Self-Initiation of Photopolymerization Reactions Using Halogenated (Meth)Acrylates

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Photoinitiators seem to be an unavoidable constituent of UV-curable formulations. Unfortunately, they cause several problems during application of the cured coatings. But do we really need them? This study deals with an investigation of various halogenated acrylates and methacrylates. At least some of them can form radicals upon irradiation and may lead to self-initiation of the polymerization of UV-curable formulations. This paper presents investigations by real-time Fourier transformed infrared (RT-FTIR) spectroscopy, laser photolysis and quantum chemical modeling on the initiation behavior and kinetics of the photopolymerization of such systems.

Introduction

Photoinitiators are an essential component of any UV-curable formulation that is required to induce a photopolymerization reaction upon UV exposure. The absorption of light by those molecules leads to the formation of reactive species and, as a consequence, to the initiation of a photopolymerization reaction. Ninety percent of all commercially available photoinitiators are radical initiators since the biggest part of UV formulations is based on acrylates and methacrylates. Photoinitiators have

to satisfy some requirements such as high absorption at the wavelength of exposure; high quantum yield for the formation of initiating species; and high reactivity of the radicals toward the monomer. For example, most photoinitiators are designed in such a way that their absorption spectra match well with the emission spectrum of the widely used medium-pressure mercury lamps. However, although photoinitiators seem to be necessary for the initiation of photopolymerization reactions, they have a number of serious drawbacks with respect to the application of the cured layers—strong smell, yellowing, migration, and limited weathering and aging stability. Moreover, their high costs are unfavorable from a commercial point of view. So, the question arises—do we really always need conventional photoinitiators?

Recently, extensive efforts have been made to search for alternatives to the use of photoinitiators. It has been observed that photopolymerization reactions can be initiated by direct excitation of (meth)acrylates via irradiation in the UV-C or vacuum ultraviolet (VUV) range of the spectrum.[1,2] Excimer lamps, low-pressure mercury lamps or UV lasers may be used as light sources. Unfortunately, the penetration depth

of short-wavelength radiation into acrylate layers is rather low since their extinction coefficients are quite high in this region (like those of most other organic matter). Depending on the specific wavelength, it is in the order of some hundred nanometers up to a few micrometers.[3] Even if some bleaching of the acrylates during irradiation is taken into account, this means that only rather thin layers can be through-cured in this way.

Another possibility is the use of thiol-ene systems that proceed via a successive propagation/chain transfer mechanism resulting in a step-growth polyaddition reaction. The UV light causes the formation of thiyl radicals from the thiol that can start the polymerization.

Moreover, donor-acceptor systems have been developed which use N-substituted maleimides as acceptor monomers, whereas vinyl ethers, alkoxy styrenes or other monomers serve as electron donors. In addition, (bis)maleimides can be also copolymerized with diacrylates via hydrogen abstraction from the triplet state of excited maleimides.

Alternatively, it has been discovered that several monomers with specific molecular structures tend to self-initiation of a polymerization reaction upon exposure to UV light. For example, vinyl acrylate was found to be its own photoinitiator.[4] Two mechanisms are probably involved in the initiation step—direct cleavage of the molecule and formation of biradicals arising from internal cyclization in the triplet state.

Another class of monomers that might be regarded as potentially self-initiating are halogenated acrylates and methacrylates. During the development of lacquer formulations with a very high refractive index, it has been observed accidentally that the varnish showed considerable

reactivity even without the addition of a conventional photoinitiator. Detailed analysis of the components of this formulation revealed that a pentabrominated aromatic acrylate (that was added at rather low concentration to the varnish) was responsible for that. A subsequent study[5] on a number of aromatic acrylates with different degrees of bromination has shown that photochemically induced homolysis of carbon-bromine bonds leads to the formation of bromine and tetrabromophenyl radicals that may both initiate a polymerization reaction.

In the present study, we extended these investigations to a broader spectrum of halogenated (i.e., fluorinated, chlorinated and brominated) acrylates and methacrylates in order to find out how the different substituents of the aromatic ring influence the initiation of a photopolymerization reaction upon exposure to UV light. Moreover, some aliphatic-halogenated acrylates were included in the investigations in order to study the effect of different molecular structures. The experimental measurements carried out by RT-FTIR spectroscopy and laser photolysis were

complemented by a detailed study based on quantum chemical modeling in order to get deeper insights into the mechanisms involved in the self-initiation of the photopolymerization of halogenated monomers.

