$  = 7.0 Hz, 2H), 1.78 (m, 2H), 1.44(m, 2H), 1.36(m, 4H), 1.34(s, 12H), 0.92 (t,  $J$  = 7.0 Hz, 3H) ppm.

**2-(4-(dodecyloxy)phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane(12b):** Purge the high-pressure bottle by nitrogen for about 1 minute, then put  $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$  (30 mg) in the high-pressure bottle, and then mix **12a** (0.50 g, 2.5 mmol) and the mixed solvent  $\text{Et}_3\text{N}/1,4\text{-dioxane}$  = 1/3 (v/v) of 12 mL into it and stir evenly with a magnet. Finally, add pinacoborane

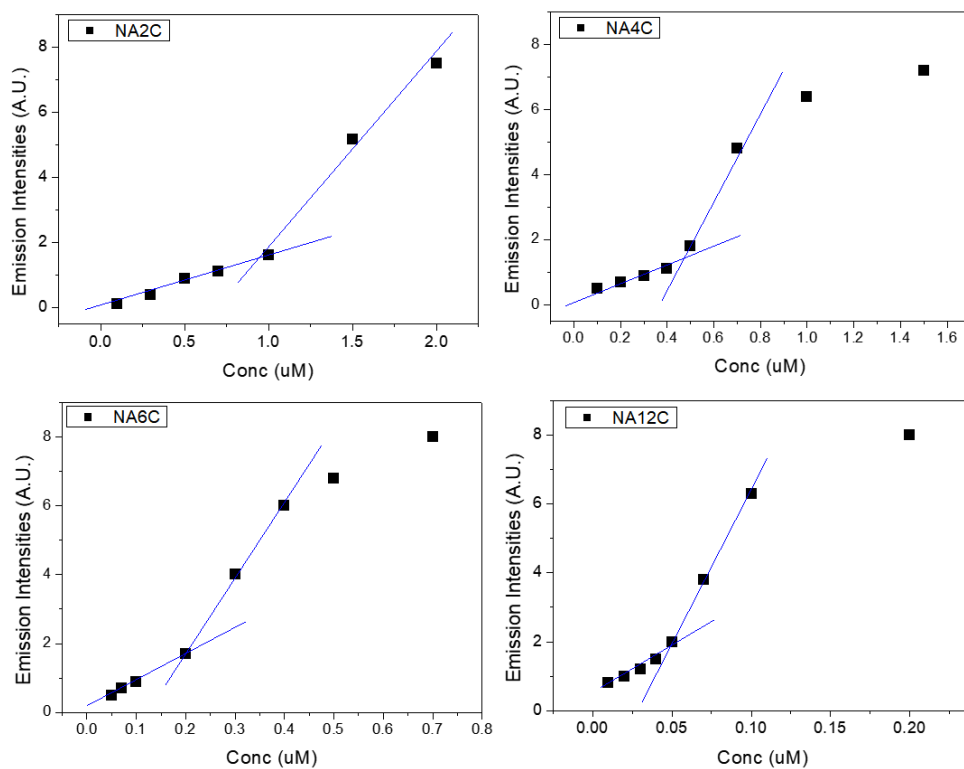
(630  $\mu$ L, 3.75 mmol) and bubble with nitrogen for about 2 minutes. After refluxing for 1 day, the mixture was cooled down to room temperature. Extracted twice with  $\text{CH}_2\text{Cl}_2/\text{H}_2\text{O}$ , and the organic layer was dried over anhydrous magnesium sulfate ( $\text{MgSO}_4$ ). Purify via silica column chromatography (the eluent was n-hexane) to obtain transparent liquid. The yield is about 70%.  $^1\text{H}$ NMR (400 Hz,  $\text{CDCl}_3$ ):  $\delta$ = 7.73 (d, 18 J = 9.2 Hz, 2H), 6.88 (d, J = 9.2 Hz, 2H), 3.97 (t, J = 7.0 Hz, 2H), 1.77 (m, 2H), 1.43(m, 2H), 1.34(s, 12H), 1.26(m, 16H), 0.88(t, J = 7.0Hz, 3H) ppm.



**Figure S1:** shows the absorption spectrum of NA1C, NA2C, NA4C, NA6C, and NA12C (20  $\mu\text{M}$ ) in different solvents (H<sub>2</sub>O, MeOH, EtOH, Acetone, DMF, DMSO, CH<sub>2</sub>Cl<sub>2</sub>, THF, EA, and Toluene)

