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Initiation of polymerization of pure 2-hydroxyethylmethacrylate under shock wave compression

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Key words: radical polymerization, shock wave compression, HEMA, oligomers

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Abstract

The ability to initiate chemical reactions without needs of potentially hazardous solvents, catalysts and radical initiators is of key importance for modern biotechnologies and medicine. In this study, laser-driven shock wave compression was performed on pure 2-hydroxyethylmethacrylate (*HEMA*) free of any additives in order to trig the polymerization process. We showed that the generated peak shock-wave pressure in the range between 2 and 5.5 Gpa permitted initiation of radical polymerization and formation of poly-*HEMA* oligomers. The reaction begin with diradicals $HEMA_2^{\bullet\bullet}$ appeared at the peak pressure and, by reactions with *HEMA* monomers, forming $HEMA_3^{\bullet\bullet}$ diradicals on the 20-ns timescale. The chain propagation further occurs on the sub-µs timescale by associations of the trimer diradicals, which, because of a smaller reactivity, do not react with *HEMA* monomers. The proposed method can be considered for scale-up of polymerization process of ultrapure organics.

1. Introduction

High pressures (HP) processing is an effective way to realization of ultrapure polymers via radical activations, otherwise requiring addition of thermo- or photo- initiators to a monomer solution. One of the most significant advantages of HP chemistry is the ability to initiate chemical reactions without the need of potentially hazardous solvents, catalysts, or radical initiators [1-3]. In the past, the HP polymerization of small, unsaturated hydrocarbon molecules in liquid and crystalline phases has been extensively studied both experimentally and theoretically [4, 5]. However, due to the reduction of molecular mobility with increasing pressure, this approach is restricted to monomers in ordered phases, in which the polymerization does not require large molecular rearrangements. Pure polymer and polymer-based materials may have a great potentiality for applications in biomedicine, photonics, etc. In the same time, any practical large-scale implementation of this method and materials is limited by a very small reaction volumes characteristic of GPa high-pressure cells. The research on scale-up of the HP-polymerization process is therefore of high actuality.

To produce and apply high pressure on a given substance in order to induce a chemical reaction, there are two main approaches: static and dynamic. For the generation of high static pressure, the application of a mechanical force to the surface of the sample that is placed in a vessel such as diamond anvil cells (DAC). Generally, the DAC allows a pressure limit above 10 GPa and to combine with probing techniques for performing in situ observations of materials [6-8] . Shock wave compression created by gas cannons, pulsed lasers, high pulsed electrical currents, and explosions is the basis for dynamic techniques. Today, it is possible to generate pressures up to 5 GPa and even higher in the condensed matter [9]. In such extreme

conditions, atomic bonding and electronic structure of species significantly changed at the ns-μs timescale compared to that in ambient conditions, creating opportunity for the activation of chemical transformations. The application of the dynamic HP processing to organic polymerization can be of interest. The main issue in this respect concerns the process kinetics, which remains largely unknown.

The poly-2-hydoxyethyl methacrylate (pHEMA) is a biocompatible material, which is considered in biomedicine for fabrication of artificial coronae, contact lenses, etc. [10-13]. It can be photopolymerized in ambient conditions using a photoinitiator [14] or without catalyst under moderate HP (< 1GPa) [15]. Recently, an important improvement of the HP polymerization process of 2-hydroxyethylmethacrylate (HEMA) has been developed in our institution [16], which was realized in a HP DAC cell by applying two-stage static HP processing. An effective polymerization has been achieved also in HEMA-based organic-inorganic hybrid materials using this method with unprecedentedly high monomer conversion yield, well above that obtained via conventional methods using thermal and photo initiators [17] at the same concentration of the inorganic component. In this work, we applied a laser driven shock-wave compression to investigate the possibility of initiation of bulk HEMA polymerization.

2. Materials and Methods

2.1. Chemicals

The monomer was supplied by Sigma Aldrich (St. Louis, MO, USA) and provided in the liquid form with the following reference: 2-hydroxyethyl methacrylate (HEMA), CAS:

868-77-9, ≥99%.