Experimental

Materials and Sample Preparation

The following halogenated acrylates and methacrylates were used in this study (Figure 1): pentabromophenyl acrylate (PBPA), pentafluorophenyl acrylate (PFPA), pentabromobenzyl acrylate (PBBA), pentafluorobenzyl acrylate (PFBA), pentabromophenyl (meth)acrylate (PBPMA), pentachlorophenyl (meth)acrylate (PCPMA), pentafluorophenyl (meth)-acrylate (PFPMA), 2,3-dibromopropyl acrylate (DBPA) and perfluoroctyl acrylate (PFOA). Moreover, their non-halogenated counterparts were studied for comparison. All monomers were purchased from ABCR (Karlsruhe, Germany).

For investigations by RT-FTIR spectroscopy, the halogenated monomers were added at concentrations of 1 or 5 wt.-% to a model lacquer formulation which

FIGURE 1

Halogenated acrylates and methacrylates used in this study

PXBA PXPA

PXBMA PXPMA

X = Br, Cl, F, H   P(M)A — Phenyl (Meth)Acrylate
                   B(M)A — Benzyl (Meth)Acrylate
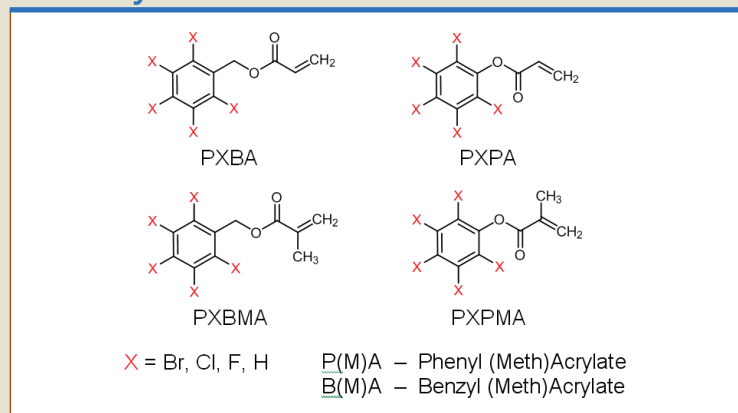
consisted of 70 wt.-% of an aliphatic urethane diacrylate (EB 270) and 30 wt.-% TPGDA (both from Cytec). No conventional photoinitiator was added. Only for comparison, the neat model formulation was provided with benzildimethyl ketal (BDMK, Irgacure 651; BASF) as the photoinitiator. All prepared blends were treated in an ultrasonic bath for 15 minutes in order to ensure complete dissolution of all components as well as to remove air bubbles.

RT-FTIR Measurements

The RT-FTIR measurements were performed with a Varian 670 spectrometer equipped with a Golden Gate diamond ATR unit and an MCT detector. Depending on the specific experimental conditions, the time resolution was set to either 17 or 50 milliseconds. FTIR spectra were recorded with a spectral resolution of 16 cm[-1]. Kinetic profiles were obtained by integration of the acrylate band at 810 cm[-1] after previous application of a fitting and band separation procedure.

In order to get layers with low and reproducible thicknesses, a drop of the sample was put on the surface of the ATR crystal and covered by a quartz plate with a mold cavity (obtained by ion beam etching) forming a gap of ~ 4.5 μm. A four-channel delay/pulse generator (Stanford Research Systems) was used as trigger for the synchronization between the FTIR spectrometer and the shutter (Vincent Associates) that controls the irradiation of the sample.

Irradiation was carried out with a medium-pressure mercury lamp. The irradiance at the surface of the diamond was 120 mW cm[-2]. The lamp was provided with a water filter to block IR radiation in order to prevent heating of the sample. Most RT-FTIR measurements were carried out with an edge filter (WG280, center wavelength 280 nm, remaining