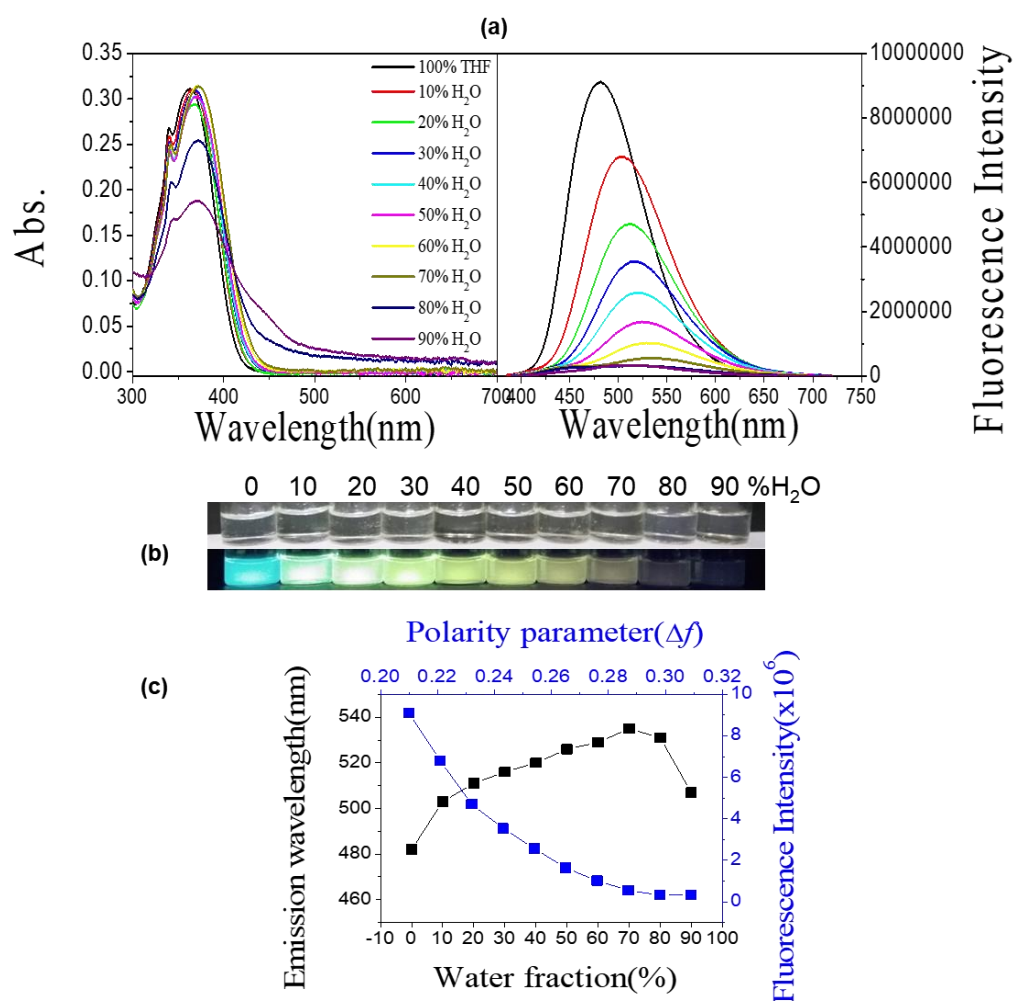


**Figure S2:** Lippert plots of compounds NAXC by solvent effect according to the solvent polarity parameter (1: Toluene, 2: EA, 3: THF, 4:  $\text{CH}_2\text{Cl}_2$ , 5: DMSO, 6: DMF, 7: Acetone, 8: EtOH, 9: MeOH, 10:  $\text{H}_2\text{O}$ )

