

# Helical bias in supramolecular polymers accounts for different stabilities of kinetically trapped states

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## RESEARCH ARTICLE

# Helical bias in supramolecular polymers accounts for different stabilities of kinetically trapped states

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**Abstract**

The idea to synthesize and self-assemble nano-graphenes with structural precision into supramolecular polymers is just one of Klaus Müllen's many pioneering contributions to the chemical sciences. To honor his impact in the field of polymer science, we here describe a study that combines experimental and computational methods in studying the stability of kinetically trapped states of supramolecular polymers. We show that the introduction of stereocenters in the sidechains allow helical supramolecular polymers based on chiral triphenylene-2,6,10-tricarboxamide monomers to escape a kinetic trap more efficiently than polymers based on their achiral analogs. Partial depolymerization of the kinetically trapped state by increasing the temperature followed by polymerization by lowering the temperature shows that monomers either polymerize on existing stacks or self-nucleate to form the thermodynamically more stable state. Chiral monomers prefer the latter more than achiral monomers.

**KEYWORDS**

chirality, kinetically trapped states, mechanism of polymerization, supramolecular polymers

## 1 | INTRODUCTION

Extensive experimental,<sup>1,2</sup> theoretical,<sup>3–9</sup> and computational<sup>10–13</sup> investigations in the field of supramolecular (co)polymerizations made it possible to unveil

many intricate thermodynamic properties, such as elongation temperature,<sup>14</sup> nucleus size,<sup>15</sup> and cooperativity.<sup>16</sup> These properties are providing detailed information about many aspects of these supramolecular (co)polymers, including the mechanism of their formation, the stability, and the length of the (co)polymers made. Moreover, different kinetic aspects of supramolecular polymerizations have been studied,<sup>16–21</sup> and were found to share many similarities with extensively studied protein fibrilization processes.<sup>22–25</sup> In spite of all the progress

Marcin L. Ślęczkowski and Mathijs F. J. Mabesoone contributed equally to this study.

Dedicated to Professor Klaus Müllen on the occasion of his 75<sup>th</sup> birthday.

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made, many questions remain to be answered: do similar thermodynamic properties of chiral and achiral monomers translate into similar kinetics of the polymerization-depolymerization processes? What is the effect of small changes in the molecular substitution pattern on the kinetics? How do competitive interactions affect the rate of (dis)assembly?

One of the most common methods to control the dynamic behavior in supramolecular polymerizations is achieved by the choice of solvents.<sup>26</sup> When a supramolecular polymer is formed in a poor solvent through a slow heating-cooling sequence and the kinetics of the assembly process at room temperature are slow, the addition of a good solvent to the system will increase the dynamics at room temperature.<sup>16</sup> Therefore addition of a good solvent decreases the chance of being trapped in kinetically determined states. When too much good solvent is added and the monomeric state is stabilized by the good solvent, no polymers are formed. Good solvents not only increase the reaction rates of all processes, they also influence the energy landscape of the polymerization.<sup>27–30</sup> These changes in energy landscape can lead to a destabilization of thermodynamically controlled supramolecular polymers or to the formation of different aggregate morphologies.<sup>31–34</sup> The increased dynamic exchange of monomers between polymers induced by good solvents is often exploited to efficiently generate thermodynamically stable structures, but the right choice of solvents has seldomly been used to non-covalently synthesize kinetically trapped structures in a controlled fashion.<sup>35</sup>