FIGURE 2

UV spectra of chlorinated (PCPMA), brominated (PBPMA, PBBA), and fluorinated (PFBA) aromatic acrylates and methacrylates, including one of the aliphatic acrylates (DBPA)
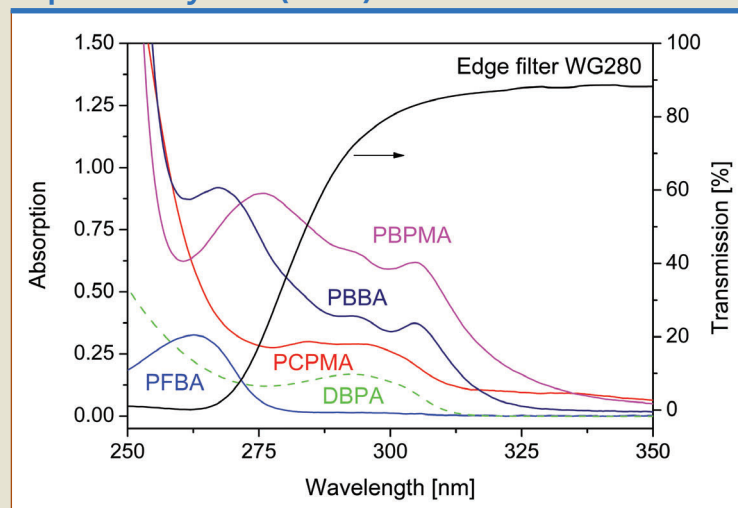
intensity 100 mW cm[-2]). Some investigations were performed with a 313 nm interference filter (Andover) that led to a reduction of the irradiance to 4.5 mW cm[-2]. Irradiation was carried out under either atmospheric or inert conditions. In order to achieve stable inert conditions, the sample compartment was purged with dry nitrogen for five minutes prior to the start of the measurements. A comprehensive description of the method is given in a previous publication.[6]

Laser Photolysis

The laser photolysis setup comprised a 308-nm XeCl* excimer laser (pulse width 7 ns) and a pulsed xenon short-arc lamp (Osram) which supplied the analyzing light. The transient recording electronics consisted of a photomultiplier (Hamamatsu) and a 500 MHz digitizing storage oscilloscope (Tektronix), which ensured an adequate time resolution. All experiments were carried out in flow-through cuvettes. The actinometry

was performed by monitoring the yield of the benzophenone triplet at 520 nm after excitation of benzophenone/acetonitrile solutions.

Quantum Chemical Modeling

Calculations were carried out with the program packages Gaussian 03 and Jaguar (Schrödinger LLC). Molecular geometries were optimized applying the density functional theory (DFT) with B3LYP hybrid functional. The electronic transition spectra of the transients were calculated in vacuum with the time-dependent (TD) DFT B3LYP/ 6-31+G(d,p) method.

Results and Discussions

UV Spectra

At first, the UV spectra of all halogenated compounds were recorded. For this purpose, they were dissolved in acetonitrile at a concentration of 5x10[-3] M. Figure 2 shows the UV spectra of a selection of chlorinated (PCPMA), brominated (PBPMA, PBBA) and fluorinated (PFBA) aromatic acrylates and

FIGURE 3

Kinetic profiles of photopolymerization reactions
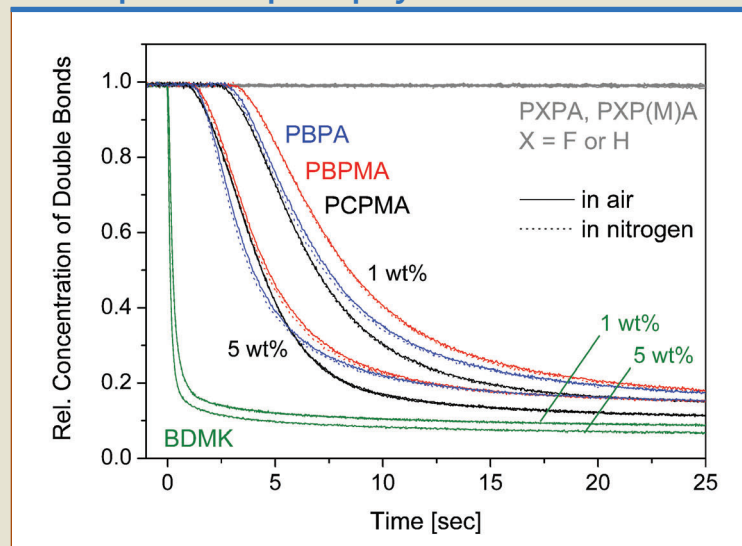
methacrylates as well as that of one of the aliphatic acrylates (DBPA). The spectra of the missing brominated and fluorinated monomers that were omitted for reasons of clarity resemble those of the related materials.

It can be clearly seen that the UV spectra of the brominated and chlorinated (meth)acrylates extend to far beyond 300 nm, whereas the fluorinated acrylates (including PFOA) do not show significant absorption above ~ 280 nm. The bands with a wavelength higher than 300 nm are related to bromine-phenyl and chlorine-phenyl bonds. The corresponding fluorine-phenyl bonds are situated at much lower wavelengths. So, much higher UV energy is required for the excitation of these bonds in the fluorinated (meth)acrylates in comparison to the related bonds in brominated and chlorinated monomers.