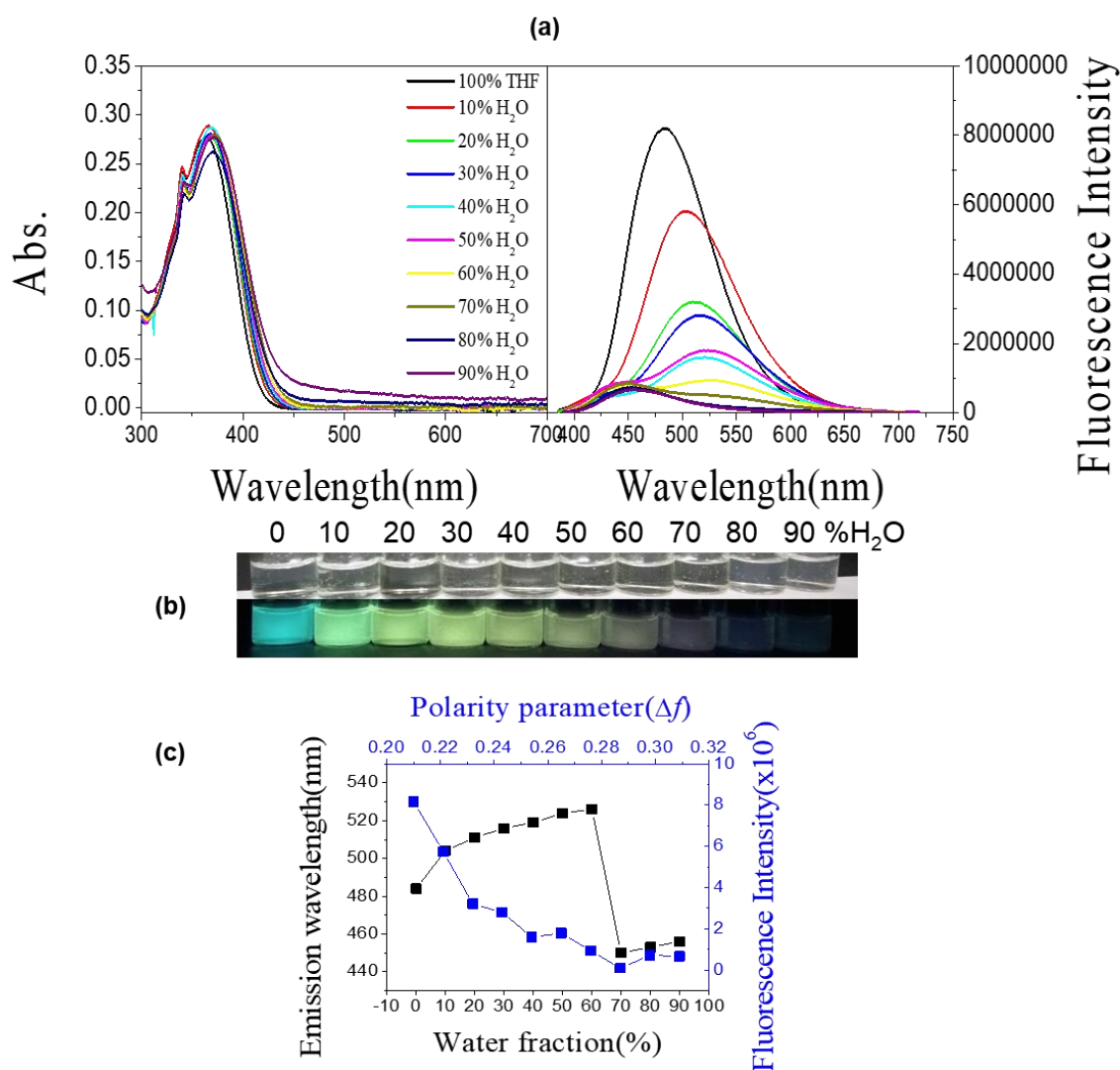


**Figure S3.** shows the critical microcapsule concentrations of compounds NA2C, NA4C, NA6C, and NA12C in H<sub>2</sub>O.