2.2. Laser-driven shock wave compression

The schema of the laser-driven shock compression (LSC) experiment depicted in Fig. 1a has been descripted in details in Ref. [18]. In short, LSC was obtained after receiving laser pulses provided by BIVOJ laser system of HiLase with energy E between 0.5 and 7 J (λ=1030 nm, pulse duration τ~10 ns) on a target with the square spot area of σ=1.94x1.94 mm². In multiple LSC experiments, laser shots have a repetition rate of 0.5 Hz. The HEMA samples were places in black-painted aluminum cells and mounted on a holder of robot Fanuc M-20iA/20M (load of up to 20 kg with 0.08 mm positioning repeatability). Water was used as a confining medium. In order to ensure stable and reliable treatment, the laser energy and beam shape were monitored by a diagnostic system based on two cameras (MANTA G-125, Allied Vision) and a calibrated photodiode. The shock wave pressures $2 \leq P_{max} \leq 6$ GPa obtained in the experiments are shown in Fig. 1b as a function of laser power density ($E/\tau\sigma$).

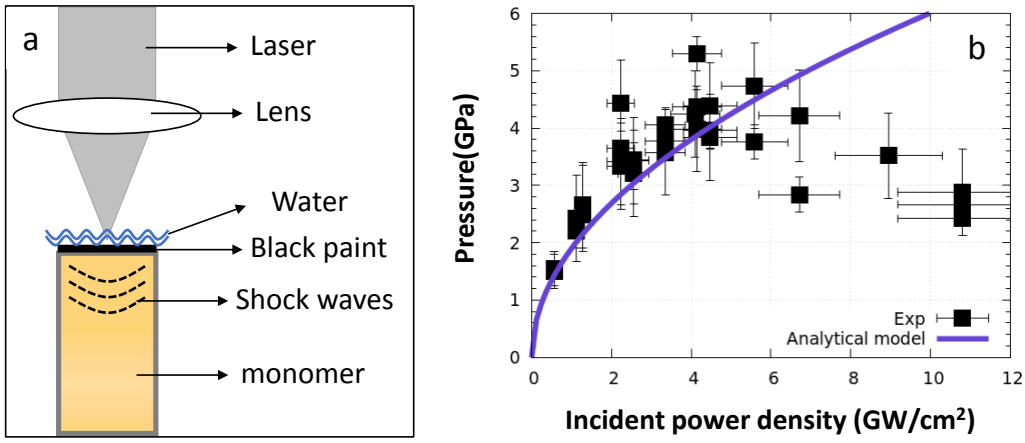

Figure 1. Schematic configuration of laser shock peening (a) and correlation between laser incident power density and generated pressure (b).

The measured pressure kinetics is shown in Fig. 2. It includes a rather sharp peak P_{max}

followed by a longer tail of the pressure decrease down to ~0.1 GPa level. The absolute values depend on the applied laser energy in a pulse. For example, in case of the maximum energy 2.5 J/pulse used in present experiments, the peak pressure of P_{max}=5.5 was attained with the full width at half maximum of $t_{1/2}$≈15 ns and relaxation time below 0.1 GPa pressure of t_r=50 ns. These results are in agreement with previous experiments and modelling of the water-confined regime [9]. The pressure kinetics will be useful for understanding of the relevant reaction rates.