Basically, two different mechanisms can initiate a photopolymerization reaction of halogenated acrylates if the whole spectrum of the mercury lamp without any filters is used for irradiation. Light with a wavelength

below about 250-260 nm may lead to a direct excitation of the acrylic double bonds.[1,2] This mechanism is completely independent of the halogenation of the monomers. Alternatively, if direct excitation is excluded by the use of suitable filters, (i.e., if irradiation is carried out in the spectral range around and beyond 300 nm) the excitation of halogen-phenyl bonds exclusively may lead to the generation of radicals and the initiation of the polymerization reaction. In order to prevent direct excitation of the (meth)acrylate molecules, all RT-FTIR measurements in the present study were performed using the edge filter or, in some cases, the interference filter. The edge filter completely blocks light below 260 nm. Its transmission spectrum is shown in Figure 2.

Real-Time FTIR Measurements

The kinetic behavior of the model lacquer containing either halogenated aromatic (meth)acrylates or their unhalogenated equivalents upon UV irradiation was followed using RT-FTIR spectroscopy. Figure 3 shows the kinetic profiles of the photopolym-

erization reaction in some of these systems. Investigations were carried out under either atmospheric or inert conditions. The kinetic curves of the model varnish containing BDMK are shown for comparison.
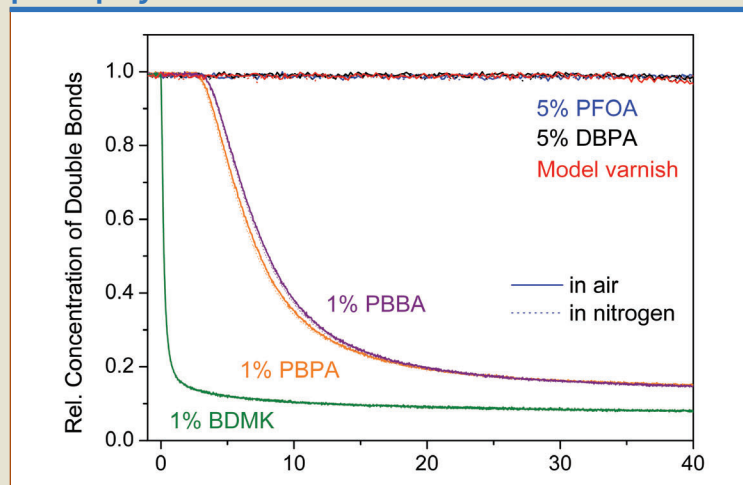
It is obvious that a conversion of the acrylic double bonds is only achieved for the formulations containing brominated or chlorinated (meth)acrylates, whereas no reaction was observed for the formulations with fluorinated (meth)acrylates or the non-halogenated pendants. This indicates that reactive species are generated only in the former systems. An increase in the concentration of the brominated and chlorinated (meth)acrylates in the model lacquer from 1 to 5 wt.-% leads to a slight rise in the reaction speed and a moderate decrease in the inhibition time, but only to a minor increase in the resulting conversion. Moreover, the difference between kinetic curves recorded in air or nitrogen, respectively, is not significant which indicates a rather low sensitivity of the reactive species toward oxygen.

Certainly, the efficiency of the brominated and chlorinated monomers is lower than that of the conventional photoinitiator. The reference varnish with BDMK reacts considerably faster; does not show an appreciable inhibition time; and achieves a higher conversion. An increase in the BDMK concentration does not lead to significant improvements in the reactivity of the system. This means that even 1 wt.-% BDMK produces sufficient radicals to attain an effective photopolymerization.

Figure 4 compares the efficiencies of aromatic and aliphatic halogenated acrylates to initiate a photopolymerization reaction. The formulations containing aromatic acrylates (i.e., brominated phenyl or benzyl acrylate, respectively) do not show any difference with respect to the kinetics of the photopolymerization

FIGURE 4

Comparison of the efficiencies of aromatic and aliphatic halogenated acrylates to initiate a photopolymerization reaction

which is due to the much lower intensity of the incident monochromatic radiation compared to that of the complete emission spectrum of the mercury lamp above ~ 280 nm. All systems with brominated acrylates and methacrylates led to identical kinetic behavior, which confirms the results reported above. The formulation with PCPMA shows a much longer inhibition time, and its polymerization speed is lower. The fact that only brominated and chlorinated monomers lead to the initiation of a polymerization reaction in this spectral range is in correlation with the UV spectra plotted in Figure 2. However, the extinction coefficients of these four monomers at 313 nm vary from 780 l mol^{-1} cm^{-1} (PBPMA) via 350 l mol^{-1} cm^{-1} (PBBA, PBPA) to 230 l mol^{-1} cm^{-1} (PCPMA), which indicates different efficiencies of the radical generation process.