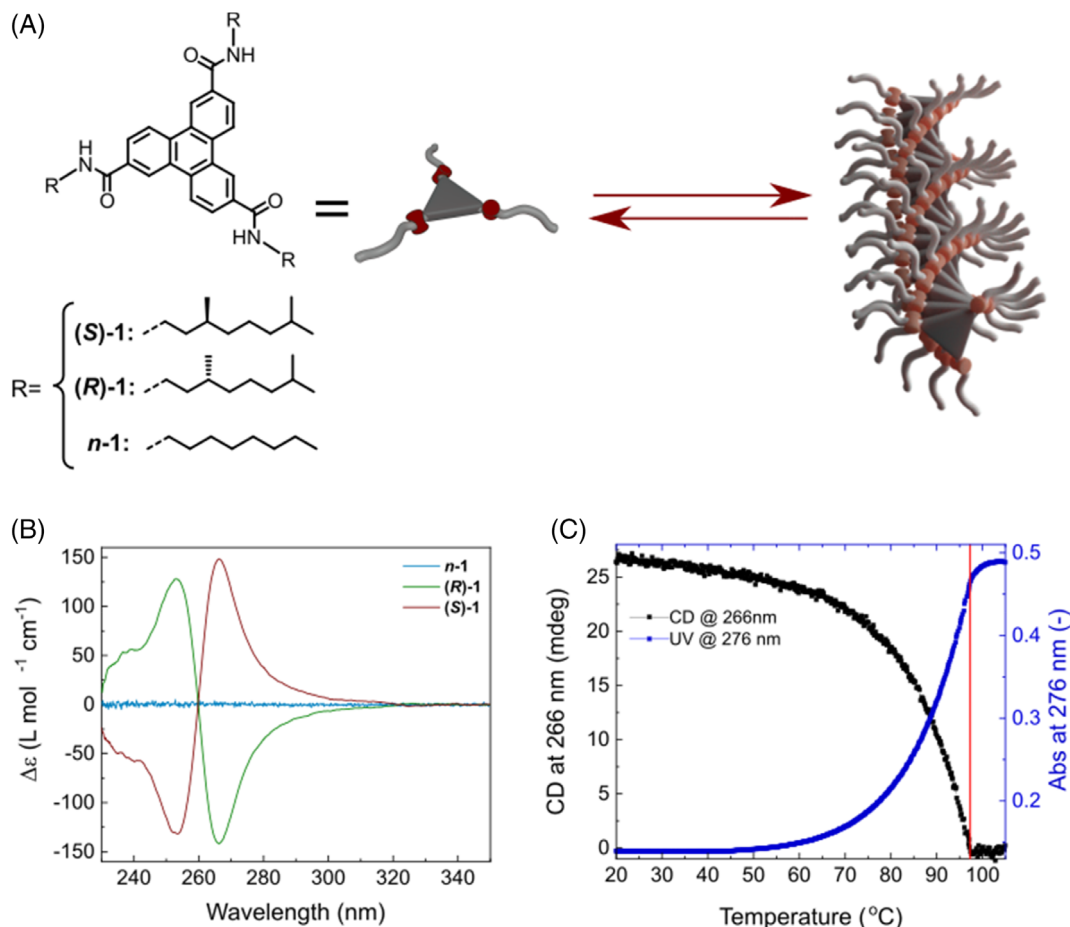
We recently reported the thermodynamically controlled supramolecular polymerization of a series of triphenylene-2,6,10-tricarboxamides (TTAs - **1**) in chlorinated solvents such as 1-chlorooctane.<sup>36</sup> By using the chiral monomer (**R**)-**1**, a strong bias for one handedness of the helical polymers is observed. Copolymerizations of (**R**)-**1** with **n**-**1** in the same solvent showed a strong “sergeant-and-soldiers” (SaS) effect,<sup>37</sup> in which the chiral monomer in small amounts can bias the helicity of all polymers, which are mainly constructed from achiral monomers. Similar preferred helical bias in polymers of achiral **n**-**1** can be introduced using chiral solvents, like (*S*)-1-chloro-2-methylbutane ((*S*)-ClMeBu).<sup>36</sup> In the current paper, we capture the chiral polymers of TTAs in kinetically trapped states and follow the depolymerization-polymerization processes by heating-cooling cycles that form the thermodynamically stable states over time. We show that differences in equilibration kinetics between chiral and achiral TTAs do not imply intrinsically different kinetic parameters of the polymerizations. Rather, the difference originates from the thermodynamic preferences of a chiral monomer to polymerize with single helicity. Using a mathematical model, we corroborate that the presence

of the helical preference in (**R**)-**1** suffices to explain its faster equilibration compared to **n**-**1**.