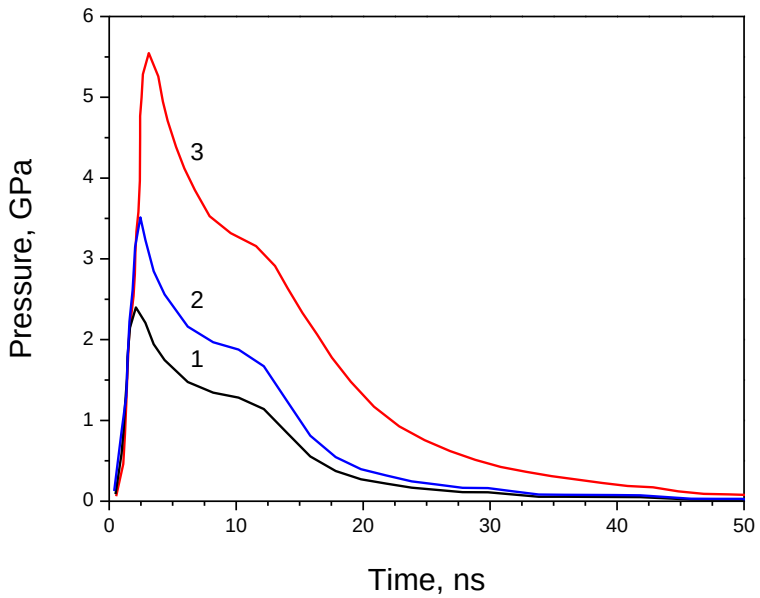

Figure 2. Pressure dependence in LSC experiments at laser pulse energy of 0.5 J (1), 1 J (2) and 2.5 J (3).

2.3.Characterization

The molecular weight and polydispersity of the polymeric chains have been evaluated using gel penetration chromatography (GPC). After been exposed to LSC, the closed cells with HEMA solutions were kept in a glove box under inert gas atmosphere until GPC analysis. For sample preparation, 50 μL of the monomer liquid treated by LSC process was

mixed with 400 µL tetrahydrofuran (THF) in order to dissolve the possible solid polymer. Dionex Ultimate 3000 GPC apparatus was equipped with a Polymer Laboratories MesoPore 3-µm column (300x7.5 mm) in an isothermal oven (35 °C) in series with a UV-visible detector (detection wavelength 210 nm). The eluent flow rate (HPLC grade tetrahydrofuran, THF) was set to 1.0 mL/min. A particular attention was paid to the calibration of column for low molecular weights 100-1000 g/mol. The calibration, shown in Fig. 3, was performed using a set of polystyrene standards (EasiCal PS-2 Polymer Laboratories, Mw = 580–20650 g/mol range) with a very narrow polydispersity, including ethylene glycol dimethacrylate (CAS: 97-90-5, molecular weight 198.22 g/mol) and lauryl methacrylate (CAS: 142-90-5, molecular weight 254.41 g/mol). Data acquisition and analysis were performed with Chromeleon V 6.80 software supplied by Dionex.

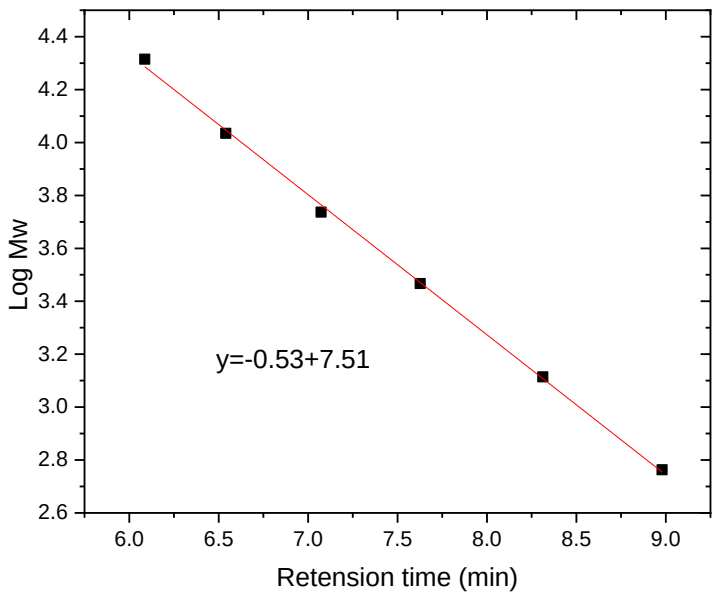

Figure 3. Calibration curve for gel penetration chromatography.

