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Polymer 44 (2003) 7671–7678
polymer
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Visible light induced polymerization of maleimide–vinyl and maleimide–allyl ether based systems

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Received 26 May 2003; received in revised form 28 August 2003; accepted 19 September 2003

Abstract

The photoinitiated polymerization reaction of maleimide–vinyl ether and maleimide–allyl ether under visible light in the presence of several visible photoinitiating systems have been investigated. Remarkable high rates of polymerization with nearly 100% of conversions are obtained. Photoinitiator free polymerization of maleimide–allyl ether systems exhibits also a quite good efficiency, comparable to that of maleimide–vinyl ether. Photoinitiators working through hydrogen abstraction like camphorquinone were shown to be able to initiate very well the visible curing of maleimide–allyl ether. The curing of a maleimide–allyl or vinyl ether formulation can be achieved within 1 s in the presence of a 4-component photoinitiating system under low light intensity. The role of a large variety of photoinitiating systems and the mechanisms involved in the initiation step are discussed.

Keywords: Photo-polymerization; visible light; Maleimide

1. Introduction

In the past years the polymerization reactions of maleimide (MI)-vinyl ether (VE) have received a new attention in the field of Radiation Curing when these systems were presented as being able to be used in photoinitiator free formulations [1,2]. This kind of reactions shows some interesting features, among them a very high cure speed (as that obtained with acrylates) and a no or low effect of the presence of oxygen [3]. Moreover, using vinyl ethers appear as an environment friendly alternative to the acrylate monomers which smell and cause skin and eye irritation.

In such systems, an acceptor molecule, the maleimide, containing an electron poor moiety (strong absorption in the 300–350 nm region) and an electron rich monomer like a vinyl ether (absorption below 250 nm) are used, thus leading to the expected presence of a charge transfer complex (CTC) although its absorption has not been detected so far by UV spectroscopy. Recent works have shown the polymerization of the maleimide–vinyl ether in photoinitiator free conditions [3]. The presence of a primary hydrogen abstraction process has been demonstrated to take place in the initiation step: it generates free radicals that can react with the monomer or participate in the propagation step of the polymerization through a chain transfer reaction ([4] and references therein). However, the polymerization efficiency sharply increased in the presence of a radical photoinitiator (like benzophenone [5] or an acyl phosphine oxide derivative TPO [6]). Several papers [7] are also currently devoted to the investigation of the mechanism involved and the hydrogen abstraction reaction [8–12], the use of maleimides as photoinitiators [7,13–15], the study of the propagation of the polymerization reaction and the formation of the polymer network [16,17], the kinetics of the reaction [18,19] and the possible applications [20–24]. One drawback of such CT polymerizable systems so far is the lack of availability of low cost, high quantities, of polyfunctional maleimides that show not the toxicity of certain low molecular weight *N*-alkyl maleimide. Some further developments are needed in that area (recently [25], maleimide derivatives, possessing a spiro structure, have been used for coatings or surface treatments).

This short literature survey shows that the photoinitiator free and UV photoinitiator containing maleimide–vinyl ether formulations in the Radiation Curing area can be considered as well known [1–24]. On the contrary, it seems that if maleimide–vinyl ether have been largely studied, the maleimide–allyl ethers (which are less expensive and less

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doi:10.1016/j.polymer.2003.09.057

A1: I819 1%

B1: I261 0.5% / CumeneH $1.5\% < x < 2\%$

B2: I261 0.5% / CumeneH $1.5\% < x < 2\%$ / RB 0.5%

B3: I261 0.5% / CumeneH $1.5\% < x < 2\%$ / RB 0.5% / MDEA $0.5 < x < 3.2\%$

C1: CQ 2%

C2: CQ 2% / Benzoyl peroxide 4%

D1: BP 2%

D2: BP 2% / MDEA 5%

F1: RB 0.5% / MDEA $1.9 < x < 3.4\%$

F2: RB 0.5% / MDEA 2.9% / Triazine B 0.5%

G1: I784 0.3%

Chart 1. Photoinitiators used: abbreviations and typical concentrations of the photoinitiating systems.

irritating than vinyl ethers) as well as the visible light ($>$420 nm) induced polymerization of maleimide–vinyl ethers have been until now never reported in the literature.

