



Ring-Opening Metathesis Polymerization of Norbornene-Based Monomers Obtained via the Passerini Three Component Reaction

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Ring-opening metathesis polymerization is a robust method to synthesize a variety of polymers by using ring-strained molecules as monomers, e.g., norbornenes. However, the synthesis of monomers with multiple functional groups remains a challenge, albeit peptide functional norbornenes have previously been used. Here, the Passerini three component reaction is exploited to synthesize norbornenes with two variable functional groups varying in bulkiness and distance from the polymerizable alkene. The results indicate that the functional groups do not affect the kinetics of the polymerization, whereas the length of the linker has a minor effect. Furthermore, a diblock-type copolymer is synthesized in a one-pot fashion, also indicating good control of the polymerization process. The thermal properties of all polymers are evaluated, highlighting the effect of monomer composition. This synthetic approach can be transferred to a variety of compounds, thus promising highly diverse polymers with complex compositions and architectures.

to functional groups while maintaining their catalytic efficiency.^[5] The introduction of functional groups through the corresponding functional monomer has allowed the synthesis of, e.g., stimuli-responsive, protein-binding, self-curing, or antimicrobial polymers via ROMP.^[6] Indeed, such monomers are often based on norbornenes due to their stability, high reactivity, and easy preparation via Diels–Alder reactions.^[7] The versatility of these monomers enables the preparation of a wide range of functional materials, such as highly conducting membranes or molecular carriers for drugs.^[8] Peptide-containing norbornenes are another example of functional monomers that are of particular interest as they carry the multifunctional character of the respective peptide, which is owed to the variety of natural and

synthetic amino acids.^[9] Recently, such monomers have been used in the synthesis of brushes or unimolecular micelles that show potential for biomedical applications as building scaffolds for the synthesis of cell-penetrating peptides.^[10] While peptide synthesis typically entails multistep reactions,^[11] multicomponent reactions (MCRs) have emerged as an alternative method to introduce multiple functionalities into a molecule in a one-step process.^[12] MCRs are particularly attractive in polymer chemistry where they have been used to introduce selected functionalities to monomers,^[13] polymer end groups,^[14] but also in the preparation of polymers,^[15] and sequence-defined oligomers with applications in drug delivery and data storage.^[16] MCRs have also been employed in metathesis reactions where, in a recent example, carboxylic acid-functionalized norbornenes and a range of aldehydes were reacted in an Ugi four component reaction, resulting in a small library of monomers with dipeptide-like functional groups.^[17] Upon ROMP, the corresponding polymers were obtained, while by selection of an enantiomerically pure aldehyde, chiral materials were also reported. Another versatile MCR is the Passerini three component reaction (P-3CR) in which a carboxylic acid reacts with a ketone or an aldehyde, and an isocyanide, yielding an α -acyloxy amide.^[18] The resulting polymers have the ability to hydrogen bond, making them interesting, e.g., in the field of drug delivery.^[15]

Inspired from the versatility of ROMP and MCRs, we herein report the synthesis of functional norbornenes using the P-3CR. This synthetic path enabled the functionalization of

1. Introduction

In recent years, ring-opening metathesis polymerization (ROMP) has attracted the interest of researchers due to its well-controlled polymerization kinetics and its versatility.^[1] ROMP is used for the polymerization of cyclic molecules with high ring strain containing an olefinic moiety, such as cyclopropenes or norbornenes.^[2] Due to the absence of chain transfer and chain termination events as well as its linear reaction kinetics, ROMP is denoted as a living polymerization.^[3] The living nature of the polymerization is often compromised due to the interaction of the functional groups present on the monomer with the organometallic catalyst.^[4] However, organometallic ruthenium complexes have been shown to exhibit high tolerance

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the monomer with two side groups by variation of the isocyanide and the aldehyde component. We investigated the ROMP kinetics of these endo and exo monomers using the Grubbs first generation catalyst while the thermal properties of the resulting polymers, i.e., glass transition and decomposition temperatures were also determined. The aim was to develop a strategy by which multifunctional monomers are synthesized and polymerized leading to polymers with designed properties.

