

## Supplementary material

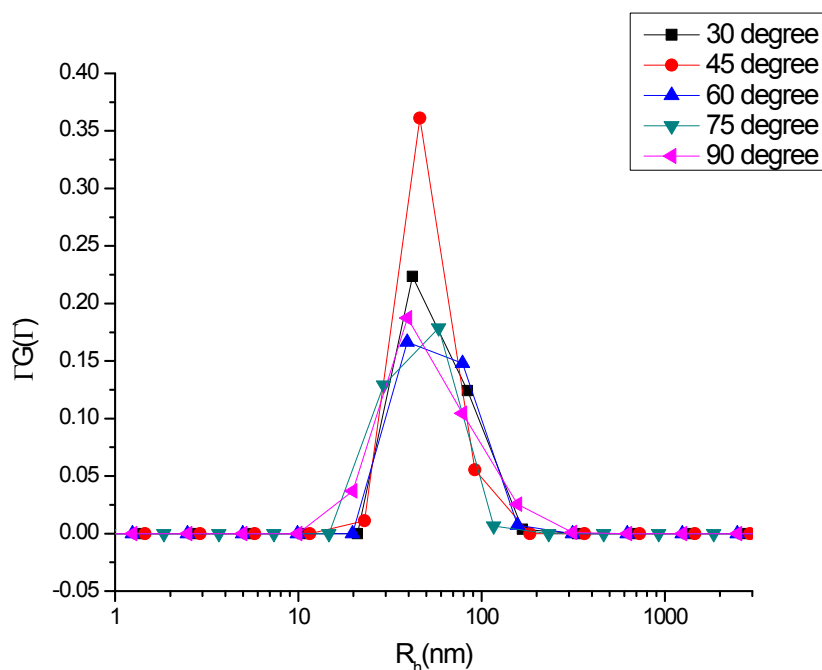
# Self-assembly of triangular polyoxometalate-organic hybrid macroions in mixed solvents

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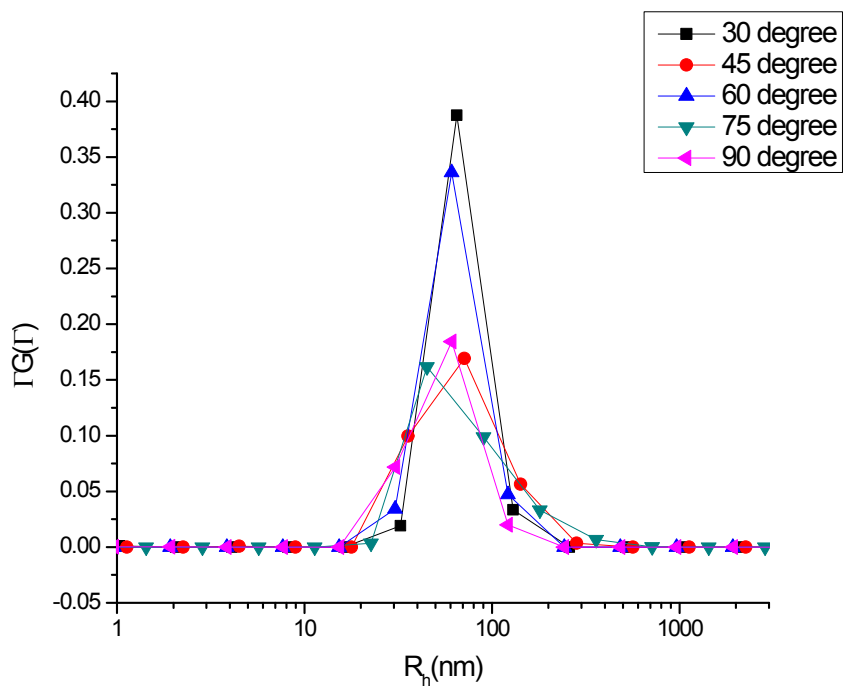
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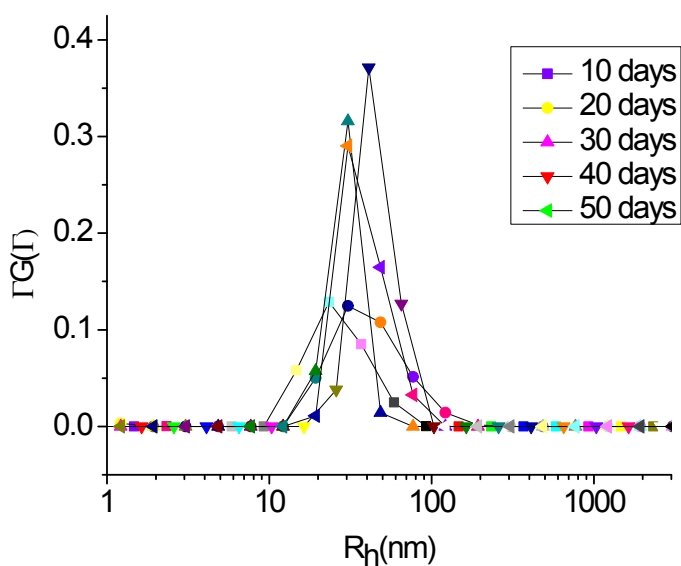
1. CONTIN analysis showing the change of the  $R_h$  distribution of vesicles from hybrid **1** in acetone/water mixed solvent containing 90 vol% acetone at different angle.



2. CONTIN analysis showing the change of the  $R_h$  distribution of vesicles from hybrid **2** in acetone/water mixed solvent containing 90 vol% acetone at different angle.



3. CONTIN analysis showing the change of the  $R_h$  distribution of vesicles from hybrid **1** in acetone/water mixed solvent containing 90 vol% acetone at different time.



4. For a typical experiment, 5 mg of hybrid **1** or **2** was dissolved in 2 mL of acetonitrile. This solution was then applied to a prepacked, cation-exchange resin column (Amberjet 1200 hydrogen form purchased from Sigma-Aldrich) then rinsed with D.I. water and acetonitrile. An

additional 20-50 mL of acetonitrile was used to elute the column, and the yellow fraction was collected. The post elution of the hybrid macroanions sample was further washed with 5 mL of diethyl ether to remove organic impurities. The final solution was transferred into a glass culture plate and kept in the dark for several days for the solvent to fully evaporate. A fine yellow-colored powder was collected and could be easily dissolved in water for further characterizations.