3. Results and Discussions

Under static HP, it has been previously shown [16] that the conversion yield of HEMA monomers exhibits a threshold at P=2 GPa and reaches a plateau with a maximum of 90% at pressures P≥6.5 GPa. This range of pressures was assigned to the formation of $HEMA_2^{\bullet\bullet}$ diradicals

$$2HEMA \rightarrow HEMA_2^{\bullet\bullet} \ , \qquad (1)$$

which activate the polymerization process on the second stage, when pressure is reduced below 2 GPa, which fits the glass transition pressure, letting the monomers free to move forming the polymer chains after the termination process:

$$HEMA_2^{\bullet\bullet} + nHEMA \rightarrow HEMA_{n+2} \qquad (2)$$

In LSC process, the peak pressure was controlled via laser energy according to Fig. 1b. The chromatograms of liquid pristine HEMA and HEMA samples after the exposition to LSC with different laser energies are shown in Fig. 4. The THF impurities and HEMA monomer (molecular weight 130 g/mol) were respectively detected at 11.64 and 9.89 min (Fig. 4a). With low laser energy of 0.5 J providing the peak pressure of 2.2 GPa (close to the diradicals activation threshold), a very weak GPC peak with the molecular weight of ~280 g/mol could be seen, which corresponds to the molecular dimers $HEMA_2$. By increasing the laser energy to 1 J (3.5 GPa), a peak of the larger chain with the molecular weight of 400 g/mol assigned to $HEMA_3$ became intense and a new rather weak peak appeared with molecular weights of 785 g/mol (8.64 min), which was assigned to $HEMA_6$. With even higher laser energy of 2.5 J, which correspond to the highest shock wave pressure of P=5.5 GPa, the peak of $HEMA_6$ intensified and three more peaks at 640 g/mol (strong), 1035 g/mol (weak) and 1180 g/mol

(weak) made their appearance, which were respectively assigned to $HEMA_5$, $HEMA_8$ and $HEMA_9$ chains. These observations allowed proposing the reaction scheme of the oligomers formation, which is discussed below.

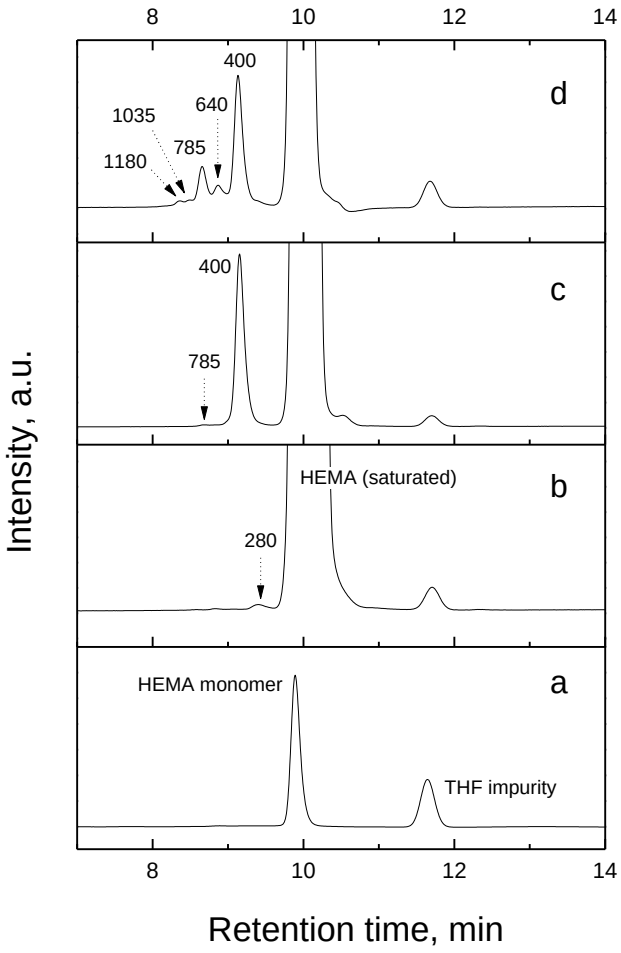

Figure 4. GPC patterns of pristine HEMA (a) and HEMA samples after LSC processing with one laser pulse of energy 0.5 J / P=2.2 GPa (b), 1 J / P=3.5 GPa (c) and 2.5 J / P=5.5 GPa (d). Molecular weights are indicated in g/mol units.