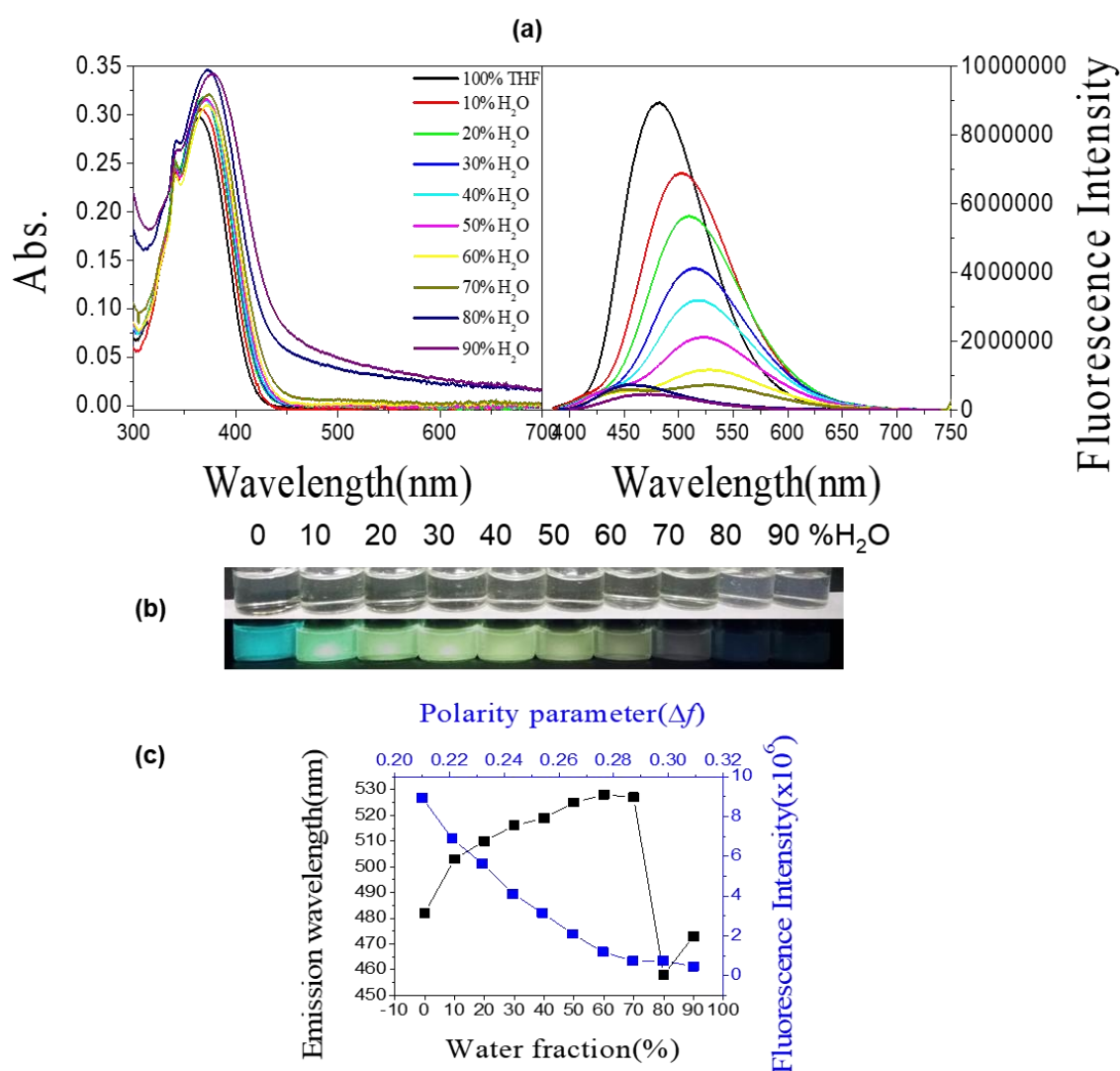




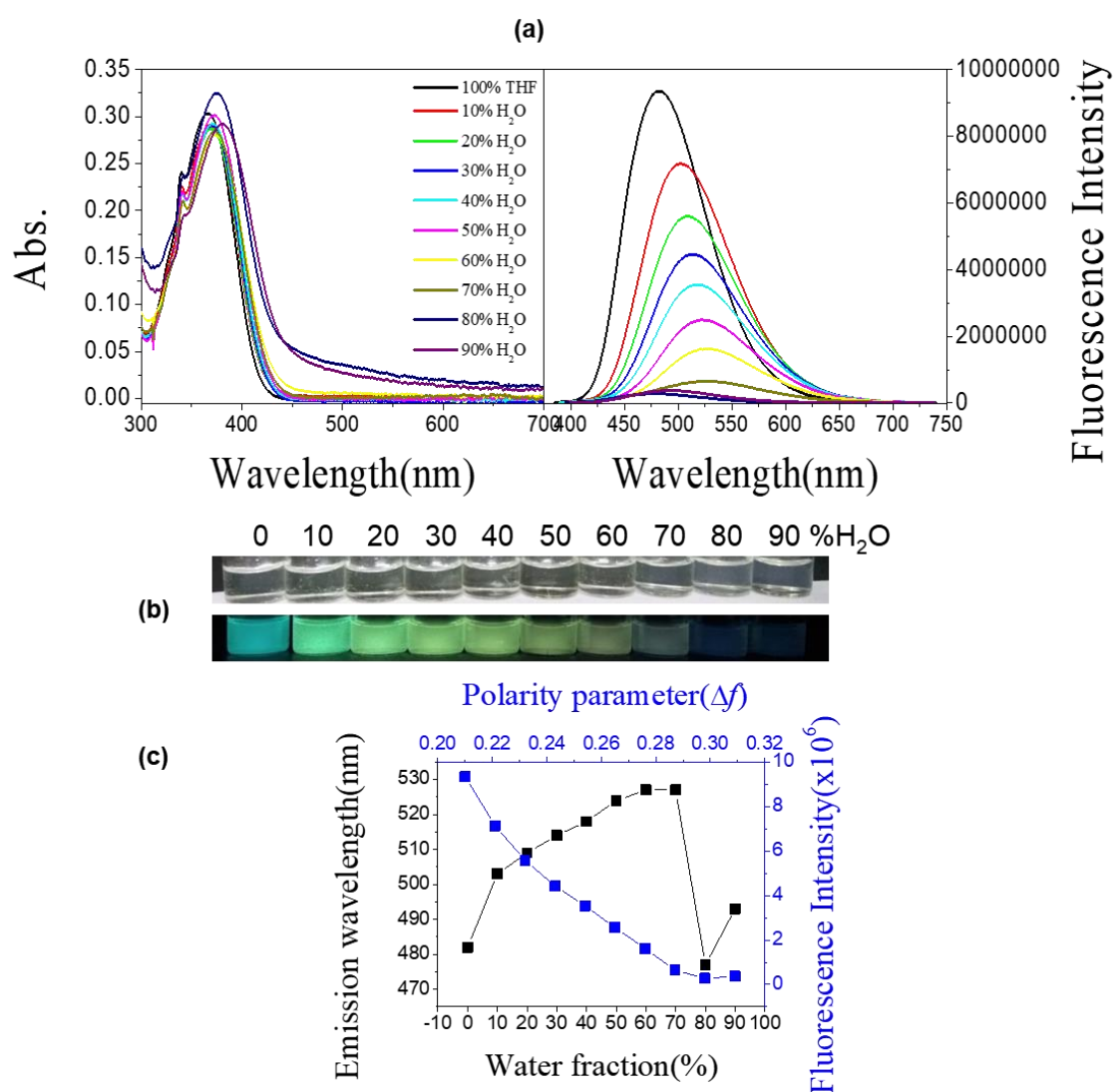
**Figure S4.** shows the (a) absorption and fluorescence spectra of 20  $\mu\text{M}$  NA1C a mixture solvent THF/ $\text{H}_2\text{O}$ . (b) images show the color change of the solutions under UV lamp (light source wavelength = 365 nm). (c) Plot showing the relation between the water fractions, emission wavelengths and intensities, and polarities from (a).