## 2 | RESULTS AND DISCUSSION

In decalin, all three analogues of **1** create helical supramolecular structures stabilized by threefold hydrogen bonding at room temperature. The UV and CD spectra of **1** in decalin are identical to the spectra observed in 1-chlorooctane, indicating an identical electronic structure of the aggregated triphenylenes (Figure 1B). The mirror image Cotton effects, with extrema at 254 and 266 nm, highlight the opposite helical biases of (**S**)-**1** and (**R**)-**1**. Analogously, the absence of a Cotton effect for **n**-**1** indicates formation of a racemic mixture of *P*- and *M*-helicities. Variable temperature UV (VT-UV) and CD (VT-CD) experiments show that in decalin, the aggregates of **1** are considerably more stable than in chlorinated solvents such as 1-chlorooctane. In decalin at concentrations as low as 5  $\mu\text{M}$ , (**S**)-**1** is molecularly dissolved only above approximately 100°C (Figure 1C). Upon slow cooling (0.5 K  $\text{min}^{-1}$ ) from the molecularly dissolved state, the monomers form thermodynamically controlled, nucleated supramolecular polymers, as indicated by the reversible non-sigmoidal UV and CD cooling curves (Figure 1C) and reproducibly obtained molar ellipticity ( $\Delta\epsilon = 140 \text{ L mol}^{-1} \text{ cm}^{-1}$  at 266 nm). The full thermodynamic characterization of the temperature and solvent-dependent thermodynamic parameters of the polymerization was obtained from fits of mass-balance models to the data and details can be found in the Supplementary Information.

Next, we studied copolymerizations of (**S**)-**1** with **n**-**1** (SaS experiment,<sup>37</sup> Figures 2A and S6a,b) and (**R**)-**1** with (**R**)-**1** (Majority Rules [MR] experiment),<sup>38</sup> Figures 2B and S6c,d) in decalin and in mixtures of 80 vol% decalin with 20 vol%  $\text{CHCl}_3$ . All samples were cooled from the molecularly dissolved state with a cooling rate of 0.5 K  $\text{min}^{-1}$ . In both the SaS and MR experiments, a non-linear dependence of the CD effect on the fraction chiral sergeant or enantiomeric excess (e.e.) is evident in the decalin: $\text{CHCl}_3$  (8:2 v:v) solutions. Saturation of the CD effect, indicative of full helical bias, is attained at around 10% Sergeant in case of the SaS experiment and an e.e. of 15% in the MR experiment in decalin: $\text{CHCl}_3$  (8:2 v:v) solutions. Our two-component mass-balance model<sup>6</sup> satisfactorily describes the MR experiment in the presence of  $\text{CHCl}_3$ , and a mismatch penalty of 0.37 kJ/mol was obtained (Figure S7).<sup>7</sup> In pure decalin, however, an almost linear dependency of the CD effect with the e.e. and fraction of sergeant is observed, which is indicative of a restricted dynamic exchange of monomers between polymers.<sup>35</sup> We confirmed this statement by performing SaS experiments in decalin at variable



**FIGURE 1** (A) Chemical structure of studied triphenylene-2,6,10-tricarboxamides (TTAs) **1** and schematic representation of assembly into helical supramolecular polymers. (B) CD spectra of TTAs **1** in decalin at room temperature corrected for concentration; (C) CD (black) and UV (blue) cooling curves of (**S**)-**1** at  $c = 5 \mu\text{M}$ , cooling rate =  $0.5 \text{ K min}^{-1}$ , the red line indicates elongation temperature  $T_e$

cooling rate, which showed that final helical bias was dependent on the cooling rate (Figure S8).

The kinetic traps that arise in decalin during the supramolecular copolymerization of two monomers raises several fundamental questions: Is the lack of amplification of asymmetry caused by thermodynamic differences in the homo- and hetero-interactions? Or is the difference in behavior driven by different kinetic properties of these two different interactions? Unfortunately, the combinatorial nature of supramolecular copolymerizations prohibits the study of these systems with simple kinetic models. Fortunately, the slow monomer exchange kinetics of **1** in decalin allowed us to study kinetic properties of supramolecular polymerizations of **1** in a different manner.