2. Results and Discussion

The synthesis of bifunctional monomers suitable for ROMP started from carboxylic acid bearing norbornenes. The functional groups were introduced via the P-3CR through the corresponding aldehyde- and isocyanide-functional moieties: 1-pentyl isocyanide or 1-adamantyl isocyanide and propionaldehyde or 1-pyrenecarboxaldehyde. In order to assess the significance of the proximity of the functional group, and particularly the α -acyloxy amide, to the norbornene alkene for the subsequent ROMP, the length of the alkane linker between the precursor norbornene (*cis*-5-norbornene-2,3-dicarboxylic anhydride) and the acid group was varied by employing either glycine or 11-aminoundecanoic acid as the short or long linker, respectively. ROMP kinetics are well-known to be influenced by stereoisomerism of the norbornene monomer,^[19] therefore, in all cases, both the endo and the exo corresponding isomers were prepared. The overall monomer synthesis yields were satisfactory, ranging from 55% to 94% with the higher yields corresponding to the exo aliphatic monomers (Table 1).

The synthesized monomers were then polymerized by ROMP (Table 2). The polymerizations were performed under argon atmosphere at ambient temperature, in degassed dichloromethane (DCM) catalyzed by a Grubbs-type catalyst. In order to obtain the different polymerization rates of exo and endo norbornenes, the first generation Grubbs (G1) catalyst was selected. The monomer to catalyst (M:I) ratio was chosen to be 50:1. After 1 h (P1–P4), or 14 d (P5–P8), the polymerizations were quenched by the addition of ethyl vinyl ether.

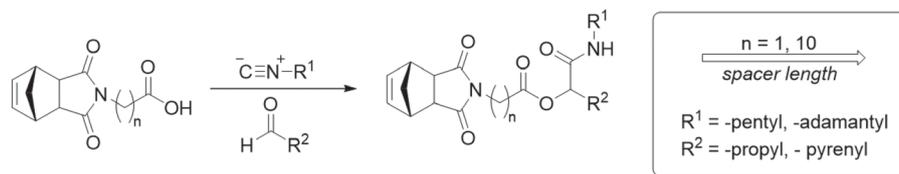
Generally, characterization of the obtained polymers by size exclusion chromatography (SEC) indicated narrow molecular weight distributions (Figures S49 and S50, Supporting Information). However, P2 showed a high molecular weight shoulder, which was not present in the SEC trace of the corresponding endo-derived polymer P6. Nonetheless, as the overall dispersity was rather low ($D_M = 1.17$), no further investigation was conducted. It is herein noted that removal of ruthenium traces by adding potassium 2-isocynoacetate per previous literature was not carried out as it led to cleavage of the side groups.^[20] The molecular weights, determined by SEC, differed from the expected (Table 2), however this was attributed to the SEC calibration being based on a different polymer, namely poly(methyl methacrylate).^[21] It is noted that consistently lower M_n s were obtained for the polymers with bulky side groups, in contrast to the consistently higher M_n s obtained for the alkyl-functional polymers. Furthermore, the glass transition temperature (T_g) and decomposition temperature (T_d) of the homopolymers were determined using differential scanning calorimetry (DSC) and thermogravimetric