In the frame of our works on the investigation of photoinitiating systems for visible light induced polymerization reactions [26], the present paper is devoted to (i) the polymerization of maleimide–allyl ethers in the presence of visible light photosensitive initiating systems (type I photoinitiator working through a cleavage process and type II photoinitiator working through a hydrogen abstraction reaction [27]) and (ii) a comparison with the polymerization of maleimide–vinyl ethers in the same conditions.

2. Experimental

2.1. Samples

The photoinitiating systems and concentrations used are listed in Chart 1. I819 (Ciba Speciality Compagny) is a bis-acyl-phosphine oxide derivative, I261 (Ciba Speciality Company) a ferrocenium salt derivative and I784 (Ciba Speciality Company) a titanocene derivative. Rose Bengal (RB), *N*-methyl-*N*,*N*-diethanolamine (MDEA), camphorquinone (CQ), benzophenone (BP), Cumene hydroperoxide (CumeneH) and all the compounds for which nothing has been mentioned were obtained from Aldrich Company. Triazine B (2-(4′-methoxy-1′-naphtyl)-4,6-bis(trichloromethyl)-1,3,5-triazine) from PCAS company (Longjumeau, France).

The formula of maleimide PMI (*N*-propyl maleimide, Fluka, St Louis MO, USA), allyl ether TMPAE (trimethylolpropane allyl ether from Aldrich Compagny) and vinyl ether HBVE (hydroxybutyl vinyl ether from International Speciality Group, New Jersey USA) used in the study are shown in Chart 2. Using hydroxylated monomers speeds up the curing of the MI–VE systems [6,18].

The maleimide–vinyl ether or maleimide–allyl ether formulation consists of an equimolar blend of the two monomers.

2.2. Technique and procedure

The crosslinking reaction was followed, as proposed [28] and originally described [29] by Real-Time Infrared Spectroscopy by using a FTIR spectrophotometer (Nicolet Avatar 360) working in the rapid-scan mode allowing an average of 2 scan/s collection rate with a 4 cm^{-1} resolution. The sample is irradiated either with the glass filtered light of a LightningCure LC5 from Hamamatsu fitted with a L8253 Xe–Hg lamp (which delivers mainly visible light above 300 nm) (Fig. 1) or with a lamp using a metal halide visible source (Osram HMI 125W). The light intensity at the sample was measured by a calibrated radiometer (IL390 Light Bug) for the UV-A domain and a bolometer for the whole UV–visible domain (Kimmon PT-3000). The kinetic of the polymerization was measured by following the disappearance of the appropriate IR absorption bands (the maleimide band at 830 cm^{-1}, the vinyl ether band at 1617 cm^{-1}, the allyl ether band at 928 cm^{-1}) which allows the calculation of the percentage of conversion as a function of time. From the linear part of these kinetic curves, the rate of polymerization Rp can be determined.

The formulated resin is applied as a uniform layer of 15 μm thickness with a calibrated wire-wound bar on a BaF_2 pellet and covered with a polypropylene sheet (laminate conditions).

3. Results and discussion

3.1. Photocuring efficiency: comparison of the reactivity of vinyl vs allyl ethers

3.1.1. MI–VE system under UV irradiation. Comparison with a Visible irradiation

The photocrosslinking reaction of a monofunctional maleimide (PMI) with a monofunctional vinyl ether

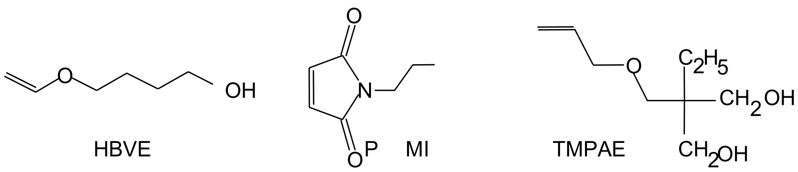


Chart 2. Vinyl ether, maleimide and allyl ether used in the study.

