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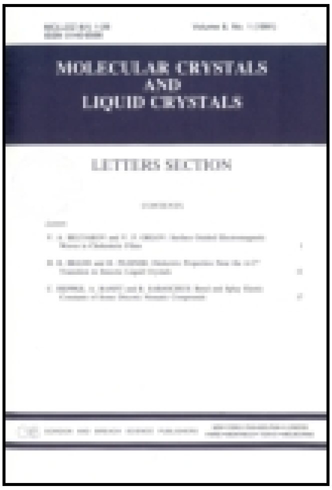

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LC Photoaligning Polymer Prepared via Selective Polymerization of Bifunctional Monomer with O-Methacryloyl and Styrene-Type Double Bonds

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LC Photoaligning Polymer Prepared via Selective Polymerization of Bifunctional Monomer with *O*-Methacryloyl and Styrene–Type Double Bonds

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*Phenylene-containing monomer **1** possessing* O*-methacryloyl and styrene double bonds with different reactivities in radical polymerization was designed as new type of bifunctional monomers suitable for preparation of LC photoaligning alkene-functionalized polymers. To elucidate an individual contribution of double bond of each type into kinetics of bifunctional monomers polymerization, thermo- and photo-induced polymerization of model monomers were investigated and compared. Polymer poly-**1** obtained by thermo-induced radical polymerization demonstrated high efficiency of LC ZLI 2293 photoalignment.*

Keywords Liquid crystal photoalignment; bifunctional monomer; selective polymerization

1. Introduction

Principally new concept for the design of LC photoalignment polymers was proposed and experimentally proven by the use of aryamidoester monomers possessing two polymerizable double bonds. The substituted arylmethacrylates which have the second double bond of tetrahydrophthalic, maleimide and methacrylamide type were developed as starting monomers for the syntheses of photoactive polymer materials with potential ability to undergo photo-Fries reaction [1–3]. The key role of the second tetrahydrophthalic/maleimide/methacrylamide double bond is its crosslinking reactions under the action of temperature or UV-irradiation that as we believe, would lead to the fixation of the photoinduced due to Fries reaction surface anisotropy [4]. However, among the most important tasks is a search of new synthetic roots giving increased yield of polymerization withought alkene-functionality crosslinking. For instance, selective polymerization of *O*-methacryloyl groups in monomers with structures similar to monomer **MAPhMA** (Fig. 1) is limited by low (up to ~30–40 wt.% monomer conversion) yield of soluble polymer product.

The presented study is devoted to the investigations of structure influence of a new bifunctional phenylene-containing monomer **1** on the possibility to carry out its selective homopolymerization under conditions of thermo- and photoinitiated radical polymerization

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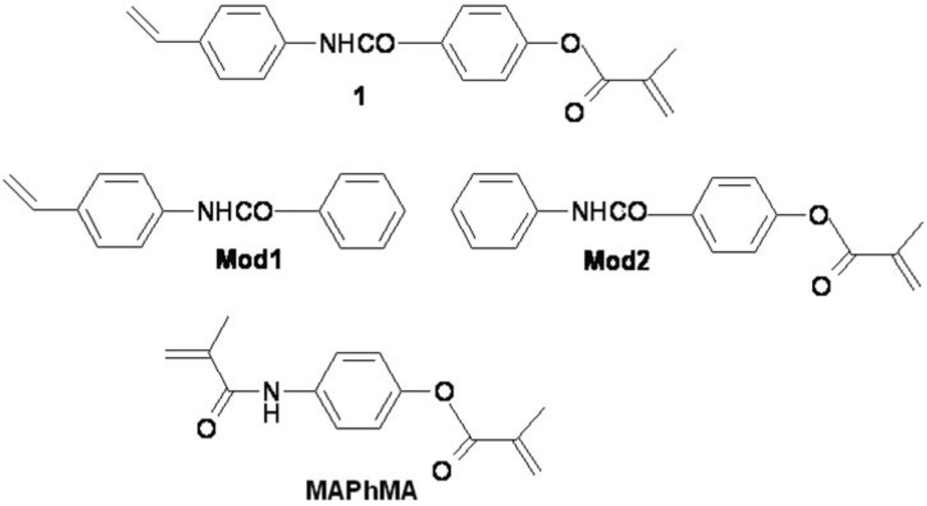


Figure 1. Chemical formulae of studied and model monomers.

in DMF solution. In addition, some model monomers having only one type of polymerizable bond were synthesized and tested at the same conditions to elucidate an individual contribution of methacrylate and vinyl group in polymerization rate and crosslinking at the polymerization of **1**. The structures of model compounds **Mod1** and **Mod2** are simplified comparing with **1** so that compound **Mod1** contains only vinyl ($C{=}CH_2$) double bond as well as compound **Mod1** contains methacrylate ($CO(CH_3)C{=}CH_2$) polymerizable bond.