analysis (TGA), respectively. The T_g s of the polymers unsurprisingly varied according to their functional groups.^[22] Both P2 and P4, which bear an adamantyl and a pyrene group, exhibited T_g s higher than the corresponding alkyl-functional P1 and P3. Furthermore, differences in the T_g s depending on the length of the alkane linker were also observed. The T_g s of P1 and P3, drastically decreased with increasing length of the alkane linker (T_g , P1 = 83 °C; T_g , P3 = –2 °C).^[23] Similarly, P2 and P4, also differed in thermal properties as the T_g of P4 was found to be 92 °C, whereas P2, which is a solid at room temperature, did not exhibit a T_g in the examined temperature region (25 °C–200 °C) (Figure S51, Supporting Information). Similarly, the endo-derived analogues exhibited comparable thermal behaviors. A thermal transition for P6 was not observed while the T_g s of P5, P7, and P8 were found to be higher than their corresponding exo analogues (Table 2, Figure S52, Supporting Information). A copolymer, namely P17, prepared by reacting equimolar amounts of M3 and M4 with G1 ($M_n = 28\,300\text{ g mol}^{-1}$, $D_M = 1.08$) exhibited a T_g between those of the corresponding homopolymers (T_g , P17 = 55 °C) (Figure S53, Supporting Information). In all cases, the T_d was above 300 °C with the decomposition occurring in multiple steps (Figures S47 and S48, Supporting Information).

In order to assess and compare the polymerization behavior of the bifunctional monomers, kinetic studies were conducted using ¹H NMR spectroscopy. The polymerizations were performed in gas-tight NMR tubes, applying the same conditions as above. The conversion at different time intervals was used to calculate apparent propagation rate constants (k_p) (from the slope of the best linear fit of the $\ln([M_0]/[M])$ versus time data) and monomer half-lives ($t_{1/2}$)^[24] (Table 3). Both endo and exo norbornenes showed pseudo first order behaviors, hence, first order kinetics were applied for analyses (Figure 1A,B).^[25] Minor trends regarding the effect of the steric hindrance of the side group and spacer length were observed: the k_p s of the exo monomers (M1–M4) showed a slight increase with increasing the spacer length (from k_p , M1 = $3.94 \times 10^{-3}\text{ s}^{-1}$ to k_p , M4 = $5.42 \times 10^{-3}\text{ s}^{-1}$), while the size of the functional groups was not found to affect the kinetics. In contrast, when endo monomers (M5–M8) were employed, slightly higher k_p s were obtained for M5 ($k_p = 1.45 \times 10^{-6}\text{ s}^{-1}$) attributed to the shorter spacer, and for M7 ($k_p = 1.23 \times 10^{-6}\text{ s}^{-1}$) attributed to the less bulky alkyl functional groups. In order to determine potential high degree of polymerization limitations, ROMP with a 200-fold excess of the exo monomer M1, with respect to the G1, was carried out obtaining homopolymer P20. Once again, a pseudo first order behavior was obtained and the calculated k_p value was found to be $4.36 \times 10^{-3}\text{ s}^{-1}$ comparable to that obtained for P9 (Table 3). Characterization by SEC indicated a narrow molecular weight distribution ($M_n = 55,600\text{ g mol}^{-1}$, $D_M = 1.16$, Figure S72, Supporting Information), thus indicating that higher degrees of polymerization are attainable with comparable kinetics.

In order to evaluate the influence of the structural characteristics of the monomer on their copolymerizability, kinetics employing two different monomers were conducted using ¹H NMR spectroscopy. The comonomer ratio was chosen to be 1:1 with a targeted total degree of polymerization (DP) of 50. Copolymerization of the exo monomers M3 and M4 resulted in a similar overall k_p , compared to the corresponding

Table 1. Norbornene bifunctional monomers obtained via the Passerini three component reaction (P-3CR).