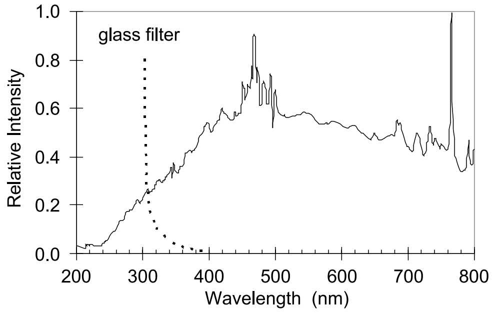


Fig. 1. Emission spectrum of the Xenon–mercury lamp used.

(HBVE) under visible light, in the presence or not of a photoinitiator, has been investigated (Fig. 2). The curing of the maleimide–vinyl ether system, under UV/Visible light is very fast and show percent conversion higher than 90%, especially in the presence of a radical photoinitiator like a bis-acylphosphine oxide (the same hold already true in the presence of a mono acyl-phosphine oxide derivative (Lucirin TPO [6])). In the absence of a radical UV photoinitiator, the curing rates are slower but a high conversion is still observed. The conversion still remains very high under only visible light above 400 nm in the presence of the I819 photoinitiator.

3.2. Rates of polymerization of a maleimide–allyl ether system in the presence of visible photoinitiators

The disappearance of the maleimide and the allyl ether double bonds under visible light and in the presence of a large variety of visible photoinitiators is followed by IR spectroscopy (at 830 cm^{-1} for PMI and at 928 cm^{-1} for TMPAE). The polymerization profiles of several systems are plotted in Fig. 3. The polymerization rates Rp and the conversion yield after 10 s of irradiation are shown in Fig. 4. For several photoinitiating systems a final conversion of almost 100% is reached for the maleimide whereas only 50% conversion is obtained for the double bonds of the allyl ether. The Rp and the conversions of the maleimide and the allyl ether monomers (AEH) are not similar (Fig. 4). It has been pointed out [6] that with a MI–VE ratio above 1, the fact that the MI is able to homopolymerise makes that the amount of unreacted VE increases. In the case of AEH, the concentration of CT complex must be lower due to the fact that AEH is a weak donor compared to VE. Therefore, in an equimolar blend, the amount of free maleimide able to homopolymerize is higher than in a MI–VE mixture. Besides the bis-acylphosphine oxides, multi-component photoinitiating systems containing a ferrocenium salt with a hydroperoxide (B1–B3, see Chart 1)) are fairly usable at wavelengths above 500 nm when a dye such as Rose Bengal is used as a sensitizer. The CQ alone leads to a good reactivity that increases in the presence of a peroxide. Classical photoinitiating combinations based on BP/amine or Dye/amine are less reactive.

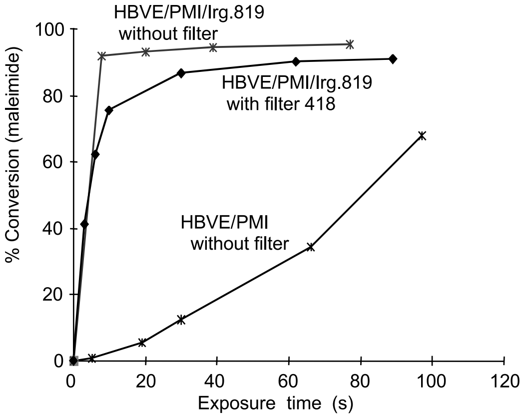


Fig. 2. Conversion of PMI/HBVE formulation with and without I819 under the light of a visible metal–halide lamp (intensity = 75 mW/cm^2).

3.3. Rates of polymerization of a maleimide–vinyl ether system in the presence of visible photoinitiators

The disappearance upon irradiation of the maleimide double bond and the vinyl group is followed by IR spectroscopy at 830 and 1617 cm^{-1} respectively. Some of the polymerization kinetics are plotted in Fig. 5. The polymerization rates Rp and conversion after 10 s of irradiation (deduced from the kinetic curves) are displayed in Fig. 6. Like for the maleimide–allyl ether, a very high final conversion of MI can be reached. On the other hand, the conversion of the more reactive vinyl ether is higher than that of the allyl ether and a nearly 1:1 reaction stoechiometry is observed. Several systems efficiently work: the same general trend is noted for maleimide–vinyl ether and maleimide–allyl ether (Figs. 3 and 5), except for the 4-component photoinitiating system based on Rose Bengal/ferrocenium salt/MDEA/cumene hydroperoxide which appears to be the best system for PMI/HBVE. CQ (C1 or C2) is less efficient as in the presence of an allyl ether and Rose Bengal/MDEA is a little better. As already observed [30] in photoinitiator free polymerization of such formulations, no complete conversion of the vinyl groups is noted since the plateau value is in general around 80%. The initial rates of polymerization and the conversions reached after 10 s of reaction (Fig. 6) are almost similar for both monomers, whatever the photoinitiating systems used (except for BP which is less efficient and has the weakest light absorption properties among the photoinitiators used but also leads to almost 100% conversion for very long irradiation times).