The Flory mechanism has been previously adopted for the self-initiation of polymerization in methacrylates (M) [19]. According to this mechanism, two monomers M combine to form a diradical $^{\bullet}M^{\bullet}_2$, which reacts with a third monomer molecule to generate two monoradicals

$M^{\bullet}_2$ and $M^{\bullet}$ able to initiate polymerization. In agreement with this mechanism and our observations under static HP [16], we postulate that the diradical $HEMA_2^{\bullet\bullet}$ is readily formed under high pressures above 2 GPa and initiates polymerization process when pressure is released below 2 GPa, i.e. that of the glass transition. This $HEMA_2^{\bullet\bullet}$ diradical is formed almost instantaneously (Eq. 1) via tail-to-tail addition of monomers occasionally having a suitable alignment in the bulk glassy HEMA. By increasing LSC peak pressure, the yield of the $HEMA_2^{\bullet\bullet}$ diradicals increased. The vanishing of the GPC peak corresponding to $HEMA_2$ dimers (Fig. 4c-d) supports our hypothesis of the very fast reaction kinetics of the dimer diradicals with surrounding monomers, forming unsaturated trimers:

$$HEMA_2^{\bullet\bullet} + HEMA \rightarrow HEMA_3^{\bullet\bullet} \qquad (3)$$

where the termination reaction $HEMA_3^{\bullet\bullet} \rightarrow HEMA_3$ probably occurs via intra molecular processes as observed in the case of MMA self-polymerization at high temperatures (>100°C) [20]. Here we postulate the formation of the trimer diradicals $HEMA_3^{\bullet\bullet}$ because of their preferential association in larger oligomeric chains, as it will be discussed below. Taking into account the characteristic LSC pressure kinetics (Fig. 2) and relevant range of pressures 0.1<P<2 GPa enabling effective HEMA monomers conversion [16], one can conclude that reaction (3) is completed within 50-ns time. Contrary to the mechanism proposed at ambient pressure [21], it seems however that the steric hindrance induced by the HP environment prevents the formation of monoradicals by transfer or abstraction of H from the diradical to a third monomer. On the other hand, Fig. 4c shows that kinetics of formation of the larger chains is slow and cannot be completed during the short time of ~100 ns. Obviously, the appearance of large oligomers at P=5.5 GPa (Fig. 4d) is due to an increase of concentration of

the activated species. Apparently, reactivity of the trimer diradicals is smaller compared to that of the dimer diradicals (3), prohibiting their fast association with monomers into $HEMA_4$. However, their association with other activated species can still be effective:

$$HEMA_3^{\bullet\bullet} + HEMA_2^{\bullet\bullet} \rightarrow HEMA_5^{\bullet\bullet} \quad (4)$$

$$HEMA_3^{\bullet\bullet} + HEMA_3^{\bullet\bullet} \rightarrow HEMA_6^{\bullet\bullet} \quad (5)$$

This conclusion is supported by our experimental observations of $HEMA_5$ (640 g/mol) and $HEMA_6$ (785 g/mol) oligomers in Fig. 4d. The smaller GPC peak of $HEMA_5$ compared to $HEMA_6$ is then explained by the strong $HEMA_2^{\bullet\bullet}$ diradicals consumption in process (3), which made the trimer diradicals more abundant in the solution. The same consideration may also explain a weak formation of tetramer $HEMA_4$ from two dimer diradicals. The subsequent reactions leading to $HEMA_8$ and $HEMA_9$ oligomers formation are due to the further associations of $HEMA_3^{\bullet\bullet}$ with species formed in processes (4) and (5):

$$HEMA_3^{\bullet\bullet} + HEMA_5^{\bullet\bullet} \rightarrow HEMA_8^{\bullet\bullet} \quad (6)$$

$$HEMA_3^{\bullet\bullet} + HEMA_6^{\bullet\bullet} \rightarrow HEMA_9^{\bullet\bullet} \quad (7)$$