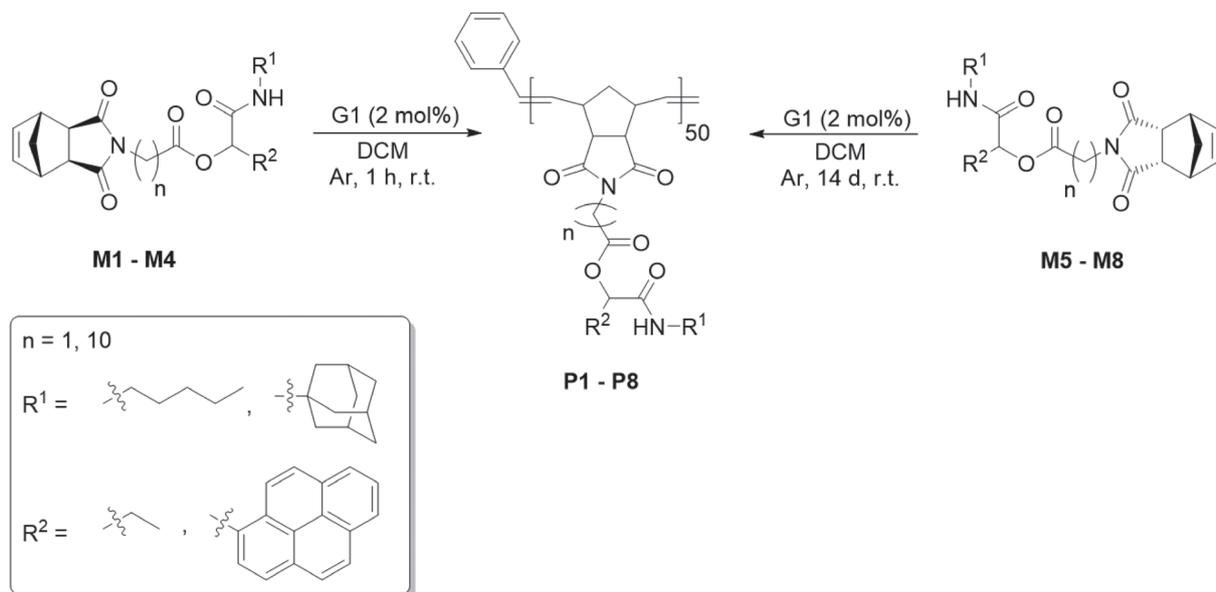
Monomer ^{a)}	Acid	Aldehyde	Isocyanide	Yield [%] ^{b)}
M1				94
M2				63
M3				92
M4				82
M5				91
M6				55
M7				71
M8				76

^{a)}Reaction conditions: 1.00 eq. acid, 1.50 eq. aldehyde, and 1.50 eq. isocyanide in dichloromethane (DCM) (1 M) were stirred for 48 h at ambient temperature; ^{b)}Yield refers to isolated yield after column chromatography.

homopolymerizations (Figure S67, Supporting Information), indicating that the bulky side groups of **M4** had no retarding effect on the copolymerization. Copolymerization (**P19**) of endo monomers **M5** and **M6** resulted in a slight increase of k_p of **M6** and in a slight decrease of k_p of **M5**, compared to their corresponding homopolymerizations (Figure S68, Supporting Information). In both cases, therefore, the copolymerization of monomers with the same stereoisomerism proceeded with

kinetics comparable to the respective homopolymerizations, regardless of the linker length or the bulkiness of the functional groups. Subsequently, we investigated the copolymerization of endo and exo norbornenes. Copolymer **P18** was synthesized using a mixture of the alkyl-functional exo monomer **M1** and the pyrenyl-adamantyl-functional endo monomer **M8**. As previously observed,^[19] such a combination slows down the polymerization of the fast monomer and accelerates that of

Table 2. Schematic: Ring-opening metathesis polymerization (ROMP) of exo (left) and endo (right) norbornene monomers using G1 catalyst. Table: Molecular weight, dispersity, and thermal characteristics of the homopolymers synthesized by ROMP of the bifunctional monomers.



