

Table 2. Calculations of p(BA)-*r*-p(MPDL) compositions during copolymerization at different temperatures

Entry	$T, ^\circ\text{C}$	Integration values for the following protons in p(BA)- <i>r</i> -p(MPDL)			Composition a:b:c (mol %)*
		B_1	P_{1-3}	M_2	
1	65	6.71	5	0.65	70:11:19
2	90	9.8	5	0.54	77:11:12
3	110	8.35	5	0.45	74:15:11

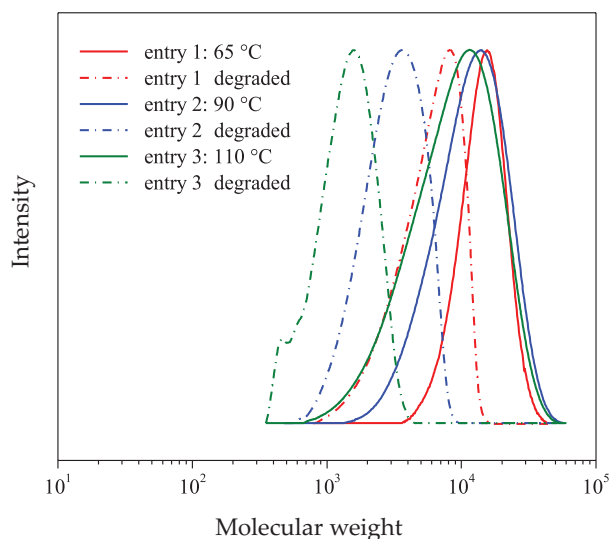
*composition a:b:c is for the copolymer structure depicted in Fig. 4.

^1H NMR spectra obtained for the polymers synthesized at different temperatures were normalized to aromatic protons, and their compositions were calculated based on integration values presented in the Table 2.

Increasing the temperature from 65 °C to 90 °C resulted in 30 % increase of incorporated MPDL *via* ring-opening process. A further increase from 90 °C to 110 °C resulted in another 20 % increase in the content of ring-opened MPDL in the copolymer. Therefore, while ring-opening efficiency could be improved by increase in temperature, the most significant improvement was detected for the first increase from 65 °C to 90 °C. A further 20 °C increase in temperature resulted in marginally higher ring-opening efficiency.

Furthermore, higher molecular weight p(BA)-*r*-p(MPDL) copolymers were synthesized at varied temperatures to evaluate their degradation behavior based on the ring-opening efficiency. As in a previous set of experiments, ring-opening efficiency increased at higher temperature (Fig. 5). Copolymerizations were conducted at higher monomer concentrations to facilitate higher yield of the targeted copolymers.

Copolymerization conditions: [BA]:[MPDL]:[EBiB]:[CuBr₂]:[Me₆TREN]:[RI] = 200:100:1:0.03:0.06:0.1, reaction solvent - DMF, 65 °C–110 °C, [BA] = 3.4 M, [MPDL] = 1.7 M. Each sample was incubated for 45 h in 5 % KOH in THF/MeOH (1/1), and the polymer was precipitated after acidification with 1 M HCl, dissolved in THF and analyzed

**Fig. 5.** GPC traces of copolymers p(BA)-*r*-p(MPDL) prepared at different temperatures before and after degradation**Table 3.** Studies of copolymers p(BA)-*r*-p(MPDL) prepared at different temperatures before and after degradation

Entry	$T, ^\circ\text{C}$	\bar{M}_n	\bar{M}_w/\bar{M}_n	f_{MPDL} %	RO, %
1	65	13 800	1.16	24	16
1 degraded		4 850	1.32		
2	90	9 500	1.43	27	45
2 degraded		2 690	1.28		
3	110	7 590	1.55	28	55
3 degraded		1 180	1.27		

by THF GPC. Such difference could potentially be relevant to the difference in the incorporation of the ring-opened form of MPDL for the sample prepared at the lowest temperature. The copolymers were incubated under basic conditions to determine their degradation properties and GPC was used to determine decrease in MW resulting from the degradation reactions (Fig. 5, Table 3).

