Supporting Information

## **Uncovering Three-body Competition of Chain Growth, Degradation**

## and Re-aggregation for Polyphenylacetylenes During Polymerization

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## Shear Effects of PPA in Solution: A Comparison of SEC Curves.

In the experiment, under a nitrogen atmosphere, identical magnetic stir bars were introduced into two 30 mL Schlenk flasks, each containing 20 mL of a toluene solution of PPA. One flask was subjected to stirring at a rate of 600 rad/min, while the other flask was kept stationary. Subsequently, samples were collected at various time intervals for analysis, in order to compare the variations in the SEC curves of PPA in toluene under stirred and unstirred conditions.



**Figure S1.** Comparison of SEC curves of PPA over time under stirring and non-stirring conditions in toluene.



**Figure S2.** Solution concentration (C) dependence of the voltage signal (V) measured at T = 298.1 K for (a) NaCl aqueous solution, (b) PPA Tol solution, (c) PPA CHCl<sub>3</sub> solution and (d) PPA THF solution.



Figure S3. The retention time  $(V_{retention})$  dependence of molar mass for narrowdistribution polystyrene standards in THF in size exclusion chromatography (SEC) measurement.



**Figure S4.** curves measured at different polymerization times for the mixed solutions in the PPA polymerization systems under different conditions of solvent and atmosphere.



**Figure S4.** Polymerization time dependence of apparent weight-average molecular weight  $(M_{w,app})$  for PPA in different polymerization systems: (a) N<sub>2</sub> atmosphere and (b) air atmosphere.



**Figure S5**. (a-c) Ultraviolet-visible absorption (UV-Vis) spectra for PPA-N<sub>2</sub> system measured at different polymerization times under different solution. (d-f) UV-Vis spectra for PPA-N<sub>2</sub> system measured at different polymerization times under different solution.