



Visible light sensitive photoinitiating systems: Recent progress in cationic and radical photopolymerization reactions under soft conditions

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ABSTRACT

Although there have been many reports on photoinitiating systems adapted to visible lights for radical photopolymerization, the challenge for the design and development of photoinitiating systems for cationic photopolymerization or concomitant radical/cationic photopolymerization (for interpenetrating polymer network IPN synthesis) with visible lights still remains open. Particularly, the recent development of cheap and easily accessible LEDs operating upon soft visible light irradiations has opened new fields for polymer synthesis. Since 2011, many novel photoinitiating systems based on organic and organometallic compounds with excellent visible light absorption have emerged and exhibited outstanding photoinitiating abilities especially for cationic photopolymerization. In this review, recent progress (mainly from 2011 to early 2014) in applications of photoinitiators and sensitive photoinitiating systems under visible lights are reported. In addition, their relative efficiencies in the photopolymerization of different monomers are exemplified and discussed.

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Abbreviations: CCRP, concomitant cationic/radical polymerization (hybrid cure); CP, cationic polymerization; CRP, controlled/living radical polymerization; DVE-3, tri(ethylene glycol) divinyl ether; EPOX, (3,4-epoxycyclohexane)methyl 3,4-epoxycyclohexylcarboxylate; EPOX-Si, (epoxycyclohexylethyl)methylsiloxane-dimethylsiloxane copolymer; EPT, pentaerythritol tetraacrylate; FRP, free radical photopolymerization; FRPCP, free radical promoted cationic photopolymerization; Iod, diphenyliodonium hexafluorophosphate; IPN, interpenetrated polymer network; ITX, 2-isopropyl thioxanthone; MDEA, methyl diethanolamine; NVK, *N*-vinylcarbazole; OBN, oil blue N; PI, photoinitiator; ¹PI, singlet state photoinitiator; ³PI, triplet state photoinitiator; PIs, photoinitiators; PISs, photoinitiating systems; PS, photosensitizers; R-Br, phenacyl bromide; R'-Cl, 2,4,6-tris(trichloromethyl)-1,3,5-triazine; TEP, thiol-ene photopolymerization; TMPTA, trimethylolpropane triacrylate; (TMS)₃Si-H, *tris*(trimethylsilyl)silane; Trithiol, trimethylolpropane *tris*(3-mercaptopropionate).

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1. Introduction

For clarity in the present review, the word “photoinitiator” (PI) is used in a very general sense, defined as follows: a PI is a substance that absorbs light and participates in photoinitiation of a polymerization reaction, alone or through primary or subsequent reactions involving one or more additional compounds. A photoinitiating system (PIS) comprises one or several compounds, containing a PI, and is able to initiate a polymerization reaction. Therefore, one has to consider one-component PISs (*i.e.*, a PI), two-component PISs (*i.e.*, PI/additive) or three-component PISs (*i.e.*, PI/additive1/additive2), *etc.* This avoids the ambiguity with the word “photosensitizer” (PS) in photopolymerization (with the words “PI” and “PS” sometimes used in an ill-defined way); with the preceding definition, a PI can obviously also play the role of a PS in the sense of photochemistry.

The design and development of promising PIs (organic or organometallic compounds) exhibiting excellent light absorption properties (especially in the visible light wavelength range with high molar extinction coefficients) and being able to be incorporated in PISs – PI+additive(s) – is highly interesting for polymer synthesis and attracts increasing attentions in various fields ranging from radiation curing, imaging and optics technologies to medicine, microelectronics, nanotechnology and material elaboration areas [1–9].

To develop high-performance PISs for various photopolymerization reactions under visible light irradiations, several factors need to be satisfied and characterized. PIs should exhibit excellent visible light absorption (with high molar extinction coefficients) that has to match the emission spectra of the irradiation sources. After absorbing visible light at the appropriate wavelength, the PI excited singlet state ^1PI (or triplet state ^3PI to some extent) should efficiently interact with additives (*e.g.*, Iod or MDEA).

Long-lived singlet (or triplet) state lifetimes of PI are decisive.

Many PIs have been proposed (see, *e.g.*, in Ref. [1]), but recent strong developments involving novel strategies have led to the design of high-performance systems specifically for photopolymerization reactions under air, under low intensity of light sources and upon visible light exposures. Very few examples of new cleavable PIs (PI → radical pair → free radicals) have been recently noted (see Section 3.1). To the contrary, a real progress has been made in the synthesis of new chemical skeletons and new modifications of existing structures, as well as in the use of new additives: this has allowed the proposal of a large number of novel PIs and PISs. The mechanisms encountered in these systems are schematically depicted in reactions (1)–(7) [10–16]: after light absorption, PI interacts with one or two additives (*e.g.*, iodonium salt, amine, *N*-vinylcarbazole, silane, chloro triazine, *etc.*; Scheme 1) to generate initiating radicals for free radical polymerization (FRP; reactions (1)–(3a) and (6)) of (meth)acrylates or thiol-ene polymerization (TEP; reactions (1), (2) and (5)), cations or radical cations for cationic polymerization (CP; reactions (1) and (2)) or free radical promoted cationic polymerization (FRPCP) of epoxides (reactions 1–3) or vinyl ethers (reactions (1), (2) and (4)); the concomitant cationic/radical polymerization CCRP (hybrid cure) of a cationic monomer/radical monomer blend can also be achieved for the manufacture of interpenetrated polymer networks IPNs. The PI/iodonium salt/(TMS)₃Si–H (or NVK) system appears now as a magic combination as (i) the same radicals (*i.e.*, phenyl, (TMS)₃Si• or Ph-NVK• radicals) and cations (*i.e.*, silylium or Ph-NVK⁺ cations) are generated whatever the kind of PIs (see reactions (1)–(3)) and (ii) the presence of the silane (the same holds true with a germane or a borane) helps to decrease the oxygen inhibition effects through the conversion of peroxy radicals to new initiating radicals [1,17,18] (reaction (7)), allows radical

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