Polymer ^{a)}	Monomer	Conversion [%] ^{b)}	$M_{n,theo}$ [g mol ⁻¹] ^{c)}	M_n [g mol ⁻¹] ^{d)}	\mathcal{D}_M ^{d)}	T_g [°C] ^{e)}	T_d [°C] ^{f)}
P1	M1	>99	18 900	23 000	1.10	83	319
P2	M2	>99	30 700	22 400	1.17	–	310
P3	M3	>99	25 200	32 000	1.07	–2	347
P4	M4	> 99	37 000	28 800	1.09	92	305
P5	M5	74	13 900	18 700	1.06	99	328
P6	M6	67	20 500	15 400	1.11	–	323
P7	M7	63	15 800	22 100	1.06	1	349
P8	M8	64	23 600	20 300	1.08	114	306

^{a)}Reaction conditions: 0.1 M monomer in DCM with 2 mol% G1 for 1 h (**P1–P4**) or 14 d (**P5–P8**) at ambient temperature; ^{b)}Determined by ¹H NMR spectroscopy; ^{c)}Calculated by the conversion; ^{d)}Determined by size exclusion chromatography (SEC); ^{e)}Onset, determined by differential scanning calorimetry (DSC); ^{f)}Temperature at 10% weight loss, determined by thermogravimetric analysis (TGA).

the slow monomer. Therefore, the pseudo first order kinetics is skewed (Figure 1C). With **M1** still present in the reaction mixture, the consumption of **M8** (in **P18**) was accelerated compared to its homopolymerization, while after complete consumption of **M1**, the polymerization of **M8** decelerated again and the pseudo first order kinetics was restored. Owing to the fast consumption of the exo monomer compared to the polymerization of the endo monomer, a diblock-like copolymer was obtained ($M_n = 16,200 \text{ g mol}^{-1}$, $\mathcal{D}_M = 1.17$).^[26] It is noted that both blocks are diluted by the building block of the other block, as evidenced by determining the copolymer composition at different time points through the conversions (Figure 1D). The first datapoint (at ≈ 6 min) showed that $\approx 6\%$ of the endo **M8** and 14% of the exo **M1** had polymerized, corresponding to a copolymer with a relative composition of 0.7 for **M1**. Over the subsequent 2 h, this value gradually increased as **M1** was rapidly consumed and reached a maximum value of 0.85. At this point, **M1** had not been fully consumed (conversion $\approx 72\%$) and **M8** had reached a conversion of 13%, thus confirming the **M8** “diluted” **M1** block composition. Over the

next days, both monomers were slowly consumed with the **M1** reaching 100% conversion after 45 h and **M8** reaching 74% conversion after 336 h. Therefore, the second block was deemed to be an **M1** “diluted” **M8** block.

3. Conclusion

In summary, we synthesized functional exo and endo norbornenes via P-3CR and subsequently polymerized them via ROMP. The polymerization process was found to be well controlled, observing linear reaction kinetics and narrow molecular weight distributions for all monomers. The resulting polymers ranged from viscous liquids to glassy solids, as determined by their glass transition temperatures, depending on the functional groups present. Furthermore, copolymers were synthesized by combining different monomers, while a diblock-like copolymer was also attainable in a one-pot reaction. The herein described strategy thus allows the straightforward design of highly diverse and multifunctional polymers.

Table 3. Summary of kinetic analyses of the homopolymerizations (P9–P16, P20) and the copolymerizations (P17–P19) of the bifunctional monomers.

Polymer	Monomer	Type	Conversion [%] ^{a)}	Reaction Time [h]	k_p [s ⁻¹] ^{b)}	$t_{1/2}$ [h] ^{c)}
P9	M1	exo	>99	0.37	3.94×10^{-3}	0.049
P10	M2	exo	>99	0.38	3.46×10^{-3}	0.056
P11	M3	exo	>99	0.28	4.56×10^{-3}	0.042
P12	M4	exo	>99	0.27	5.42×10^{-3}	0.036
P13	M5	endo	83	335	1.45×10^{-6}	133
P14	M6	endo	75	335	1.05×10^{-6}	183
P15	M7	endo	78	340	1.23×10^{-6}	156
P16	M8	endo	74	340	1.05×10^{-6}	183
P17	M3	exo	>99	0.27	5.57×10^{-3}	0.034
	M4	exo				
P18	M1	exo	>99	336	–	–
	M8	endo	74			
P19	M5	endo	76	334	1.11×10^{-6}	173
	M6	endo	77			
P20 ^{d)}	M1	exo	>99	0.37	4.39×10^{-3}	0.044

^{a)}Determined by ¹H NMR spectroscopy before quenching; ^{b)}Calculated from conversion using first order kinetics; ^{c)}Calculated from the linear regression fits using $t_{1/2} = \ln(2)/k_p$; ^{d)}The monomer to G1 ratio was 200:1.