Over the course of our study of the polymerization of (**R**)-**1**, we noticed that rapid cooling of concentrated ( $300 \mu\text{M}$ ) solutions in decalin from  $170^\circ\text{C}$  produces polymers that show an inversed Cotton effect (Figure 2C). This result implies that the competition between helical states we previously observed in 1-chlorooctane<sup>36</sup> is also present in decalin and that, in contrast to 1-chlorooctane,

the high-temperature state in decalin can be trapped by rapid cooling. Studies on related triphenylamine<sup>39</sup> systems showed that helical inversion may arise from reorganizations of the amide conformation. However, in the case of TTA we attributed the change in helical preference arises from side-chain-solvent interactions.<sup>36</sup> Monitoring the CD effect of trapped (**R**)-**1** in decalin over 2 months shows that the CD effect decreases very slowly from 5 to  $-20$  millidegree at 266 nm (Figure S9a), and the full equilibration to the thermodynamic more stable state requires several months. Similarly, when **n-1** is polymerized in a 1:1 mixture of (*S*)-ClMeBu and decalin, homochiral helical supramolecular polymers are obtained<sup>36</sup> and the helicity of these polymers is retained after the chiral solvent is removed (Figure 2D). The decrease of the CD effect proceeds very slowly, and the kinetically trapped structures are stable over several months (Figure S9b). In other words, we have two different approaches to arrive at kinetically trapped states for (**R**)-**1** and **n-1**, states that show decent stabilities in time at room temperature.