2. Experimental Section

Materials and Methods

All solvents of p.a. quality (Aldrich) were stored over molecular sieves of 3 or 4 Å. Other chemicals were purchased from Aldrich and used without further purification. Bifunctional monomer **MAPhMA** was synthesized as described in [3]. Model monomers **Mod1** and **Mod2** were prepared in one or two steps synthetic procedures.

Monomer **Mod1** was synthesized with standard procedure of 4-aminostyrene condensation with benzoic acid. Yield (51 wt.%). M.p.: 160–161°C. R_f – value (acetone:hexane:benzene = 10:5:7) = 0.78.

^1H NMR (400 MHz, DMSO-d_6), ppm: 10.16 (NH), 7.96 (2H, Ar), 7.48 (2H, Ar), 7.76 (2H, Ar), 7.50 (2H, Ar), 6.63 (1H, =CH), 5.67 (1H, $=CH_2$), 5.15 (1H, $=CH_2$).

Monomer **Mod2** was synthesized by condensation of aniline with 4-(methacryloyloxy)benzoic acid previously obtained by acylation of 4-oxybenzoic acid with methacryloyl chloride. Yield (61 wt.%). M.p.: 111–113°C. R_f – value (acetone:hexane:benzene = 10:5:7) = 0.85. ^1H NMR (400 MHz, DMSO-d_6), ppm: 10.15 (NH), 8.02 (2H, Ar), 7.25 (2H, Ar), 7.78 (2H, Ar), 7.21 (2H, Ar), 6.33 (1H, $=CH_2$), 5.87 (1H, $=CH_2$), 1.73 (3H, CH_3).

Bifunctional monomer **1** was synthesized by condensation of 4-(methacryloyloxy)benzoic acid with 4-aminostyrene in the presence of DCC in tetrahydrofuran cooled to 0°C for 3 days. Yield (79 wt.%). M.p.: 167°C. R_f – value (acetone:hexane:benzene = 10:5:7) = 0.83.

^{1}H NMR (400 MHz, DMSO-d_6), ppm: 10.21 (NH), 8.02 (2H, Ar), 7.27 (2H, Ar), 7.74 (2H, Ar), 7.23 (2H, Ar), 6.66 (1H, =CH_2), 5.15 (1H, =CH_2), 2.05 (3H, CH_3).

IR (KBr): 3334 (NH), 2928, 2853 (aryl, alkyl), 1736 (C=O, ester), 1650 (C=O, Amide I), 1514 (overlapped, aryl + Amide II), 1326 (Amide III), 1208 (C—O), 1122 (COC), 936 (vinyl, C—H), 842 (1,4-disubstituted aryl); further intensive signals, 1404, 1164, 902, 764, 626 cm^{-1}.

Anal. Calcd. for $C_{19}H_{17}NO_3$ (307.3): C, 74.25; H, 5.58; N, 4.56. Found: C, 74.28; H, 5.61; N, 4.52.

Thin layer chromatography was performed on Merck Kieselgel plates 60-F254. ^{1}H NMR spectra were recorded with a Varian 400 NMR spectrometer with tetramethylsilane as internal standard in DMSO-d_6 as solvent. IR spectra were run on a Bruker IFS-66 FTIR spectrometer. An elemental analysis was performed with a Perkin-Elmer 2400 elemental analyser. GPC-measurements were performed using PSS-SDV columns and DMF eluent containing LiBr at a flow rate of 1.0 mL/min. The calibration curves for GPC analysis were obtained using PSS polystyrene standards (1000–400000 D).