3.4. The role of the photoinitiating system

Under UV light exposure (in that case, the energy can be absorbed by the maleimide), the polymerization of the photoinitiator free maleimide–vinyl ether formulation has been already depicted in Refs. [3,6,7–24] and is recalled in Scheme 1: generation of two initiating radicals through hydrogen abstraction (likely electron transfer followed by

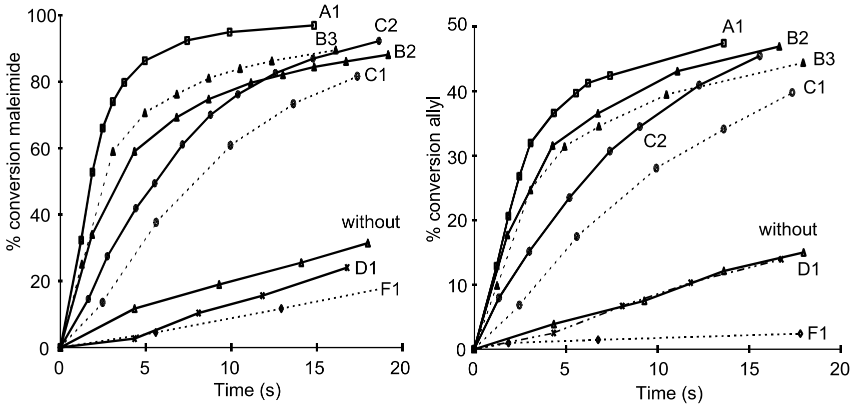


Fig. 3. Curing of the PMI-TMPAE formulations: conversion of the IR maleimide band (left) and the IR allyl ether band (right) in the presence of several photoinitiators. Glass filtered light. Intensity: 207 mW/cm^2.

proton transfer) from the vinyl ether (VEH) to the maleimide (MI) and then reaction of these radicals with the CT complex, thus leading to an alternating copolymer. This reaction can proceed either through homopolymerization of the CT complex or according to a cross-over process where the maleimide derived radical reacts with VEH; this cross-over process has been ruled out [6]. The same holds true in the presence of a mono phosphine oxide derivative.

Under visible light exposure, the energy is mainly absorbed by the photoinitiating system. In the maleimide–vinyl ether polymerization in the presence of type I