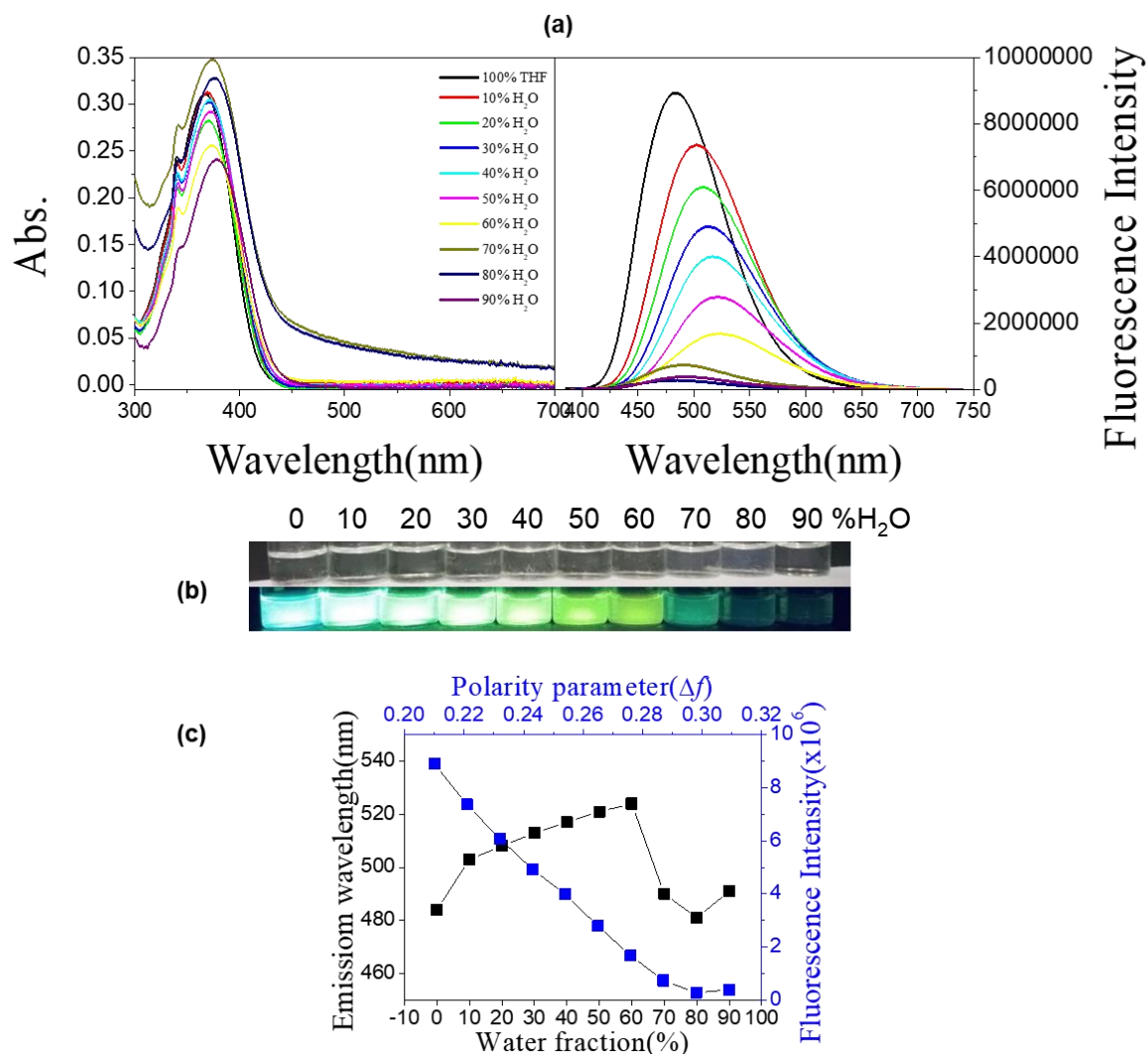
**Figure S5.** shows the (a) absorption and fluorescence spectra of 20  $\mu$ M NA2C a mixture solvent THF/H<sub>2</sub>O. (b) images show the color change of the solutions under UV lamp (light source wavelength = 365nm). (c) Plot showing the relation between the water fractions, emission wavelengths and intensities, and polarities from (a).



**Figure S6.** shows the (a) absorption and fluorescence spectra of 20  $\mu$ M NA4C a mixture solvent THF/H<sub>2</sub>O. (b) images show the color change of the solutions under UV lamp (light source wavelength = 365 nm). (c) Plot showing the relation between the water fractions, emission wavelengths and intensities, and polarities from (a).



**Figure S7.** shows the (a) absorption and fluorescence spectra of 20  $\mu$ M NA6C a mixture solvent THF/H<sub>2</sub>O. (b) images show the color change of the solutions under UV lamb (light source wavelength = 365nm). (c) Plot showing the relation between the water fractions, emission wavelengths and intensities, and polarities from (a).



**Figure S8.** shows the (a) absorption and fluorescence spectra of 20  $\mu$ M NA12C a mixture solvent THF/H<sub>2</sub>O. (b) images show the color change of the solutions under UV lamp (light source wavelength = 365 nm). (c) Plot showing the relation between the water fractions, emission wavelengths and intensities, and polarities from (a).