The results obtained by RT-FTIR spectroscopy clearly show that brominated and chlorinated aromatic and methacrylates may serve as rather efficient photoinitiators of a photopolymerization reaction, whereas

reaction. Consequently, the slightly longer distance between the aromatic ring and the acrylic double bond in the molecule, which may lead to somewhat better decoupling of both structures, has no effect on the efficiency of the generation of radicals. Moreover, the comparison with Figure 3 also shows that the differentiation between acrylate and methacrylates is insignificant. At equal concentration, the three monomers (PBBA, PBPA and PBPMA) lead to the same kinetics of the polymerization reaction.

However, the situation is completely different if aromatic and aliphatic halogenated monomers are compared. Whereas the former led to a reactivity of the formulation, no reaction is observed in the latter systems. Additional investigations without the edge filter also revealed some reactivity for the aliphatic mixtures, but this behavior is most probably related to the direct excitation of acrylate bonds by the short wavelength emission of the mercury lamp since some reactivity is observed even for

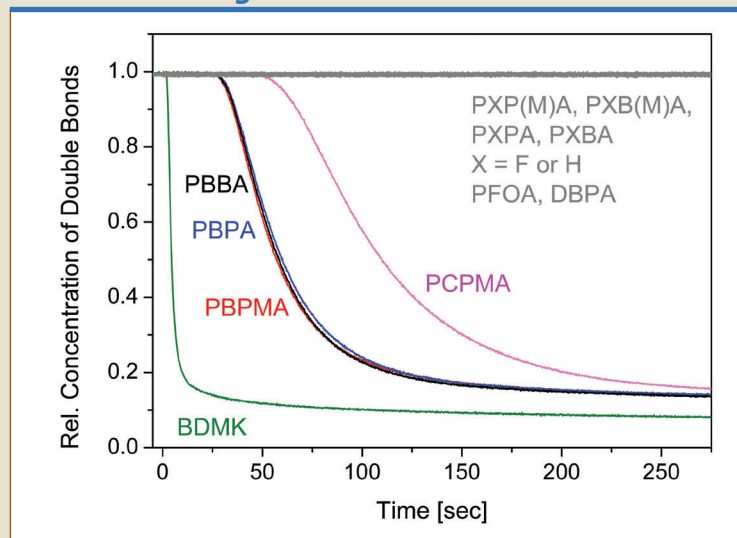the model lacquer without any other component under these conditions.

Finally, irradiation was carried out using the 313 nm interference filter. Results are shown in Figure 5. The time scale of the reaction is much longer than in the previous investigations,

FIGURE 5

Irradiation using the 313 nm interference filter

their fluorinated and non-halogenated counterparts (as well as halogenated aliphatic acrylates) are ineffective for this purpose.
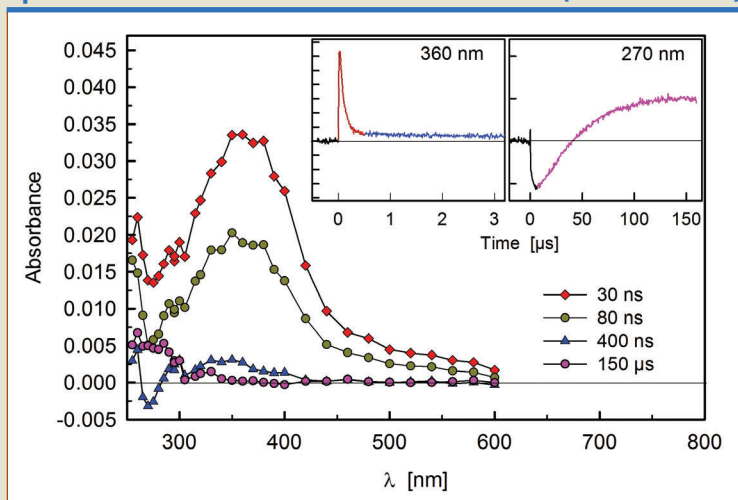
Laser Photolysis

Laser photolysis measurements were carried out to reveal possible pathways of the initiation and to determine the quantum yield of the formation of radicals. The nitrogen-purged samples were exposed to laser flashes with an excitation wavelength of 308 nm. Figure 6 shows the absorption spectra of the resulting short-lived transient species of a PBBA/acetonitrile solution (7.5×10^{-4} mol l^{-1}). At first glance, these spectra with a main absorption at 360 nm and a shoulder extending to 600 nm resemble the spectrum of the $Br_2^{\bullet-}$ radical, but the missing characteristic peak at 700 nm and the short decay time argue against that hypothesis. Hence, this short-lived transient was assigned to the triplet state of PBBA, which is also in accordance with its faster decay in the presence of oxygen.