As expected, according to this analysis, the p(BA)-*r*-p(MPDL) copolymer with highest MPDL content in the ring-opened form was characterized by the largest decrease in MW. Since the total incorporation of MPDL in these copolymers varied insignificantly, it is likely that drastic difference in the amount of ring-opened MPDL *vs.* MPDL incorporated *via* vinyl addition is responsible for more efficient degradation of copolymers prepared at 90 °C and 110 °C compared to the copolymer prepared at 65 °C.

In the next set of experiments, MPDL was copolymerized with water-soluble monomers, such as OEOA₄₈₀, OEOMA₅₀₀ and PEOMA_{2k} (Table 4).

The initial polymerization reaction for OEOA₄₈₀ was conducted at 65 °C with the ratio of reagents identical to BA/MPDL copolymerization (Table 4, entry 1). According to the analysis, the final copolymer contained around 20 % of MPDL, and 32 % of this MPDL underwent ring-opening (Fig. 6). This was consistent with the results obtained for BA/MPDL copolymerization.

In the next experiment, OEOA₄₈₀/MPDL copolymerization was conducted at 90 °C to improve percentage of MPDL incorporated into the copolymer in its ring-opened form. Additionally, the targeted degree of polymerization (DP) was increased to 1500. To date, most of synthesized copolymers with CKA were characterized by rather low MW (10 000–20 000), with some systems reaching ~50 000 [55]. However for certain biological application the prepa-

Table 4. Copolymerization of MPDL with hydrophilic monomers by ICAR ATRP

Entry	$M_1/M_2/I/CuBr_2/L/RI$	M_1	Time, h	\bar{M}_n	\bar{M}_w/\bar{M}_n	f_{MPDL} %	RO, %
1	200/100/1/0.03/0.06/0.1	OEOA ₄₈₀	10.7	31	1.07	20.5	32
2	1000/500/1/0.15/0.3/0.5	OEOA ₄₈₀	10	50	1.37	9.2	62
3	1000/500/1/0.15/0.3/0.25	OEOMA ₅₀₀	10	147	1.73	6.1	82
4	1000/500/1/0.75/1.5/0.25	OEOMA ₅₀₀	6	125	1.49	5.9	74
5	150/150/1/0.03/0.06/0.1	PEOMA _{2k}	13	52	1.08	6.0	96

Volume – 5 ml total; reaction solvent – DMF; $T = 90\text{ }^\circ\text{C}$; M_1 – BA, M_2 – MPDL; L – Me₆TREN; [I] = [EBiB] = 5 mM; RI – radical initiator: V40 ($T_{11/2=10h} = 88\text{ }^\circ\text{C}$); entries 1–2: $[M_1] = 1\text{ M}$, $[M_2] = 0.5\text{ M}$; entry 5: $[M_1] = 0.3\text{ M}$, $[M_2] = 0.3\text{ M}$; RO – % of MPDL monomer in ring-opened form to ring-closed form; f_{MPDL} – fraction of MPDL incorporated into the polyether backbone, final \bar{M}_n was measured by DMF GPC with MALLS detector.

ration of degradable high MW polymers would be especially beneficial for the reasons stated earlier and because lower MW polymers could be removed from a physiological circulation without need for their degradation. In a similar manner to copolymerization with BA, copolymerization of OEOA₄₈₀ with MPDL at 90 °C resulted in the formation of a copolymer with a higher percentage of MPDL with ring-opened structure (Table 4, entry 2). The percentage of incorporated MPDL which underwent ring-opening during this copolymerization reached 62 %. The fraction of MPDL incorporated into the pOEOA₄₈₀ backbone was, however, less than 10 %.