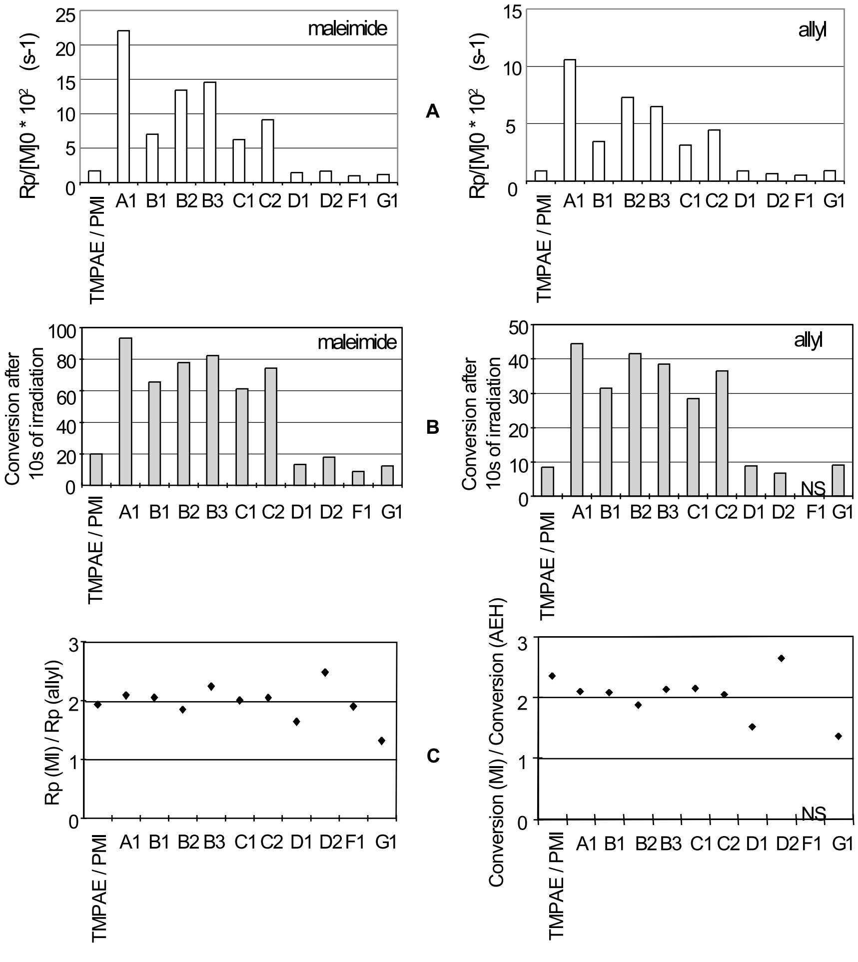


Fig. 4. The photocuring of the PMI-TMPAE formulations in the presence of the visible photoinitiated systems. (A) Rp of the maleimide and of the allyl ether (B) conversion of the PMI and of the TMPAE; (C) ratio between Rp (PMI) and Rp (TMPAE) (left); ratio between conversion of the PMI and the conversion of the TMPAE after 10 s of irradiation (right). NS: not significant.

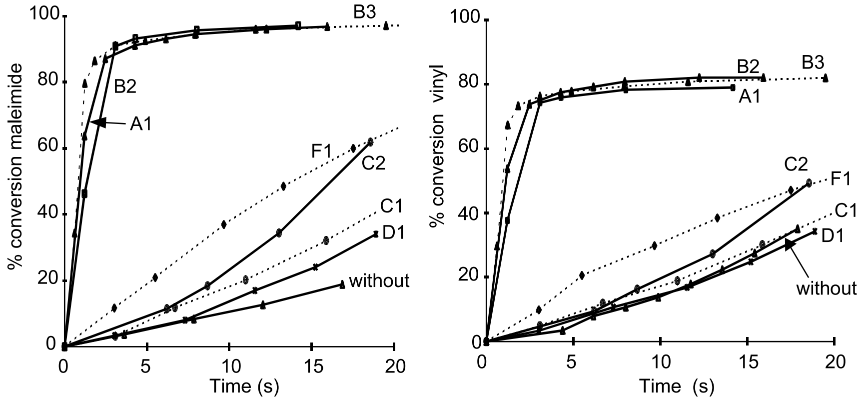


Fig. 5. Conversion of the PMI-HBVE formulation in the presence of several photoinitiators: (left) the PMI IR bands; (right) the HBVE IR bands, under a glass filtered of a Hg–Xenon lamp. $I_0 = 207$ mW/cm^2.