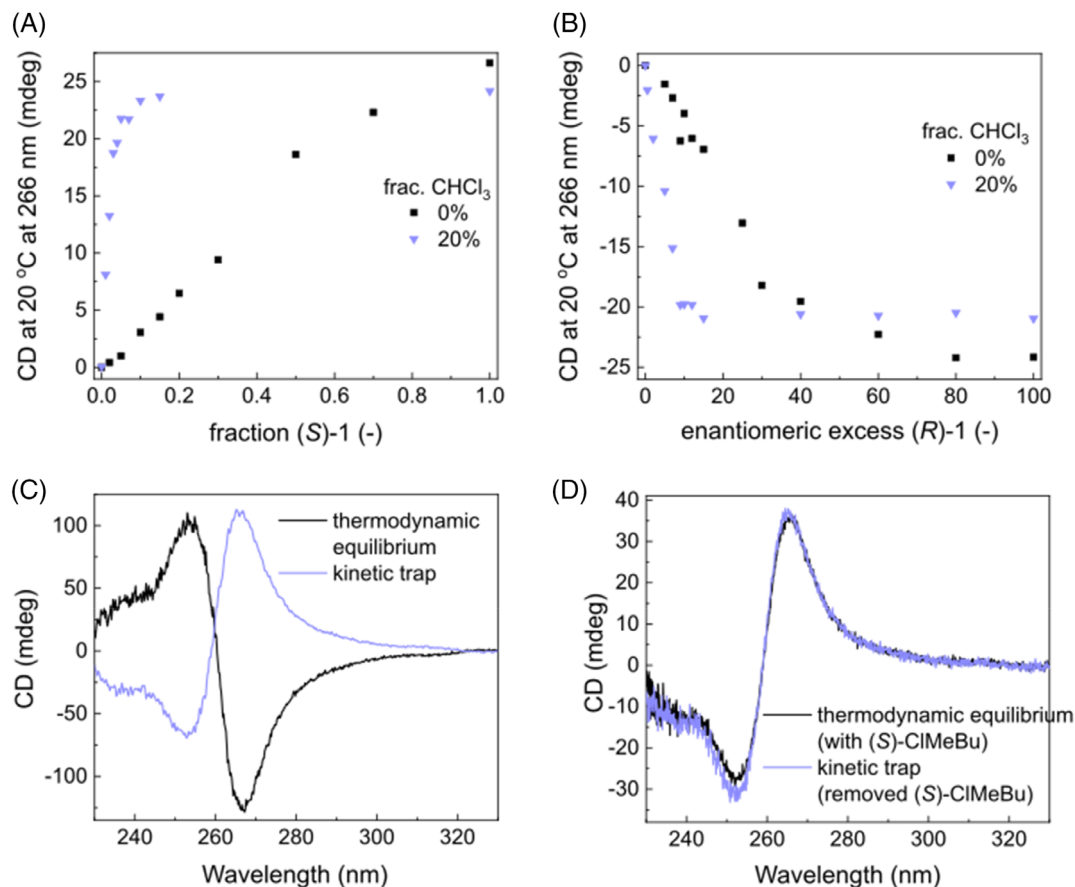
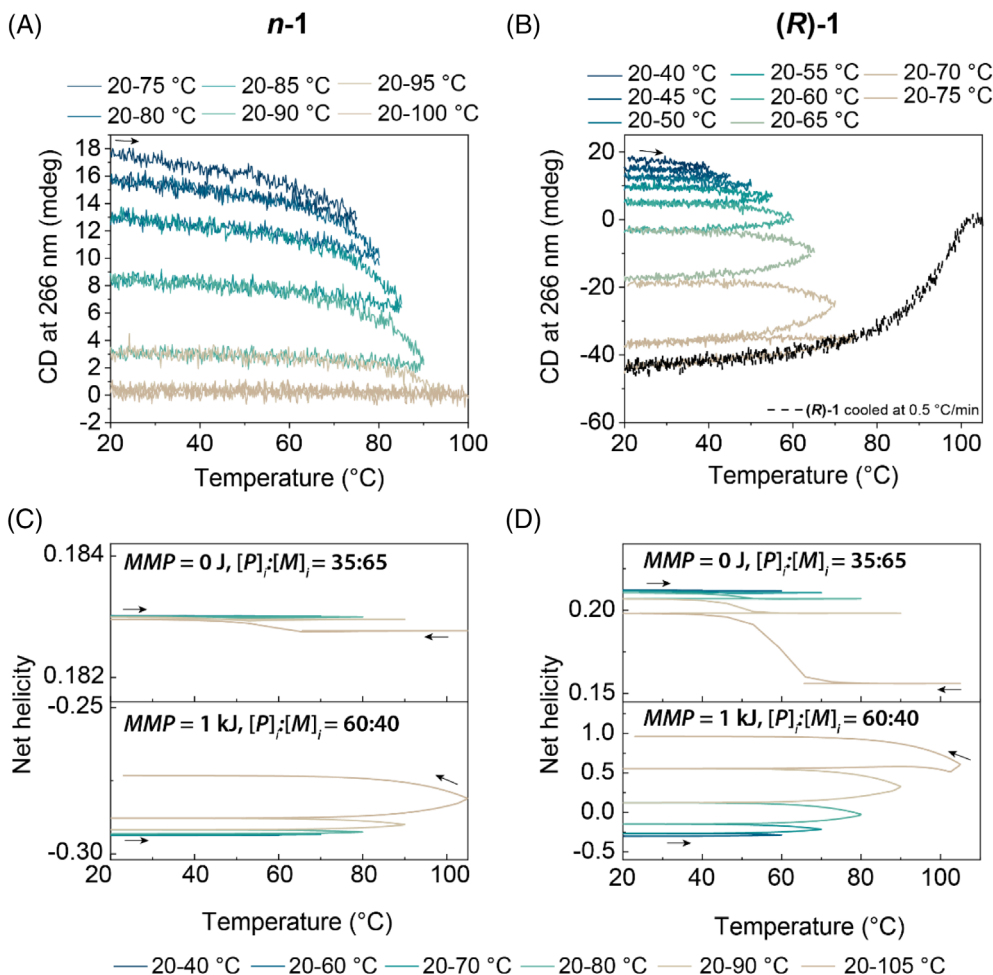


FIGURE 2 (A, B) CD signal at 266 nm at 20°C of samples with a total monomer concentration of 7.5  $\mu\text{M}$  and various fractions of **(S)-1** copolymerized with (A) **n-1** or (B) **(R)-1**; (C) CD spectra of a sample of 300  $\mu\text{M}$  **(R)-1** in decalin after slow and fast cooling; (D) CD spectra of 7.5  $\mu\text{M}$  **n-1** solutions in decalin before and after removal of the **(S)-ClMeBu** cosolvent