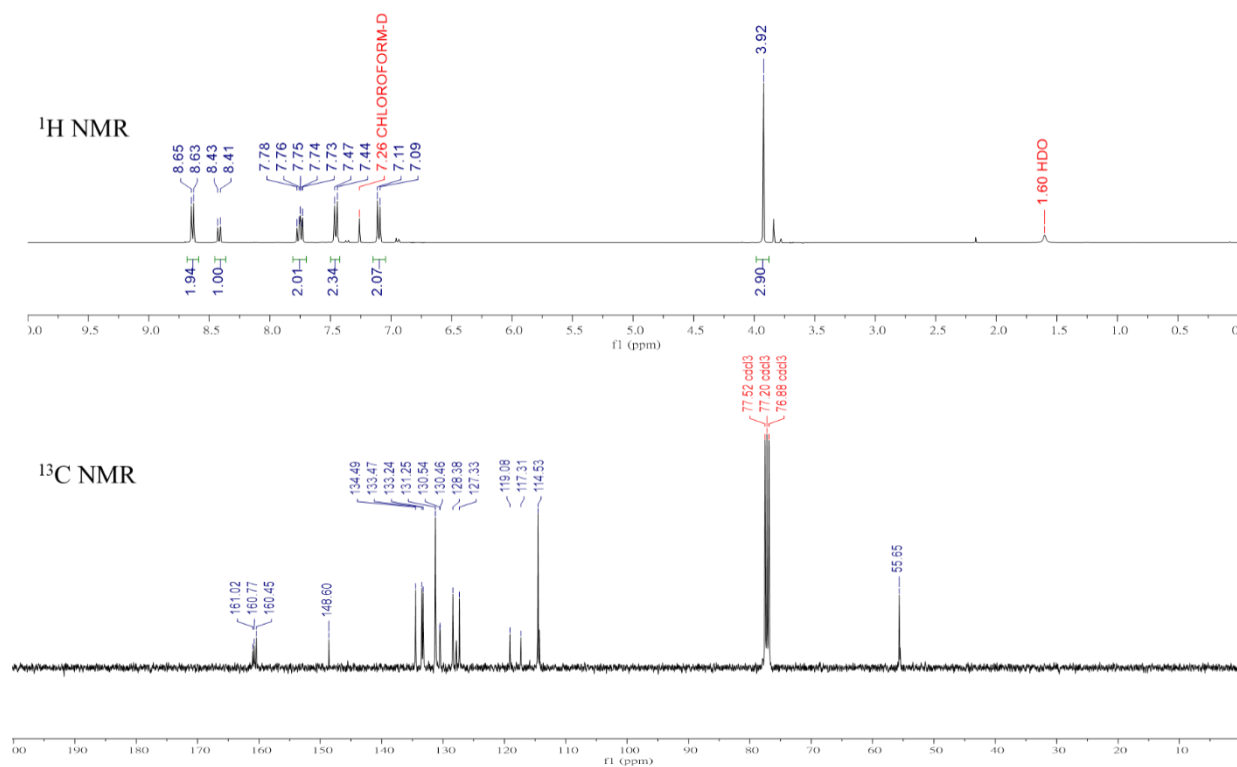


Figure S9. 400 MHz  $^1\text{H}$  NMR spectrum (up) and  $^{13}\text{C}$  NMR spectrum (down) in  $\text{CDCl}_3$  of NA1C.