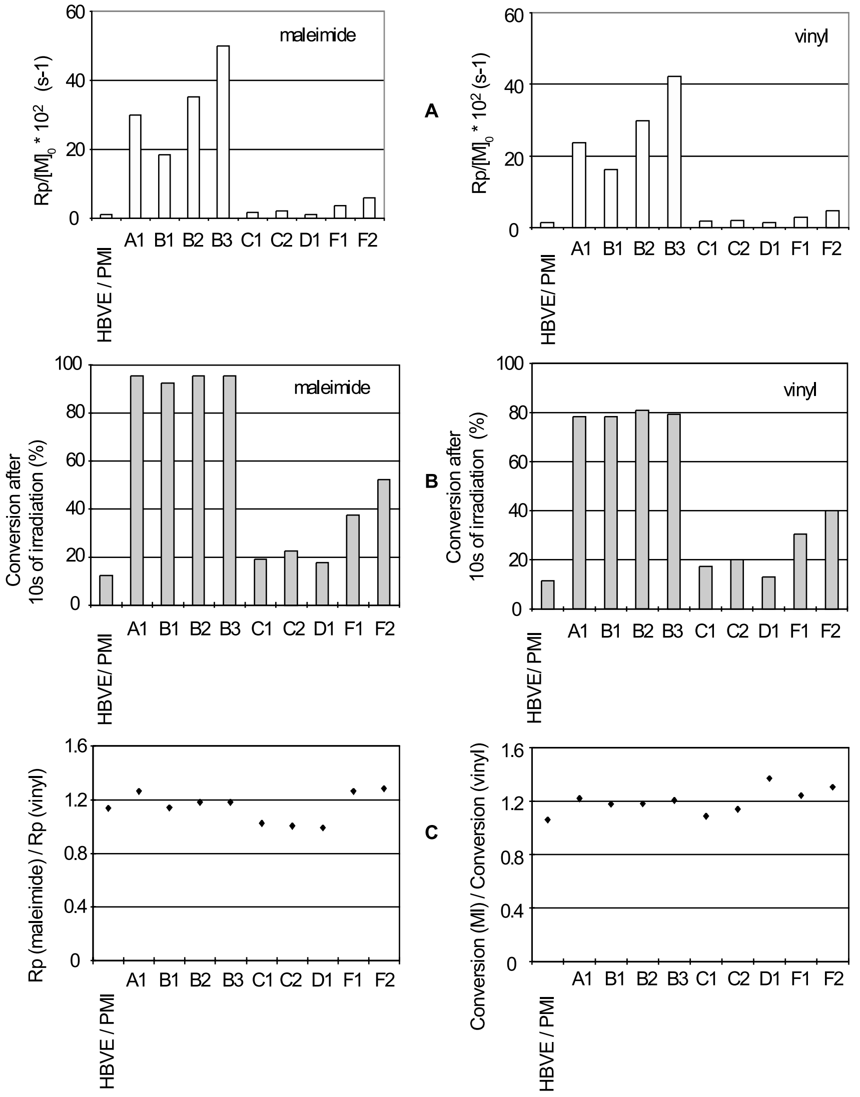
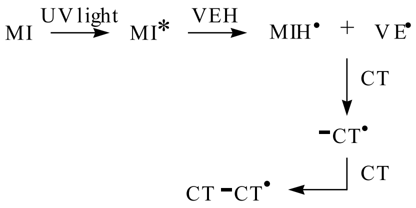


Fig. 6. (A) Rates of polymerization Rp and (B) percentage of conversion after 10 s of irradiation for the formulation PMI-HBVE in the presence of differents photoinitiators; (C) ratio between Rp (PMI) and Rp (HBVE) (left); ratio between conversion of the PMI and the conversion of the HBVE after 10 s of irradiation (right).

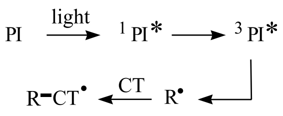


Scheme 1.

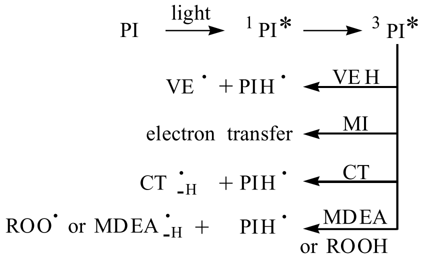
photoinitiators (such as the bis-acylphosphine oxides), initiating radicals are generated through a fast cleavage in the triplet state (so that no other processes of deactivation can compete) and will react with the CT complex (Scheme 2). With the type II photoinitiators such as benzophenone/MDEA or CQ/ROOH, several kinds of initiating radicals are formed through electron transfer between the long lived triplet state of the ketones and VEH, MI, CT, MDEA, ROOH. In that case, in contrast to what is observed with photoinitiators having short-lived triplet states, other processes of quenching, e.g. physical monomer quenching, oxygen deactivation can compete (Scheme 3). However, the absence of any substantial difference in the behaviour of the photoinitiator towards the two monomers at the beginning of the polymerization (Figs. 4(C) and 6(C)) suggests that the reaction also proceeds, under visible light, through a homopolymerization of the CT complex.