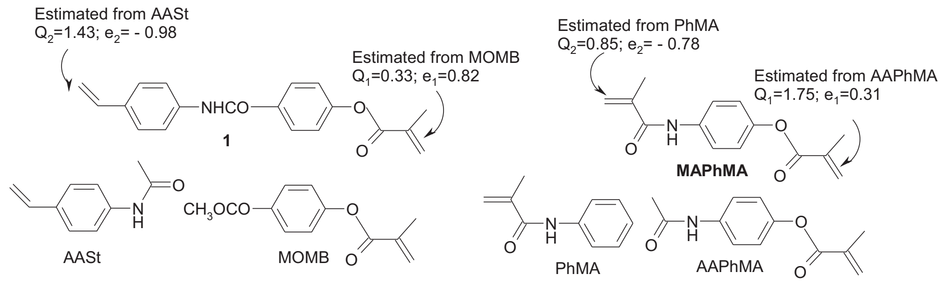
The kinetics of the monomers was studied by dilatometry under argon or in the air using DMF as a solvent. The yield of the polymers was determined by gravimetry. In a case of thermoinitiated polymerization the solution of 0.172 mol/l of monomer **1**, **Mod1** or **Mod2** were heated to 80°C at the presence of 0.0023 mol/l of 2,2′-azobis(isobutyronitrile) (AIBN) as initiator. In a case of photopolymerization, the temperature was maintained at 30°C and 0.0023 mol/l concentration of benzoin was used for initiation. The polymerization system was irradiated by full spectrum of high pressure mercury lamp (I = 4.5 mW/cm^2).

The quality of LC photoalignment was investigated in the following way. A 2-wt.% solution of the investigated polymer in dimethylformamide was spin coated (3000 rpm, 1 min) on the glass substrate. The obtained film was pre-backed during 10 min at 100°C, then backed at 180°C during 1 h. After that, the polymer film was normally irradiated by polarized UV light of mercury lamp (I = 28 mW/cm^2, t = 15 min) and then obliquely irradiated by unpolarized UV (I = 100 mW/cm^2, t = 1 min). At last, a symmetric optical cell (the cell thickness is 20 mm) with antiparallel LC alignment was made from the irradiated substrates. The cell was filled by liquid crystal ZLI 2293 ($\Delta\varepsilon > 0$). The quality of liquid crystal alignment was estimated by the observation of a sample in polarized light (a light box and a polarizing microscope) using evaluation scale with five grades: (1) excellent; (2) good (single alignment faults); (3) satisfactory (minor alignment faults); (4) bad (big number of alignment faults in a form of inversion walls, flowing patterns, etc.) and (5) no alignment.

3. Results and Discussion

Design and Syntheses of 4-{[(4-vinylphenyl)-anilino]carbonyl}phenyl-2-methylacrylate (bifunctional monomer 1)

Alkene-functionalized asymmetrical monomer **1** for selective polymerization was designed due to the expected higher contrast of radical polymerization reactivity of its *O*-methacryloyl and vinyl groups in comparison with previously investigated di-methacrylic monomer **MAPhMA** [3] (Fig. 1). The reactivities of the substituted *O*-methacryloyl and vinyl groups in **1** were estimated through Q and e values [5] of model monomers (Sheme 1) MMOB (Q_1 = 0.33, e_1 = 0.82) and AASt (Q_2 = 1.43, e_2 = −0.98) taken from [6] and [7],



Scheme 1. Molecular structures of model monomers.

respectively. To predict polymerization selectivity, homopolymerization of an asymmetrical divinyl monomer **1** can be considered as a copolymerization of two types of the reactive double bonds.

In contrast with **MAPhMA** with relative higher value of intrinsic reactivity (Q) of O-methacryloyl double bond estimated by $Q_1 = 1.75$ of model monomer AAPhMA in comparison with NH-methacryloyl double bond estimated by $Q_2 = 0.85$ of model monomer PhMA, in a case of monomer **1** $Q_2 = 1.43$ indicates the higher intrinsic reactivity of the substituted styrenyl group. In both bifunctional monomers **MAPhMA** and **1** charge difference between e_1 and e_2 should cause the same polarity activation effects for the participation of O-methacryloyl group in copolymerization (see Table 1).

Quantitative analysis using Alfrey-Price equations [5] gave estimated reactivity ratios r_1 of about 0.053 and r_2 of 0.74 in a case of bifunctional monomer **1**. These values indicate that the styrenyl groups have higher reactivities than do the O-methacryloyl groups and further suggest that the polymerizations of **1** can be considered as opposite to **MAPhMA** polymerization process because in **MAPhMA** O-methacryloyl double bonds have higher reactivity ($r_1 = 1.47$) then NH-methacryloyl one ($r_2 = 0.21$).