At the end of the decay (i.e., at times longer than about 400 ns after the laser flash), a weak absorption remains detectable (see left inset in Figure 6). Since bromine radicals do not absorb in this spectral range, this absorption is expected to be related to the tetrabromophenyl radical. This assignment is also supported by quantum chemical calculations. Much later, a new band appears around 260-270 nm (right inset in Figure 6), which was tentatively assigned to molecular Br_2 resulting from the recombination of bromine radicals.

Due to the fact that bromine radicals do not absorb in the spectral region accessible to the measurements, it is impossible to observe their release and to define the rate constant of this process directly. In order to overcome these difficulties, $(C_2H_5)_4NBr$ was added to the PBBA solution since this

FIGURE 6

Absorption spectra of the short-lived transient species of a PBBA/acetonitrile solution (7.5×10^{-4} mol l^{-1})

bromide leads to the fast formation of $Br_2^{\bullet-}$ with bromine radicals. In fact, the transient spectra recorded after laser photolysis reveal a strong absorption at 360 nm and a weak band at ~700 nm, which are due to $Br_2^{\bullet-}$. Then the bromide concentration was increased. Under conditions where quantitative trapping of the bromine atoms occurs, the yield of $Br_2^{\bullet-}$ should be identical to the photochemical yield of bromine radicals. Using the triplet state of benzophenone as actinometer, a quantum yield of 0.31 was determined for the formation of bromine radicals from PBBA.

Similar results were obtained for all pentabrominated acrylates and methacrylates in this study. Generally, their quantum yield for the formation of bromine radicals was found to be about 0.3, which is in the same order as typical quantum yields of conventional photoinitiators.[7] Moreover, these results show that the specific type of the double bond—acrylate or methacrylate—does not have any influence on the photochemistry of the formation of radicals.

Laser photolysis experiments on the pentachlorinated methacrylate (PCPMA) with excitation at 308 nm and 266 nm did not provide any evidence for the release of chlorine radicals resulting from the homolysis of chlorine-phenyl bonds. After addition of $(C_2H_5)_4NCl$ as scavenger, no absorption at 340 nm was observed in the transient spectrum which was expected for $Cl_2^{\bullet-}$ species in analogy to the band of $Br_2^{\bullet-}$ at 360 nm. However, steady-state photolysis with irradiation at 254 nm led to a range of products in the GC/MS measurements (which can likely be assigned to an isomer of PCPMA); to pentachlorophenol formed by a α–fragmentation of the carboxylic group; and to two isomers indicative of chlorine release. The data show that the initiation apparently proceeds via several pathways in the case of the chlorinated methacrylate. Further work on this question is currently in progress.

In the case of the aliphatic brominated acrylate (DBPA), the addition of $(C_2H_5)_4NBr$ to the solution led to the appearance of the well-known absorptions at 360 nm and

700 nm which are characteristic for the formation of $Br_2^{\bullet-}$. Nevertheless, in the RT-FTIR measurements, a significant polymerization reaction with this monomer was only observed upon irradiation with short-wavelength radiation. Finally, no formation of reactive species was observed by laser photolysis at 266 nm for any of the fluorinated monomers.

Quantum Chemical Modeling

In order to shed light on the mechanisms involved in the self-initiation of the photopolymerization using halogenated (meth)acrylates, extensive quantum chemical calculations were executed to simulate and evaluate possible reaction pathways. After optimization of the molecular geometries of the monomers, electronic structures and energies of major transients (such as triplet states and neutral radicals) were determined. In the next step, the energies of excitation and relaxation schemes, as well as of possible reaction pathways, were calculated. Finally, frequencies and intensities of the UV-Vis absorption bands were estimated.