In the direct initiation process under UV light exposure or in the sensitized initiation process under visible light, the polymerization of the maleimide–allyl ether formulation should have been expected, in a similar way, to obey to Schemes 1–3 (where VEH has to be replaced by AEH). However, the differences noted for the rates of polymerization and the percentage of conversion after 10 s for the two monomers (Figs. 3 and 4), suggest that the mechanism of the polymerization should likely exhibit some change compared to that of the maleimide–vinyl ether polymerization (Scheme 1). The particular reactivity of the allyl ether may be related to the fact that it possesses a highly abstractable allylic hydrogen; therefore chain transfer reactions and branching on the allyl unit might occur and modify the polymer network structure compared to the case of the MI–VE system. Moreover, homopolymerization of MI can compete. This point concerning the polymerization mechanism deserves to be investigated by studying for example the structure of the polymers.

Moreover, if the reaction is carried out in aerated conditions, a photooxidative process involving oxygen can also contribute to some extent to the overall mechanism. In that case, after hydrogen abstraction from AEH, the reaction of the allylic radical with molecular oxygen leads to a peroxide which in turn can cleaves itself to generate initiating radicals being able to induce a crosslinking reaction.



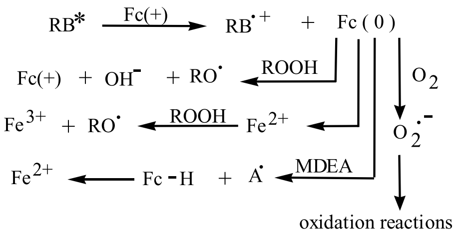
Scheme 2.



Scheme 3.

In the very interesting 4-component photoinitiating system based on Rose Bengal/ferrocenium salt/MDEA/cumene hydroperoxide, the formation of the initiating radicals can be depicted as mentioned in Scheme 4 (from what is known elsewhere in this particular photoinitiating system [31]): the presence of an intramolecular ion pair complex between the photosensitizer and the ferrocenium salt Fc(+) -I261-(as shown in another paper [32]) makes the primary step of the reactions involve an efficient electron transfer (this process is obviously not diffusion controlled) which leads to the formation of the reduced iron arene salt Fc(0) responsible for the further chemical processes originating from this species: interaction with molecular oxygen leading to the oxygen radical anion and then to oxidation products; electron transfer reaction with the hydroperoxide leading to an alkoxy radical; exchange reactions that finally lead to the formation of a ferrous species which reacts with the hydroperoxide and thus generates an alkoxy radical. The possible quenching of the excited states of RB by MI, AEH (or VEH) or the CT complex should not compete with the intramolecular RB/Fc(+) interaction in the ion pair complex. However, the maleimide can be involved in the next steps of the initiating mechanism. The reduction of the maleimide by Fc(0) (leading to initiating radical in the presence of an hydrogen donor [3]) can be in competition with the reduction of the hydroperoxide (Scheme 4).

All the radicals formed in Schemes 2–4 (may be, although nothing is known in that case, except the ketyl type radical PIH derived from the photoinitiator which is not an initiating radical for acrylate polymerization but a scavenging species of the growing macromolecular chains) can initiate the free radical polymerization of the maleimide–allyl or-vinyl ether formulations; Measurements of the rate



Scheme 4.

constants of interaction between the different partners (which should reflect, for a large part, the difference in reactivity observed) would help to understand the role of the driving reactions in the excited states.

4. Conclusions

The maleimide–vinyl ether and maleimide–allyl ether curable formulations are shown to be good candidates for a fast crosslinking (with an almost 100% conversion) of the MI, under visible lights. Two particular photoinitiating systems have been found to be very efficient in the case of maleimide–allyl ether: camphorquinone and a 4-component combination. The polymerization of the maleimide based formulations here occurs under a simulated sunlight, which proves that such photoinitiating systems are very promising for outdoor applications. In another field, under an Ar + laser exposure (line at 488 or 514 nm), only the 4-component combination will be operative. Excited state processes involved in these systems during the initiation step of the polymerization and measurements of the rate constants of interaction between the different partners as well as the visible light induced curing of other formulations will be presented in forthcoming papers.

Acknowledgements

Thanks are due to the European Community for financial support (GROWTH contract GRD1-2000-25541) and Perstorp AB (Sweden) for the supply of allyl ether derivatives.

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