Next, we investigated fundamental properties of the equilibration process of the kinetically trapped solutions of **(R)-1** and **n-1** with VT-CD experiments. We monitor the CD intensity of these samples during heating from room temperature to increasingly higher temperatures and cooling to room temperature in consecutive heating-cooling cycles (Figure 3). For this, we first prepared a sample of kinetically trapped polymers of **n-1** by evaporating volatile **(S)-ClMeBu** from a 1:1 **(S)-ClMeBu**: decalin mixture. The CD intensity was recorded during consecutive heating-cooling cycles from 20°C to 75, 80, 85, 90, and 95°C of samples of **n-1**. The results show that in the cooling runs, the CD intensity only partially recovers from the heating-induced partial depolymerization of the trapped states and in the last cycle reaching 95°C the system is fully racemized. The results are explained as follows. Upon heating, the kinetically trapped polymers depolymerize partly at higher temperatures, increasing the fraction of free monomers in solution. Upon cooling, the CD intensity changes only slightly with every heating cycle, even when the temperature approaches the elongation temperature

(Figure 3A). This slow decrease in CD intensity indicates that monomers liberated during the heating cycle can polymerize in two different ways. Either the liberated monomers repolymerize onto already existing, kinetically trapped, polymers or they nucleate into a thermodynamically stable racemic mixture of *P*- and *M*-helices, which does not contribute to the CD intensity. The recovery of a large fraction of the original CD intensity upon cooling during heating-cooling cycles to the temperatures 75–85°C indicates that at these temperatures, the former process is dominant. As expected, the latter process becomes more dominant at higher temperatures when more monomers are present, indicated by the gradual decrease of the CD intensity after the heating-cooling cycles at the highest temperatures. Nonetheless, only in the final heating cycle, which heats the sample to just below the elongation temperature (95°C), full thermodynamic equilibrium is reobtained, as indicated by the absence of CD intensity in the cooling trace. Thereby, the consecutive heating-cooling runs show that the kinetically trapped polymers have a high thermal stability.





**FIGURE 3** VT-CD curves of consecutive heating–cooling cycles of kinetically trapped (A) 7.5  $\mu\text{M}$  *n*-1 solutions in decalin after evaporation of (*S*)-ClMeBu and readjustment of concentration by compensating the evaporated solvent by dilution with extra decalin and (B) 7.5  $\mu\text{M}$  solutions of (*R*)-1 in decalin after rapid cooling. Heating/cooling rate = 0.5 K min<sup>-1</sup>. (C, D) calculated net helicities, defined as the difference between monomers in *P*- and *M*-helical stacks, during thermal equilibration of fully non-equilibrated systems of 5  $\mu\text{M}$  solutions of supramolecular polymers with thermodynamic stabilities equal to *n*-1 (C) and (*R*)-1 (D) in the absence (upper panels) and presence (lower panels) of a helical mismatch penalty. The initial ratio between *P*- and *M*-helices are given by  $[P]_i:[M]_i$ . the kinetic parameters in (c) are  $a = 10^9$  and  $E_a = 1$  kJ, and in (D) are  $a = 8 \times 10^7$  and  $E_a = 1$  kJ

A similar experiment on (*R*)-1 shows a considerably different result (Figure 3B). After fast cooling a 7.5  $\mu\text{M}$  decalin solution of (*R*)-1, approximately 65% of the monomers was captured in the kinetic trap and this sample was subjected to similar heating–cooling cycles as of *n*-1. In contrast to the previous observation, the sample was completely converted into its thermodynamic state during the first heating–cooling cycle to 75 °C. We followed the equilibration process in more detail by recording VT-CD curves starting at 20 °C to upper temperature ranging from 40 to 75 °C with 5 °C increments per cycle. During the heating–cooling cycles, the CD effect decreases up to 10 of millidegrees per run, indicating that (*R*)-1 returns to the thermodynamic equilibrium state much faster than *n*-1. The faster equilibration of (*R*)-1 at low temperatures compared to the trapped

polymers of *n*-1 indicates that the non-equilibrium state of (*R*)-1 is more labile. Already at 75 °C, the sample attains the full thermodynamic equilibrium state. Apparently, the monomers of (*R*)-1 prefer to self-nucleate on their own preferred helical polymer. This strong contrast in observed equilibration efficiencies raises the question whether fundamental kinetic factors of the polymerization, such as activation energies or pre-exponential Arrhenius factors, in the chiral and achiral monomers are fundamentally different, despite the very similar structural architectures formed by the two molecules.