As a result of the appeared “magic numbers” n=2, 3, 5, 6, 8, 9 of $HEMA_n$ oligomers in the initial polymerisation propagation, we propose a generalised schema of the HEMA chains growth at high pressures based on the diradicals association:

$$kHEMA_2^{\bullet\bullet} + mHEMA_3^{\bullet\bullet} \rightarrow HEMA_{k+m}^{\bullet\bullet} \qquad (k, m = 1, 2, 3, \ldots) \quad (8)$$

Furthermore, we ascribe diradical nature to these species, because of their selective association with $HEMA_2^{\bullet\bullet}$ and $HEMA_3^{\bullet\bullet}$ diradicals, despite of a low concentration in the reaction solution. As mentioned above, in all these reactions the terminations reaction may occurs via intra molecular processes. We notice that the GPC analysis used in the present

studiy do not allow us concluding about a specific termination mechanisms involved in the formation of the observed oligomers. However, since the chains propagation takes place at pressures below that of the glass transition, we conclude that a longer confinement time (t>>100 ns) of the solution at moderate pressures of 0.1<P<2 GPa would be required in order to effectively complete the polymerization process. The effect of a longer exposure to these relatively lower pressures could be seen in Fig. 5, where the GPC sample analysis after an exposition to 1 and 5 laser pulses of 2.5-J energy is shown.

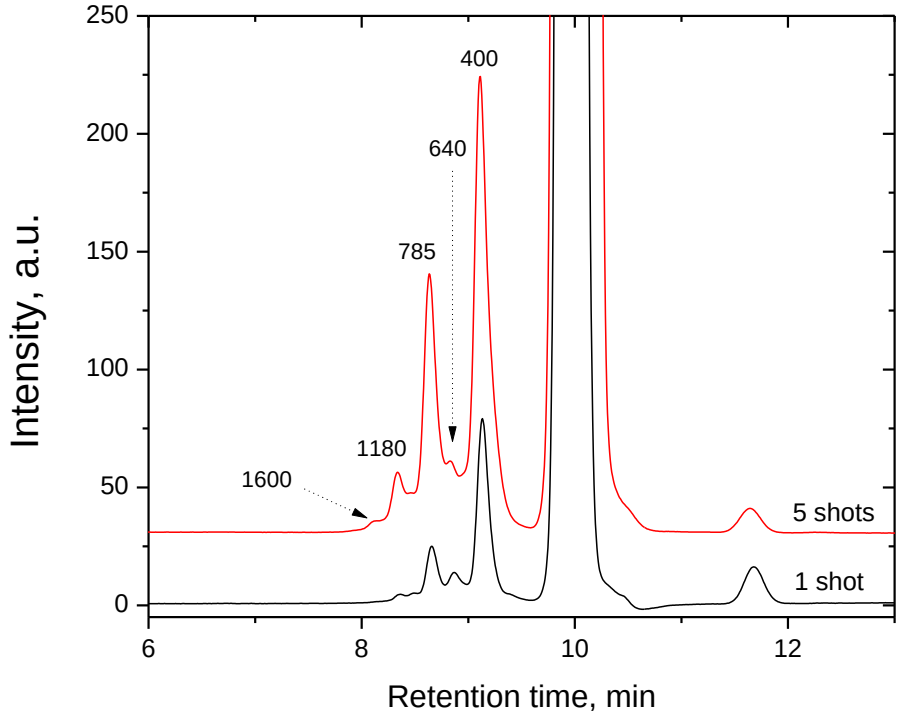

Figure 5. GPC patterns of HEMA samples after LSC processing with 1 and 5 laser pulses of energy 2.5 J (peak pressure 5.5 GPa).