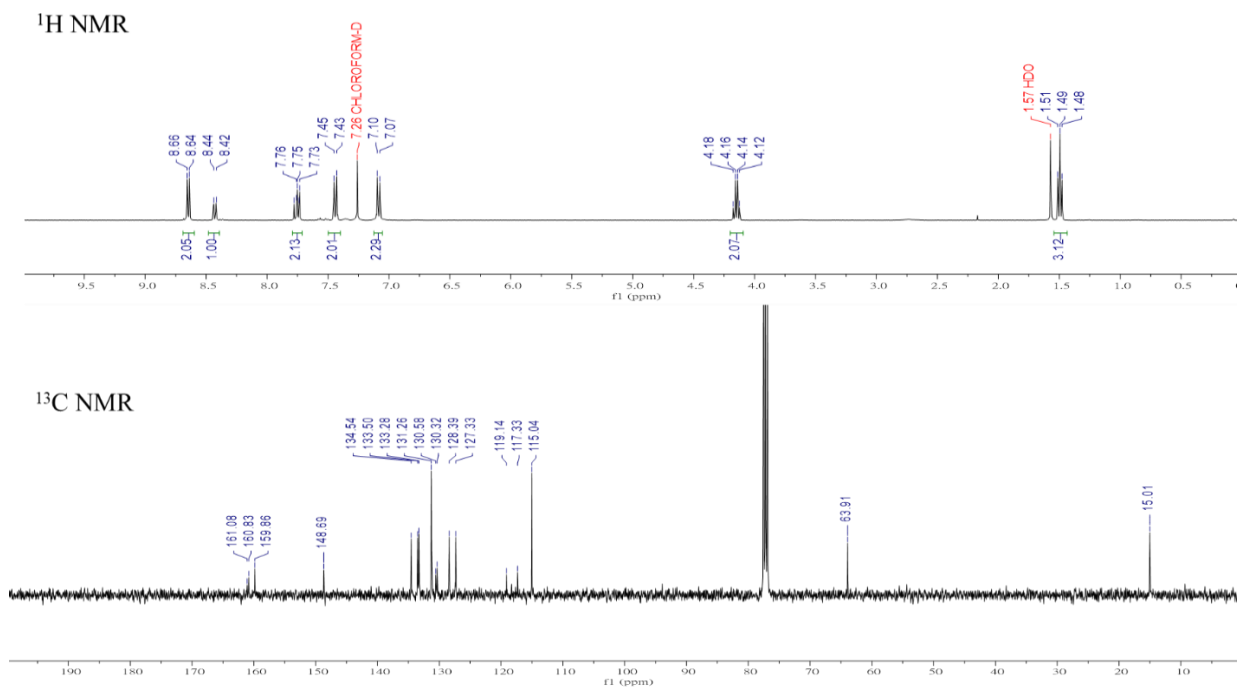
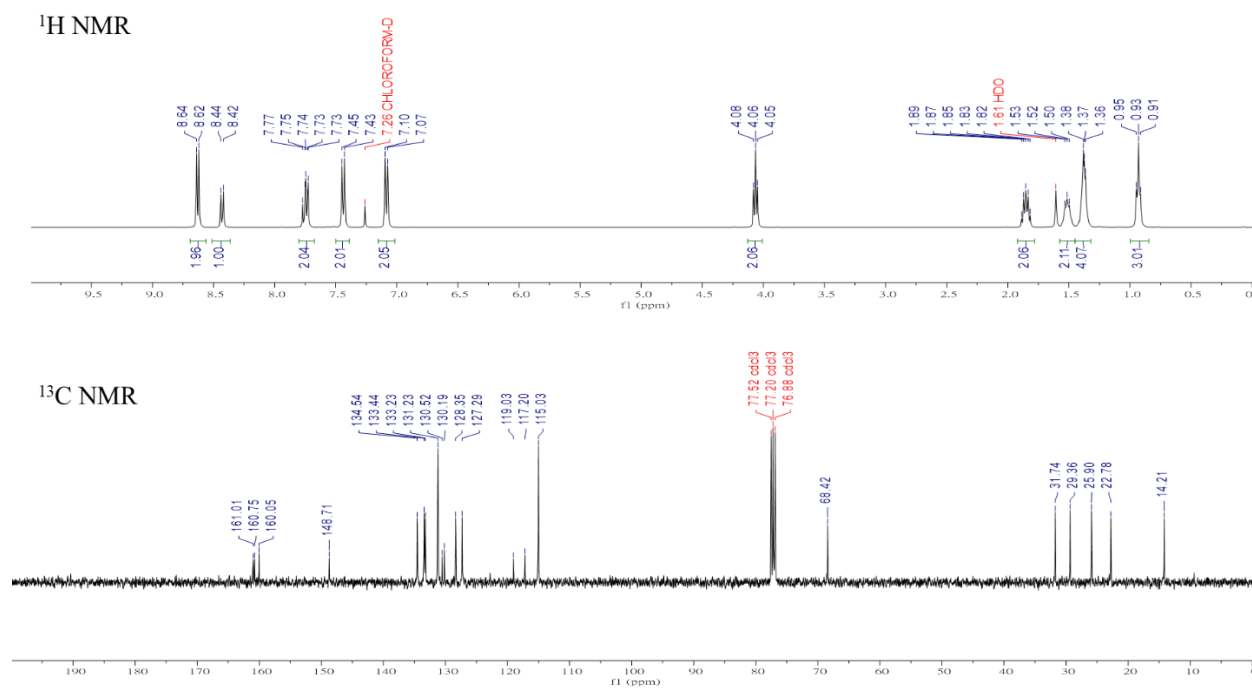
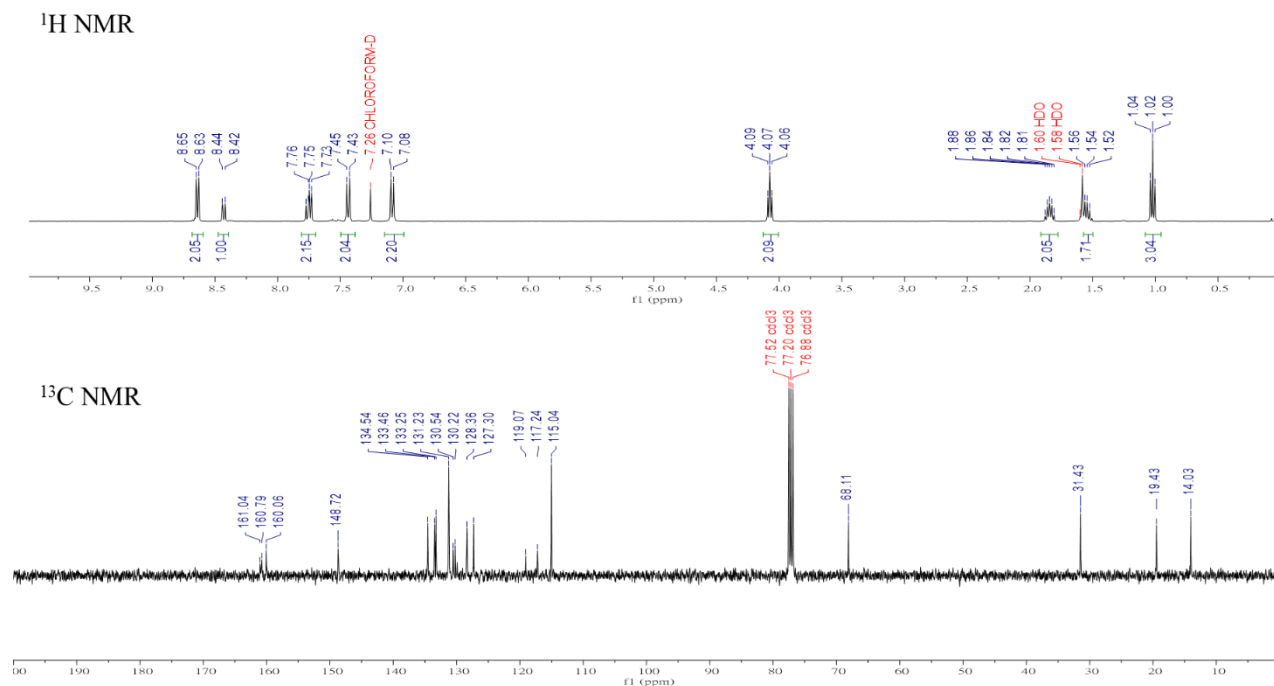


Figure S10. 400 MHz  $^1\text{H}$  NMR spectrum (up) and  $^{13}\text{C}$  NMR spectrum (down) in  $\text{CDCl}_3$  of NA2C.