When MPDL was copolymerized with OEOA methacrylate analogue, OEOMA₅₀₀, the overall incorporation of MPDL was lower (Table 4, entry 3–4). Polymerization resulted in high MW copolymer of almost 150 000, but its \bar{M}_w/\bar{M}_n value was relatively high indicating limited control over polymerization. In the presence of a higher concentration of catalyst, control over polymerization improved and resulted in formation of copolymers with lower \bar{M}_w/\bar{M}_n , 1.49 vs. 1.73 with 6 % of incorporated MPDL. Even though copolymers of MPDL with OEOMA₅₀₀ were characterized by higher \bar{M}_w/\bar{M}_n compared to copolymerization with acrylate OEOA₄₈₀, it was possible

to obtain polymers with $MW > 120\ 000$ with $\bar{M}_w/\bar{M}_n \sim 1.5$ (Table 4, entry 4).

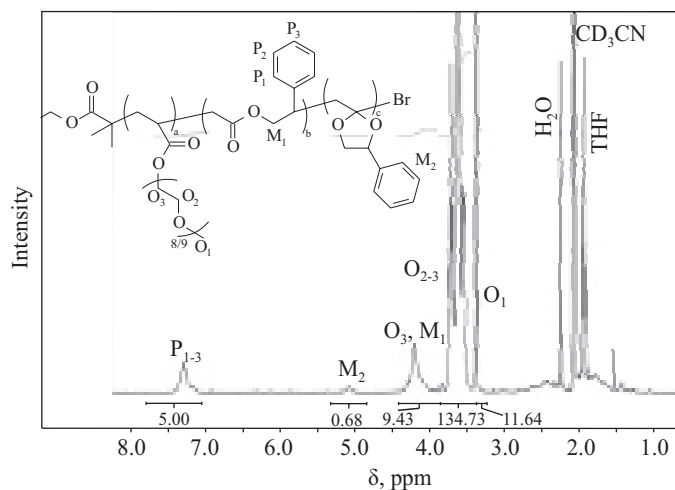
The degradability of both p(OEOA₄₈₀)-*r*-p(MPDL) and p(OEOMA₅₀₀)-*r*-p(MPDL) was evaluated by incubating the copolymers in 5 % aqueous KOH. Hydrolytic degradation results were analyzed by aqueous GPC to determine the decrease in MW with time (Fig. 7, Table 5).

Table 5. Degradation studies of hydrophilic copolymers (hydrolysis in 5 % aq. KOH)

Sample	Time, h	\bar{M}_n	\bar{M}_w/\bar{M}_n
p(OEOA ₄₈₀)- <i>r</i> -p(MPDL)	0	15 500	2.27
	20	4 620	1.40
	48	4 610	1.34
p(OEOMA ₅₀₀)- <i>r</i> -p(MPDL)	0	35 200	2.75
	20	8 540	1.86
	48	7 780	1.97

After 20 h, the molecular weight of both the water-soluble polyacrylate and polymethacrylate copolymers decreased by a factor of 3–4, and did not change over the next 28 h, indicating a full degradation had occurred. Final degradation products were characterized by $\bar{M}_n < 10\ 000$, according to calibration with PEO standards. However, it is important to point out that even though apparent \bar{M}_n (based on linear PEO standards) of degradable copolymers were only 15 500 for p(OEOA)-*r*-p(MPDL) and 35 200 for p(OEOMA)-*r*-p(MPDL), MW of copolymers as measured by MALLS detector was more than 100 000. Degradation of this higher MW fraction of copolymers resulted in formation of degraded products with MW significantly below their initial values.

The final copolymerization in this series of experiments was the copolymerization of MPDL with a PEO-MA_{2k} macromonomer. This was evaluated to determine if this procedure would form a degradable brush copolymer by the “grafting through” method (Table 4, entry 5). The synthesized polymer was characterized by incorporation of a similar fraction of MPDL (~6 %) as the lower MW OEOMA₅₀₀ monomer, however, according to proton NMR analysis, 96 % of the MPDL units had undergone ring-opening during the copolymerization (Fig. 8).