In case of an additive process, the GPC patterns could not be expected to change much. In contrast, relative intensities of the peaks at smaller times, which correspond to larger HEMA

chains were significantly intensified with an increase of the shock waves number. In particular, while relative intensities of $HEMA_3$ and $HEMA_5$ remain the same, those of $HEMA_6$ and $HEMA_9$ increased significantly. The last corresponds to the 5-times longer reactions time of the trimer diradicals in processes (5) and (7). Furthermore, a shoulder appeared at 8.15 min, which corresponds to chains with the molecular weight of 1600 g/mol. According to Eq. 8, this corresponds to a successive association of four trimer diradicals $HEMA_3^{\bullet\bullet}$ (m=4), which last step is:

$$HEMA_3^{\bullet\bullet} + HEMA_9^{\bullet\bullet} \rightarrow HEMA_{12}^{\bullet\bullet} \qquad (9)$$

All these observations support the conclusion on the polymerisation propagation at moderate pressures (below 2 GPa) in course of diradicals association. We notice in this connection that the m-series of Eq. 8 involving $HEMA_3^{\bullet\bullet}$ diradicals dominate the initial chains propagation process. In contrast, the chains propagation by association of $HEMA_2^{\bullet\bullet}$ diradicals is of lower probability because of their rapid reaction (3) with the surrounding monomers, completed on the sub-μs timescale, giving rise to $HEMA_3^{\bullet\bullet}$ diradicals.

The methacrylate diradical $^{\bullet}M^{\bullet}_2$ has been shown to be key compound in the initiation mechanism of the spontaneous polymerization of methyl methacrylate [22]. However, even at high temperature the reaction is extremely slow and showed a low conversion of monomer to polymers. On the other hand, in methyl methacrylate an efficient conversion of monomers to oligomers such as unsaturated dimers and trimers or 1,1,2,2-tetrasubsitutes cyclobutanes has been observed [20, 23-25] These earlier studies have suggested that diradicals cannot grow into two directions, and they have to be terminated first on one side by a transfer reaction in order to yield high polymers. The low polymerization yield is then the result of a competition

with the efficient self-termination processes of dimers and trimers diradicals. The spontaneous polymerization therefore propagates at ambient conditions via transfer agents forming polymerizable monoradicals. Our results clearly indicate that that the reactivity is affected by high pressures where the polymerization propagates through the oligomer diradicals association towards much larger polymeric species.

Because of a large affected volume compared to common high-pressure cells and attainable 5 GPa pressures required for an effective radicals activation, LSC can be considered for the scale-up of HPR polymerization process [16]. Unfortunately, the short shock wave exposure time to the relevant moderate pressures of ≤0.1 μs is not sufficient to accommodate the chains propagation kinetics and, consequently, it does not allow effectively completing the polymerization process. However, the required pressure of the sub-GPa range is not restrictive and could be achieved in large volumes using conventional available techniques [26]. Therefore, combining shock-wave compression with post-exposure to moderate static pressures could be main steps of a large-scale process for ultrapure polymers production.

4. Conclusions

We report on initiation of the 2-hydroxyethylmethacrylate (HEMA) polymerization process via laser-driven shock-wave compression. The gel penetration chromatography (GPC) analysis of the chains mass was performed after the bulk monomer solution exposure to different laser energies and number of pulses. We showed that generated pressure in the range between 2 and 5.5 Gpa permitted initiation of the radical polymerization with the formation

of oligomers $HEMA_n$ (up to n=12). We confirmed the process initiation with $HEMA_2^{\bullet\bullet}$ diradicals and showed that they rapidly react on the 15 ns timescale with HEMA monomers forming $HEMA_3^{\bullet\bullet}$ diradicals. The subsequent dominant chain propagation by association of $HEMA_3^{\bullet\bullet}$ diradicals was evidenced. The shock-wave compression can be considered for the scale-up of ultrapure organic polymerization process.

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