Figure 7 shows a schematic summary of the results of the quantum chemical modeling of the excitation of pentahalogenated methacrylates and the subsequent reaction steps. In the case of phenyl methacrylate and its fluorinated derivative, the highest occupied molecular orbital (HOMO) is located at the aromatic ring in the singlet ground state. However, after excitation by UV light and intersystem crossing, the spin density of the triplet state is shifted to the methacrylic double bond. The excess energy is partially dissipated through twisting of the orbitals of the double bond. For this reason, radical formation is possible in principle, but requires too much energy. Similar results have also been observed in the past for non-halogenated aliphatic acrylates.[8]

FIGURE 7

Schematic summary of the results of the quantum chemical modeling of the excitation of pentahalogenated methacrylates and subsequent reaction steps
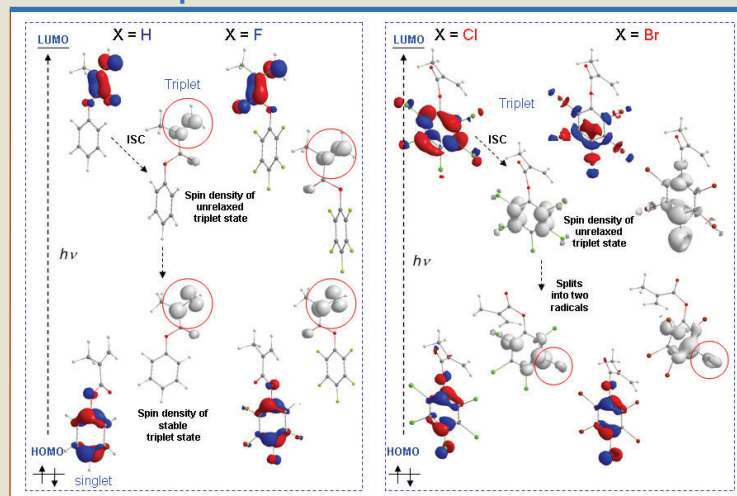
This means that radicals may only be generated by irradiation in the UV-C or VUV which corresponds to the direct excitation mechanism reported earlier.[1,2] However, no halogenation of the monomers is required for this process.

In the case of the chlorinated and brominated methacrylates, the HOMO orbitals are also located at the aromatic ring. After UV excitation and intersystem crossing occurs, some rearrangement of the electron distribution takes place, but the spin density is still localized at the ring. The orbitals get antibonding character and the bond distances between phenyl carbon and halogen atoms increase, which finally leads to the release of a bromine or chorine radical. The process of Br or Cl dissociation from the aromatic ring is thermodynamically favorable since it is exothermic ($\Delta H < 0$) and exergonic ($\Delta G < 0$). The energetical differences between the various splitting positions at the ring are in the range of only 2-3 kcal mol^{-1}, which means that the release can occur

from each of them (with a slight preference of the meta position).

Evidently, the splitting leads to the generation of two radicals—the bromine (or chlorine) and the tetrabromophenyl (or tetrachlorophenyl) radical. Both are able to initiate a polymerization reaction. According to modeling calculations, the reaction enthalpies for the addition of the radicals to a double bond are about -8 and -40 kcal mol^{-1} for the bromine and the tetrabromophenyl radical, respectively. This result implies that the latter radical might be more efficient with regard to initiation. On the other hand, bromine radicals do not react with oxygen, which is expected to lead to a reduced sensitivity to oxygen inhibition.

Quantum chemical calculations were also carried out to model the excitation of the aliphatic halogenated acrylates. As an example, the energy scheme of DBPA is shown in Figure 8. It is obvious that the energy of the excited singlet state is very high