Thus, homopolymerization of bifunctional monomer **1** should result in preferable formation of polystyrene-type main chain polymer bearing side free O-methacryloyl fragments. At the same time, the general structural similarity with earlier described arylmethacrylates [1–4], monomer **1** would open a new class of bifunctional monomers suitable for preparation of LC photoaligning alkene-functionalized polymers.

Monomer **1** was synthesized readily by the condensation of 4-(methacryloyloxy)benzoic acid with 4-aminostyrene in the presence of DCC in

Table 1. Calculated reactivity ratios (r_1, r_2) of double bonds in bifunctional monomer **1** in comparison with **MAPhMA**

Bifunctional monomer	$(Q_1,e_1)^*$	$(Q_2,e_2)^*$	r_1	r_2	r_1/r_2
1: MMOB versus AAST	(0.33,0.82)	(1.43,−0.98)	0.053	0.74	0.07
MAPhMA: AAPhMA versus PhMA	(1.75,0.31)	(0.85,−0.78)	1.47	0.21	7.00

*(Q, e) values of N-(p-phenyl)methacrylamide (PhMA) and 4-(N-(acetyl)amino)styrene (AASt) are cited from [6], for methyl-4-(methacryloyloxy)benzoate (MMOB) and p-acetylaminophenylmethcrylate (AAPhMA) are cited from [7]. The reactivity ratios were calculated based on Alfrey-Price equations: $r_1 = (Q_1/Q_2)\exp[-e_1(e_1\text{-}e_2)]$; $r_2 = (Q_2/Q_1)\exp[-e_2(e_2\text{-}e_1)]$

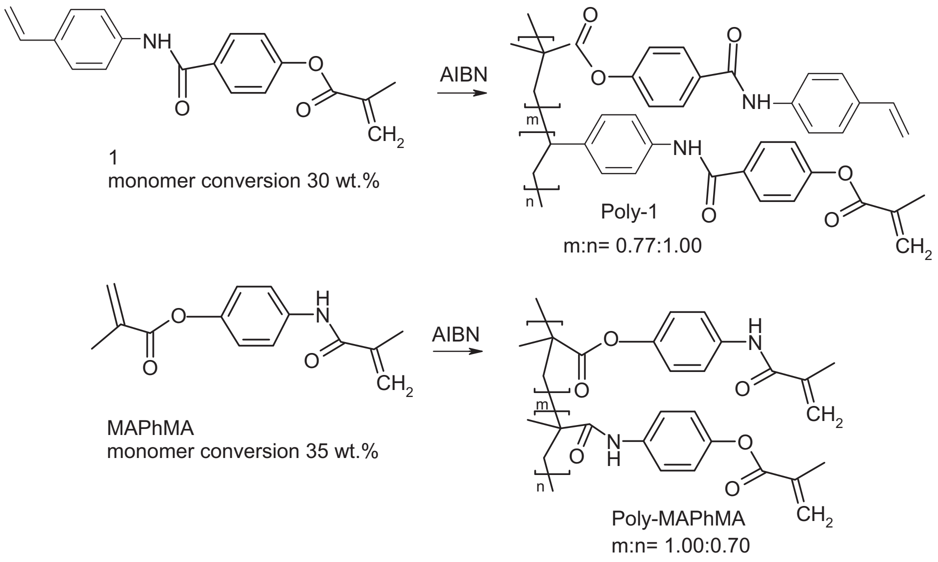
tetrahydrofuran. The yield for **1** was 79 wt.%. Confirmation of their structures was made by ^{1}H NMR and IR spectroscopy as well as elemental analysis.

Syntheses and Photoalignment Peculiarities of Alkene-Functionalized Polymers.

As it was shown in [2], the LC photoaligning properties of poly-**MAPhMA** is strongly dependent on monomer conversion (polymerization yield). At high monomer conversion (usually higher than 40 wt.%) the polymer products are highly crosslinked and so insoluble in organic solvents used for film coating purpose. At lower conversions (typically, 30–40 wt.%) material can be dissolved, but quality of LC alignment is rather poor. Finally, if the conversion is lower than some critical value, acceptable LC alignment was detected. The critical value of monomer conversion for poly-**MAPhMA** was found at around 35 wt.%.