**Fig. 6.** ¹H NMR of purified copolymer p(OEOA₄₈₀)-*r*-p(MPDL) synthesized at 65 °C (300 MHz, CD₃CN)

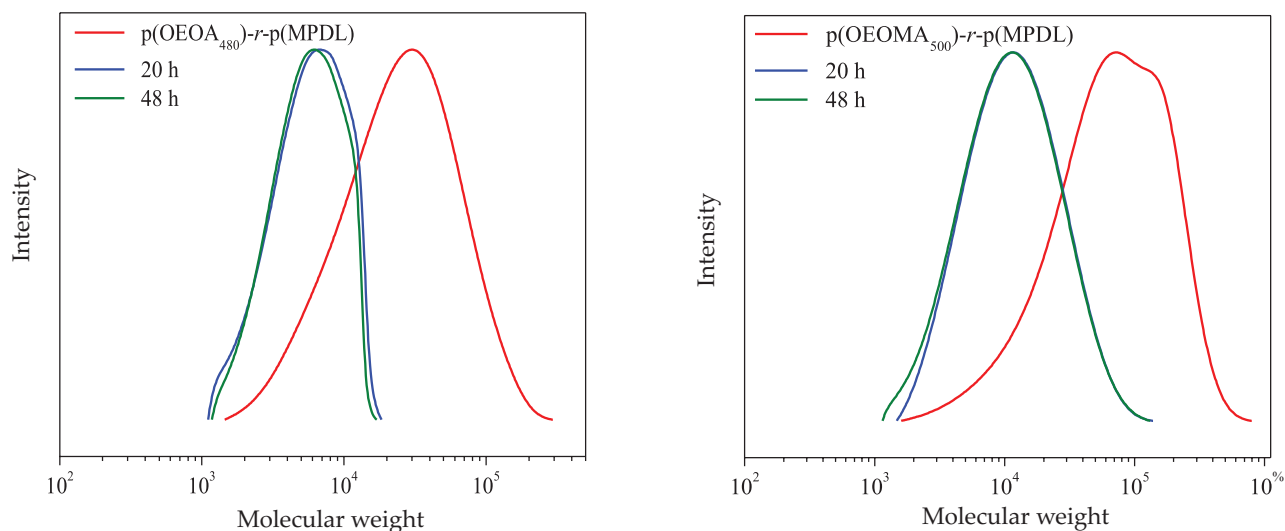


Fig. 7. Degradation studies of hydrophilic polymers; all samples were neutralized by 1 M HCl and analyzed by water GPC in PBS at pH = 7 (calibrated with linear PEO standards)