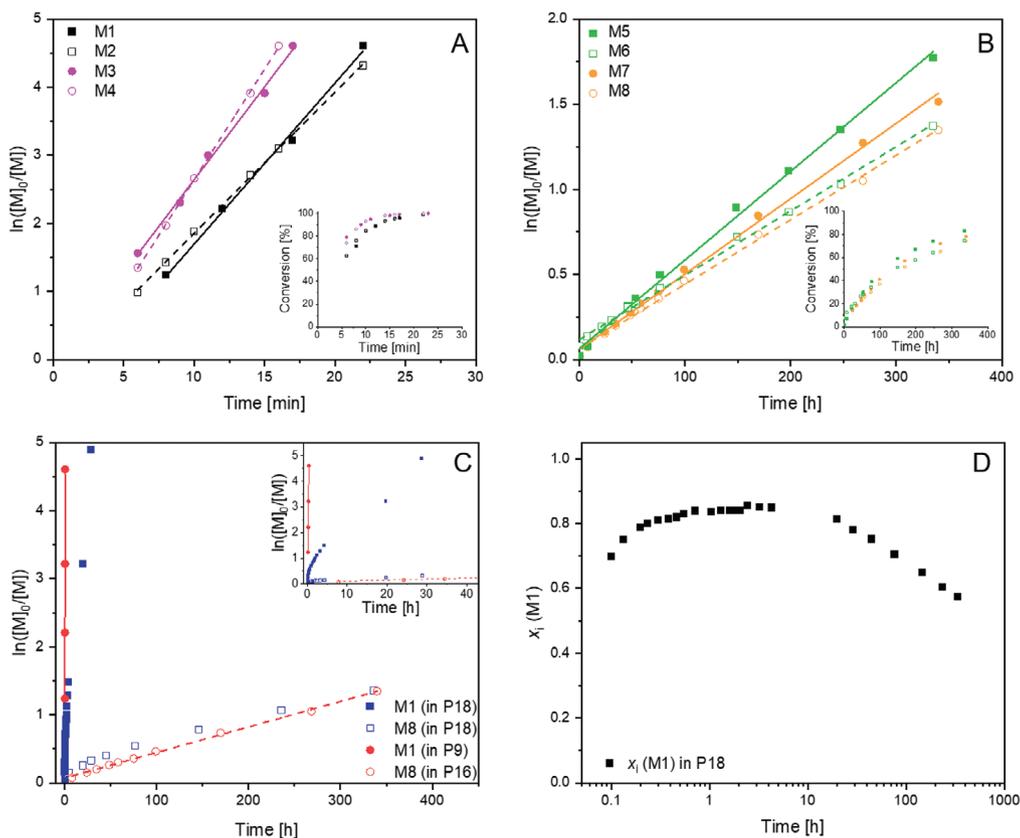


Figure 1. Kinetic plots of the A) homopolymerizations of exo monomers **M1–M4**, B) homopolymerizations of endo monomers **M5–M8**, C) copolymerization (**P18**) of **M1** and **M8** and homopolymerizations of the respective monomers, and D) molar fraction (x_1) of **M1** in the copolymer **P18** as a function of polymerization time, based on ¹H NMR spectroscopy data. Lines represent linear fits.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that supports the findings of this study are available in the supplementary material of this article.

Keywords

kinetics, living polymerization, multicomponent reactions, norbornenes, Passerini, ROMP

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