Having a comparable with poly-**MAPhMA** synthetic conditions, homopolymer of **1** was synthesized by thermoinitiated radical polymerization using AIBN as the thermal initiator at 80°C for 45 min up to monomer **1** conversion 30 wt% (Scheme 2). The soluble polymer obtained ($M_w = 25000$, $M_n = 13900$, PD = 1.80) was analyzed by ^{1}H NMR spectroscopy for quantitative determination of each type of free double bonds. The analysis based upon comparison of the resonance intensities of the vinyl proton ($-C\underline{H}=CH_2$) at 6.68 ppm of styrenyl fragment of **1** and the resonance intensities of one from two vinylidene protons ($-CH=C\underline{H_2}$) at 5.89 ppm of *O*-methacryloyl fragment for polymerizations (Fig. 2c).

The resonance intensities ratio of the vinyl proton ($-C\underline{H}=CH_2$) and vinylidene proton ($-CH=C\underline{H_2}$) indicates that, the styrenyl groups were polymerized with higher conversion then *O*-methacryloyl finally giving polymer with two type of side alken functionalities (Sheme 2). Due to more high reactivity of vinyl double bonds the molar ratio vinyl:*O*-methacryloyl free side bonds in the polymer poly-**1** is around 0.77:1.00. In contrast with **1**, monomer **MAPhMA** was polymerized with opposite selectivity and the resulting poly-**MAPhMA** has excess of side free *NH*-methacryloyl groups even at the slightly higher



Scheme 2. The polymerization pathways for studied monomers.