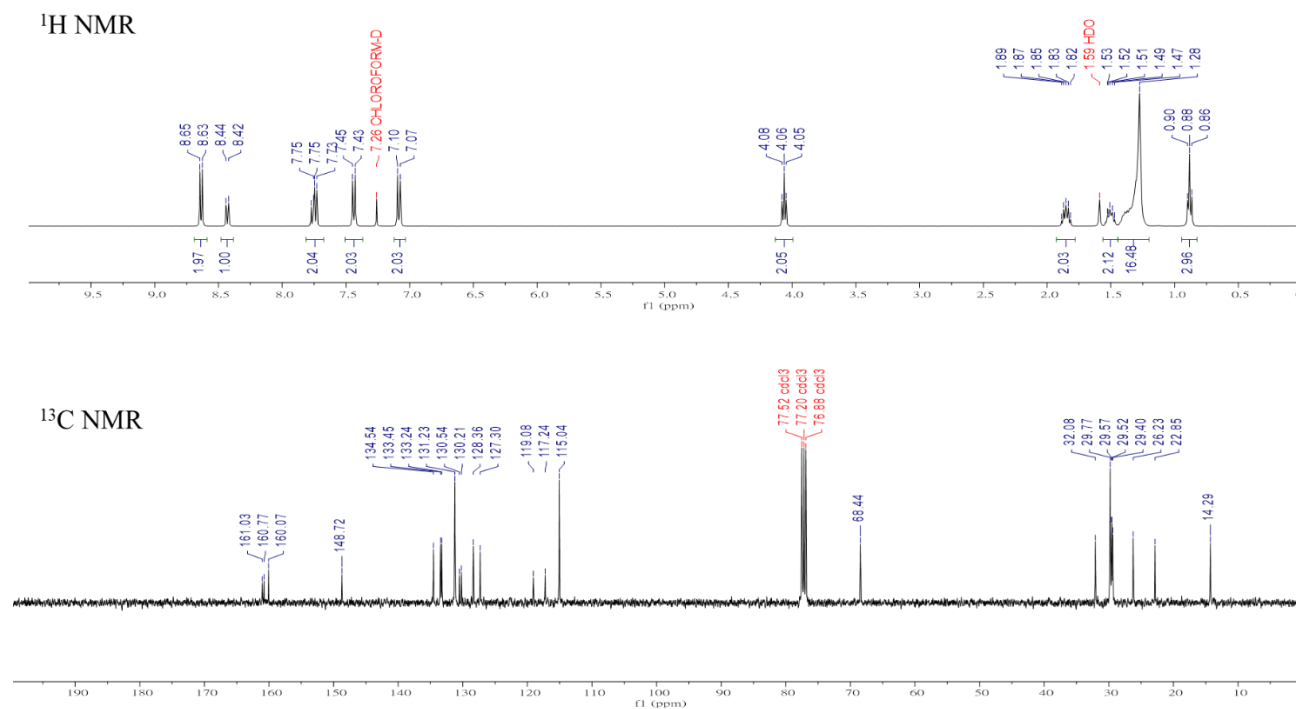


Figure S13. 400 MHz <sup>1</sup>H NMR spectrum (up) and <sup>13</sup>C NMR spectrum (down) in CDCl<sub>3</sub> of NA12C.

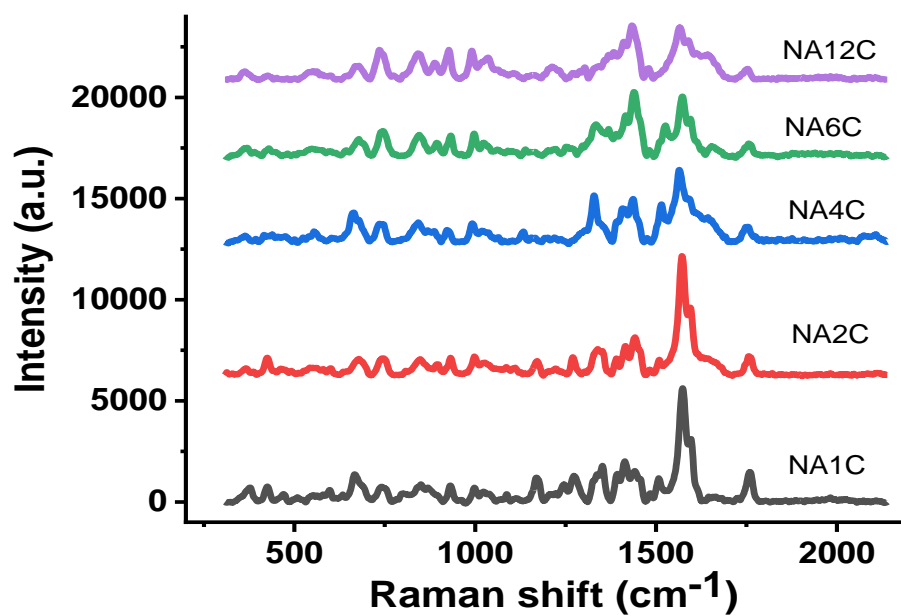


Figure S14. The Raman spectra of NAXC. Excited wavelength: 633 nm laser (power: 0.35 mW), exposure time: 1s, accumulation number: 20 time