To shed light on this apparent dichotomy in stability of the kinetically trapped states of (*R*)-1 and *n*-1, we constructed a kinetic model that describes the temperature-dependent polymerization and depolymerization of the monomers in the two helical states (full details in the

Supplementary Information). Using this model, we simulated the equilibration kinetics during the thermal cycling experiments of kinetically trapped supramolecular polymers in the presence and absence of a helical preference, introduced by the mismatch penalty. In these simulations, we used the thermodynamic stabilities of the polymers as obtained from the fits of the homopolymerization. We then start the simulation from a distribution of *P*- and *M*-helical polymers as obtained by the evaporation of (*S*)-ClMeBu and the rapid cooling for simulations without and with helical preference, respectively (Figure 3C,D). A comparison of the trends observed in the simulations and experiments show several interesting features. Our simulations of systems with the thermodynamic parameters of the more stable ***n*-1** yield only minor changes in the net helicity in the parameter regime probed (Figure 3C). In contrast, simulations of a system with the thermodynamic parameters of (***R***)-**1** show significant changes in net helicity, even though pre-exponential factors in the Arrhenius equation in these calculations are an order of magnitude lower (Figure 3D). Most interestingly, however, the presence of a helical preference in the simulations results in considerably faster equilibration for both sets of thermodynamic parameters simulated (Figures 3C,D and S10). We presume that the broken symmetry in the nucleation of new homochiral nuclei is at the origin of this considerably faster equilibration of monomers with a chiral preference. The other parameters in the simulations, the activation energy and pre-exponential factors, appear to only have a moderate effect on the observed equilibration rate for supramolecular polymers of a given thermodynamic stability (Figures S11 and S12). As such, our calculations show that helical preferences in supramolecular polymerizations can lead to strong enhancements in equilibration kinetics of structures far from thermodynamic equilibrium, a finding relevant to the analysis of kinetically controlled supramolecular polymerizations.

### 3 | CONCLUSION

In conclusion, we have presented two different routes to arrive at kinetically trapped states of helical supramolecular polymers based TTA monomers. While the chiral versions of TTA can be trapped by fast cooling from high temperatures in decalin, the achiral version uses a volatile chiral cosolvent and its ability to deracemize supramolecular polymers made of ***n*-1**. After removal of the chiral solvent, the decalin solution with biased helicity of the achiral monomers remains kinetically stable at room temperature. The kinetic stabilities of the kinetically trapped states of the two helical polymers differ as shown

in heating–cooling cycles by step-by-step increasing the maximum temperature in each cycle. Theoretical simulations of the experimental results indicate that the chiral monomers formed during depolymerization prefer to polymerize in their preferred thermodynamically state. The achiral monomers, however, give intrinsically no helical bias and prefer growing on the still existing polymers. Although the presence of stereocenters in monomers typically does not create large differences in thermodynamic stabilities, chirality and the helical preference originating from it can significantly affect the equilibration kinetics of trapped states. The concepts presented in this paper can further advance control in the selective preparation of thermodynamically and kinetically controlled supramolecular structures in the same environment. We hope that such insights will lead to the incorporation of kinetically controlled living supramolecular polymers and thermodynamically controlled nucleated polymers in a new generation of responsive materials.

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### CONFLICT OF INTEREST

There are no conflicts to declare.

### AUTHOR CONTRIBUTIONS

**E. W. Meijer:** Conceptualization (lead); supervision (lead); writing – review and editing (lead). **Marcin L. Ślęczkowski:** Conceptualization (supporting); formal analysis (lead); investigation (lead); visualization (equal); writing – original draft (equal). **Mathijs F. J. Mabeoone:** Formal analysis (lead); methodology (equal); software (lead); validation (equal); writing – original draft (equal); writing – review and editing (equal). **Marco D. Preuss:** Data curation (equal); formal analysis (equal); investigation (equal); methodology (equal); validation (equal); writing – review and editing (supporting). **Yorick Post:** Data curation (equal); investigation (supporting); writing – review and editing (supporting). **Anja R. A. Palmans:** Supervision (supporting); writing – review and editing (equal).

### DATA AVAILABILITY STATEMENT

The data presented in this study are available from the corresponding author upon reasonable request.

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