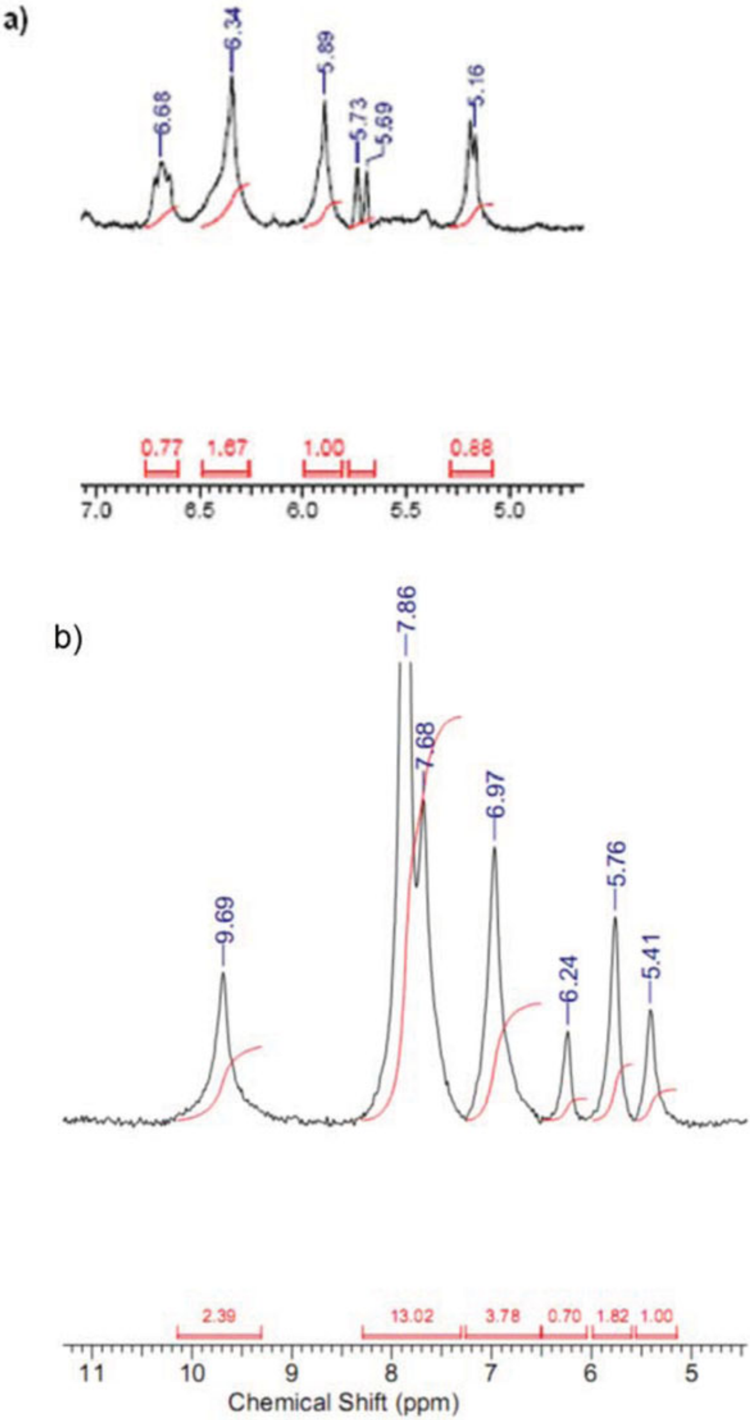


Figure 2. Fragment of ^{1}H NMR spectrum of: a).Poly-**1** at monomer conversion 30 wt.%; b). Poly-**MAPhMA** obtained with monomer conversion 35 wt.%.

Table 2. Polymerization yield, solubility and characteristics of LC alignment for poly-**1** in comparison with poly-**MAPhMA**

Monomer	Yield, wt.%	Solubility	Alignment quality	LC pretilt angle
1	30	DMF	good/excellent	~1.0°
MAPhMA	35	DMF	good/excellent	0.4°–1.2°

conversion (*m*:*n* = 1.00:0.70). This ratio was calculated from the comparison of the resonance intensities of one vinylidene proton ($=\underline{CH_2}$) at 6.24 ppm of *O*-methacryloyl fragment of **MAPhMA** and the resonance intensities of one vinylidene proton ($=C\underline{H}_2$) at 5.41 ppm of *NH*-methacryloyl fragment for poly-**MAPhMA** obtained with monomer conversion 35 wt.% (Fig. 2d).

It can be concluded that an increase of theoretical reactivity ratios (r_1/r_2) difference from 0.07 to 7.00 while maintaining the same charge difference between e_1 and e_2 in **1** and **MAPhMA** did not give any enhancement of selectivity at thermo-induced polymerization of monomer **1**.

Nevertheless, the obtained poly-**1** was tested as LC photoaligning material at the identical testing conditions as for poly-**MAPhMA** [2]. As one can see (Table 2) the poly-**1** alignment layer demonstrates the same grade of LC ZLI 2293 alignment quality and LC pretilt angle closed to poly-**MAPhMA** obtained at the same monomer conversion. The LC cells based on them endure aging at 120°C over 2 h without any noticeable alignment deterioration. It should be noted that at higher conversions of **1** (~40 wt.%) material can be hardly dissolved and quality of LC alignment is rather poor. This observation is completely same to **MAPhMA** type monomers [2] and designed structure of **1** did not give any growth of bifunctional monomer polymerization yield before crosslinking.

Monomer Structure and Polymerization Rate

To elucidate an individual contribution of each type double bond into kinetics of radical polymerization of bifunctional monomer **1**, thermo- and photo-induced polymerization of model monomers was investigated and compared.

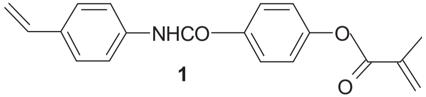
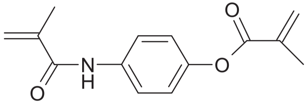
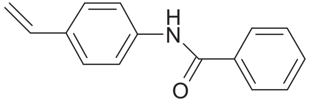
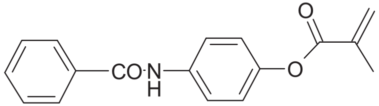
Dilatometry was employed to record conversion versus time curves for bifunctional monomer **1** as well as for model monofunctional monomers **Mod1** and **Mod2**. Thermo-initiated radical polymerizations were carried out at 80°C using 0.172 M concentration of **1**, which corresponds to 10 wt.% monomer in DMF and 0.0023 M concentration of 1wt.% AIBN towards monomer weight. Photo-induced radical polymerizations were examined at 30°C in DMF using benzoin as photo-initiator and the same other conditions. A high pressure mercury lamp was used as a source of UV light.