FIGURE 8

Energy scheme of DBPA—an example of a quantum chemical calculation carried out to model the excitation of the aliphatic halogenated acrylates
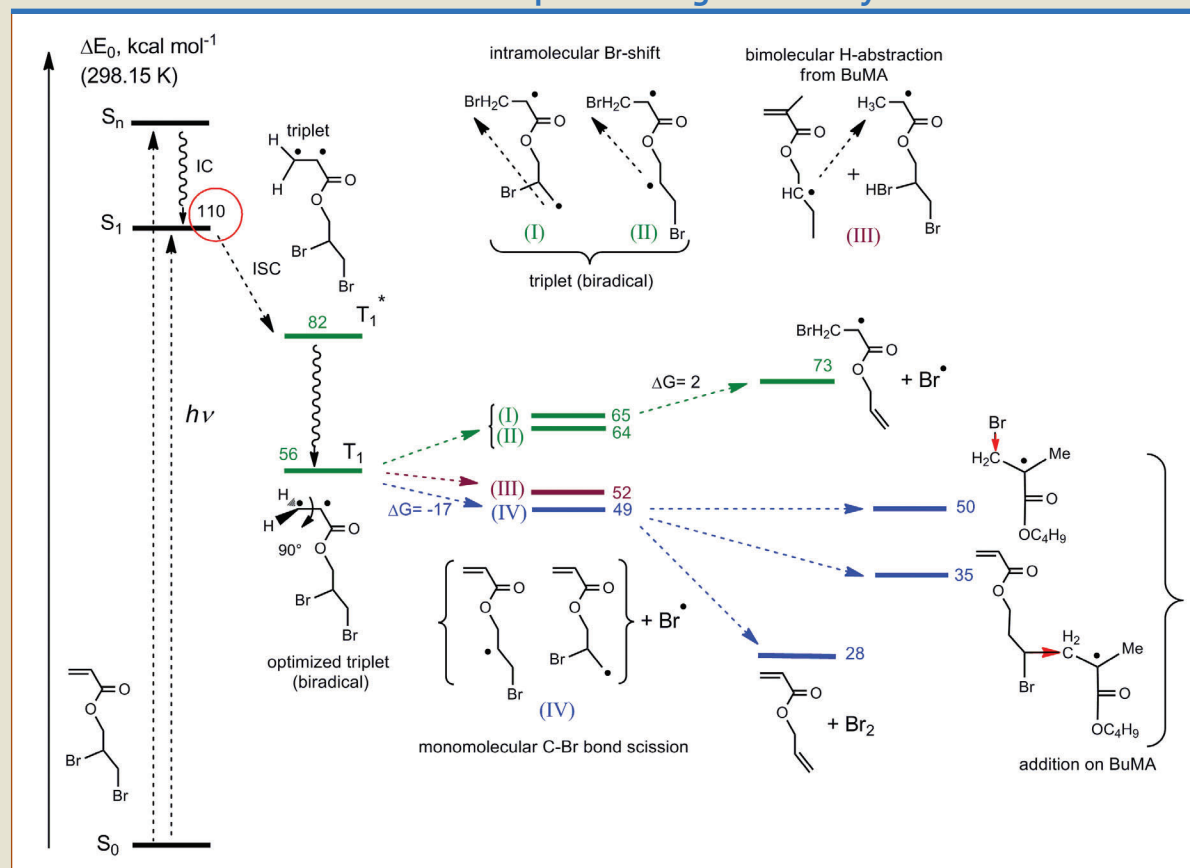
(~110 kcal mol^{-1}) which, consequently, requires high energies for excitation such as those of UV-C or VUV photons. However, such photons lead to a direct excitation of the acrylic double bonds of any acrylate regardless of their halogenation.

Conclusions

In this study, it was clearly shown that brominated and chlorinated aromatic (meth)acrylates may self-initiate a photopolymerization reaction upon UV exposure. If they are added in rather low concentration to a conventional acrylate formulation, they can serve as photoinitiator. In the case of brominated monomers, the release of radicals was detected by laser photolysis and the quantum yield of this process was found to be about 0.3. It is insignificant for the generation of reactive species if acrylates or meth-acrylates are used as monomers. The pentachlorinated methacrylate also led to a fairly fast photopolymerization reaction. However, chlorine radicals were not observed by laser photolysis, whereas several products were detected after steady-state photolysis. Hence, the mechanism of initiation is not yet understood.

In contrast, fluorinated (meth)-acrylates do not release radicals upon irradiation with a medium-pressure mercury lamp, since the energy required for excitation is too high. But, of course, they can be excited by short-wavelength UV radiation like any other acrylate. The same result was also obtained for the aliphatic fluorinated monomer (PFOA).

In the case of the aliphatic brominated acrylate (DBPA), the formation of radicals was clearly detected in the laser photolysis experiments in spite of the rather high excitation energy, but no polymerization reaction was observed in the RT-FTIR measurements. Further work is in progress in order to clarify the open questions and to complete the results obtained so far. ◗

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Batman-Inspired Grappling Hook
Created at BYU

Batman has gone through so many iterations that we've lost count, but one thing that's always been consistent is his fantastic set of gadgets and toys. The grappling hook is the most famous of the bunch, propelling the dark knight to the city rooftops. Students at Brigham Young University were tasked with a challenge—create a grappling apparatus similar to Batman's that can get four people up a vertical structure in 20 minutes or less.

The team immediately began researching the best method for being able to scale the 90-foot high platform, and arrived at a two-part attachment system. Their projectile is mounted with a head that is covered in an adhesive patch which should attach to the wall. Underneath is a light-cured adhesive strip, activated by UV light, that turns into a permanent epoxy. A system of pulleys then helps the user pull themself up.

The system that the team designed is able to support members of the marines, as well as the 300-pound gear they have to carry. While the challenge states that four members must ascend 90 feet in 20 minutes, the grappling hook the students designed can lift people around 30 feet per minute. Sure, it's not quite as cool or compact as what Batman carries, but it's a step in the right direction. Check out the video online (*www.slashgear.com*) to see it in action. ◗