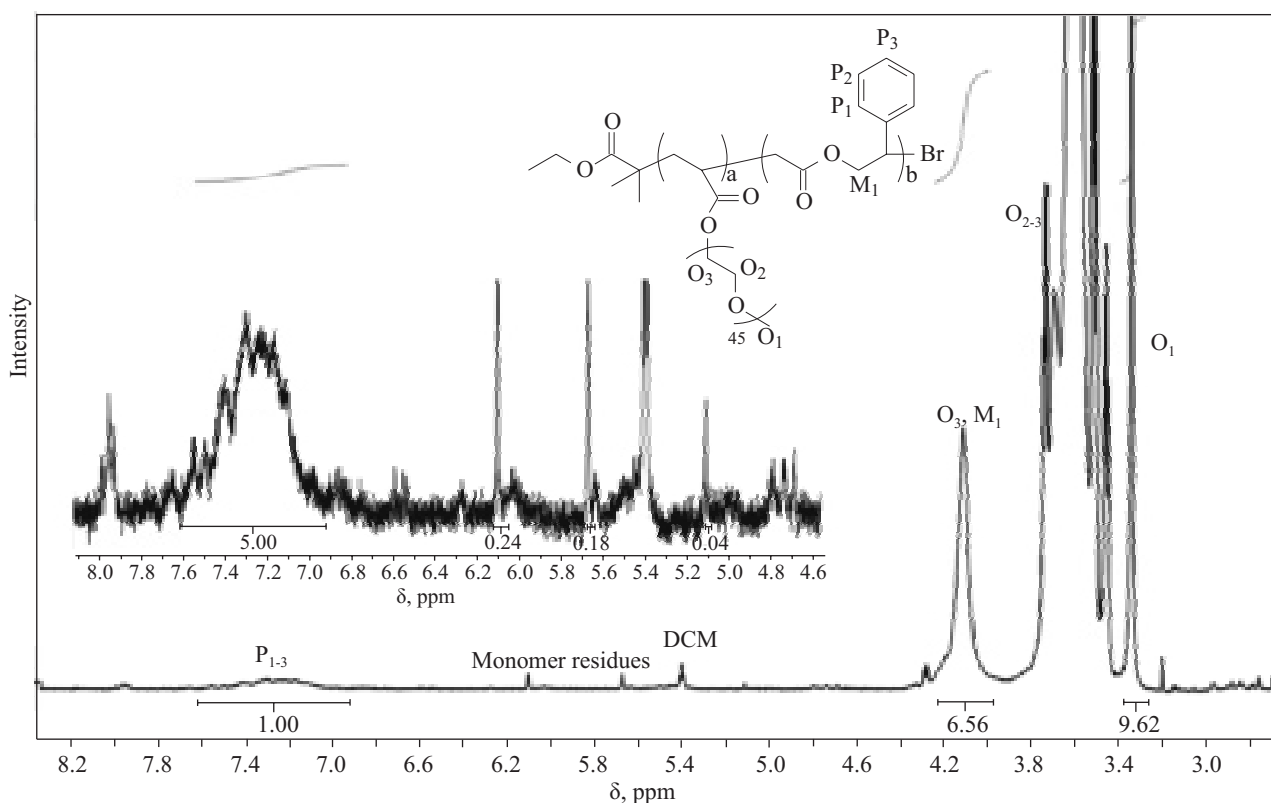


Fig. 8. ¹H NMR of purified copolymers p(PEOMA_{2k})-r-p(MPDL) (500 MHz, CD₃CN) with insert with zoomed in region 4.8–8 ppm

Besides structural difference of this type of macromonomer from other utilized monomers, the copolymerization was performed at very low comonomers concentrations (0.3 M) resulting in a relatively slow rate of polymerization (30 % monomer conversion in 13 h). This result indicated that it would be important to investigate further if copolymerization under dilute conditions and at a slower rate of polymerization could increase the prevalence of ring-opening of MPDL over vinyl-addition [70].

CONCLUSIONS

Degradable functional copolymers were synthesized by ATRP *via* copolymerization of methacrylates with MPDL as an exemplary CKA monomer. The efficiency of ring-opening of MPDL during copolymerization, which is required for formation of the degradable units in the backbone of the copolymer, increased at higher temperatures. MPDL was successfully copolymerized with both

acrylates and methacrylates, and copolymers with acrylates were characterized by higher levels of incorporation of MPDL into the copolymers (~2 to 3 times), compared to copolymers with methacrylates. High MW copolymers, $MW > 100\,000$, were synthesized and successfully degraded forming fragmented chains below the renal threshold limit.

The final copolymers were characterized by relatively high dispersities, and the measured MWs were lower than theoretically predicted. The ring-opening efficiency of MPDL incorporation varied with different comonomers, which could be explained by several differences in reaction conditions including monomer concentration, deactivation efficiency, or (cross)propagation rate coefficients. Thus, additional detailed studies have to be performed to identify all side reactions and establish conditions for more effective ring-opening with specific comonomers despite temperature effects, and also to determine how to control MW, $\overline{M}_w/\overline{M}_n$ and produce well-defined copolymers of complex architectures.

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