Calculated from experimental kinetic curves parameter of total polymerization rate V_t was employed for further evaluation of effective constant K_Σ of thermo- and photo-induced polymerization. The constant was evaluated from the equation

$$V_t = K_\Sigma \times [I]^{0.5} \times [M],$$

where [I] - molar concentration of initiator, [M] - molar concentration of monomer [5].

Table 3. Effective polymerization constant K_{Σ} of thermo- and photo-induced polymerization for monomer **1** in comparison with polymerization of **MAPhMA** and model monomers

Bifunctional monomers	 **1**	 **MAPhMA**
K_{Σ}, thermo–induced polymerization, $l^{0.5}/(mol^{0.5} \times s)$	3.84×10^{-3}	5.30×10^{-3} *
K_{Σ}, photo-induced polymerization, $l^{0.5}/(mol^{0.5} \times s)$	0.14×10^{-3}	8.80×10^{-3} *
Model monomers	 **Mod1**	 **Mod2**
K_{Σ}, thermo–induced polymerization, $l^{0.5}/(mol^{0.5} \times s)$	3.60×10^{-3}	4.20×10^{-3}
K_{Σ}, photo-induced polymerization, $l^{0.5}/(mol^{0.5} \times s)$	No polymerization	0.15×10^{-3}

*K_{Σ} values are cited from [8].

The obtained values of K_Σ for both thermo- and photo-induced radical polymerizations of bifunctional monomer **1** and model monomers in comparison with earlier investigated bifunctional monomer **MAPhMA** are presented in Table 3.

Data presented in Table 3 could be summarized in the next conclusions:

- Effective constant K_Σ of thermo-induced polymerization of monomer **1** ($K_\Sigma = 3.84 \times 10^{-3}$ $l^{0.5}/(mol^{0.5} \times s)$) is close to **MAPhMA** ($K_\Sigma = 5.30 \times 10^{-3}$ $l^{0.5}/(mol^{0.5} \times s)$) what could be explained by close K_Σ values of thermo-induced polymerization of individual vinyl and *O*-methacryloyl double bonds estimated from polymerization of monofunctional model monomers **Mod2** ($K_\Sigma = 4.20 \times 10^{-3}$ $l^{0.5}/(mol^{0.5} \times s)$) and **Mod1** ($K_\Sigma = 3.60 \times 10^{-3}$ $l^{0.5}/(mol^{0.5} \times s)$).
- In contrast, K_Σ of photo-induced polymerization of monomer **1** ($K_\Sigma = 0.14 \times 10^{-3}$ $l^{0.5}/(mol^{0.5} \times s)$) is an order of magnitude less than **MAPhMA** ($K_\Sigma = 8.80 \times 10^{-3}$ $l^{0.5}/(mol^{0.5} \times s)$). This could be explained by inactivity of styrene-type double bond at the photo-induced polymerization. It was found that model monomer **Mod1** could not be polymerized at all.
- Bifunctional monomer **1** due to combination of two factors such as higher reactivity of vinyl double bond in comparison with *O*-methacryloyl one (estimated by reactivity ratios $r_1 = 0.053$ and $r_2 = 0.74$, Table 1) and simultaneous inactivity of styrene-type double bond at the photo-induced polymerization, photopolymerizes with effective constant value almost the same to *O*-methacryloylmodel **Mod2** (0.15×10^{-3} $l^{0.5}/(mol^{0.5} \times s)$) and the polymerization stops at monomer conversion less than 5 wt.%.

As a result, photo-induced radical polymerization of **1** could not be employed as possible method for elimination of crosslinking at high monomer conversions as it was employed in a case of substituted arylmethacrylates which have the second double bond in tetrahydrophthalic fragment [9].

4. Conclusions

Bifunctional monomer 4-{[(4-vinylphenyl)-anilino]carbonyl}phenyl-2-methylacrylate (**1**) would open a new class of bifunctional monomers suitable for preparation of LC photoaligning alkene-functionalized polymers. An increase of theoretical reactivity ratios (r_1/r_2) difference of its *O* -methacryloyl- and styrenyl-type double bonds from 0.07 to 7.00 while maintaining the same charge difference between e_1 (0.82) and e_2 (−0.98) did not give any enhancement of selectivity at thermo-induced polymerization of monomer **1** in comparison with 4-(methacryloylamino)phenyl methacrylate (**MAPhMA**).

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