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SURFACE PHOTOGRAFTING POLYMERIZATION OF STYRENE ON LDPE FILM

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ABSTRACT

Styrene (St) was photografted onto LDPE substrates with different methods including a one-step method, pre-irradiation method, and pre-grafting method. Percent conversion (CP) and grafting efficiency (GE) of LDPE/St polymerization systems were measured by the gravimetric method. The results show that the one-step method is not very effective, no matter which photoinitiator (BP, BPO, Irgacure 651 and ITX) is used. The pre-irradiation technology, by which the dormant groups are introduced onto the substrates in advance, is favorable relatively. If the pre-grafting method is employed, AA is the most preferred one among the monomers examined (AA, MA and MAH). In addition, it is found that in the absence of photoinitiators and at lower temperatures (lower than 50°C), no grafted polymer is produced; but at a high temperature (e.g., 80°C), a remarkable amount of grafted polymer of St is formed (GE = 44.9%). The phenomena indicate that St is prone to be grafted onto LDPE film by auto-initiation.

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Key Words: Photografting polymerization; Styrene; LDPE film

INTRODUCTION

Photografting polymerization is one of the effective means to be used to improve the surface properties of polymer materials, such as hydrophilicity,^[1] dyeability,^[1,2] photo-stability,^[3,4] slippery surface,^[5] temperature responsive characteristic,^[6] etc. The monomers usually applied are acrylic acid and its derivatives.^[7] Recently, surface functionalization has become one of the most active research fields.^[8,9] In order to obtain functionalized surface, benzene-ring is of significant importance by which a large number of functional groups can be introduced onto the surface of substrates. Styrene, containing benzene-ring, is thus the desired monomer. However, in our earlier work,^[10] it has been found that St is one of the problem-monomers, i.e., it is difficult to graft St onto substrates by means of normal methods.^[11] Few papers concerning grafting St onto substrates were reported, but the grafting polymerizations were mostly conducted in gas phase.^[12]

In this paper, extensive efforts were made to graft St onto LDPE substrates induced by UV radiation. Several methods, i.e., one-step and two-step methods, were tried to form LDPE-g-PS with appreciable grafting yield. Here, the two-step method includes pre-irradiation and pre-grafting methods. During the experiments, it was unexpectedly observed that some St was grafted onto the LDPE film under the condition that no initiator was added. This indicates that St may be included in those monomers that are prone to be photografted by auto-initiation. Therefore, this behavior of St was also investigated in detail.

EXPERIMENTAL

Materials

Photoinitiator

Benzophenone (BP, chemically pure, from Shanghai Reagent Plant No. 1, China), thioxanthone (ITX, AR grade, from PHT, USA), benzoyl-dimethylketal (Irgacure 651, AR grade, from Ciba, Switzerland) and benzoyl peroxide (BPO, AR grade, from Jiangsu Hongsheng Chemical Plant, China) were used as received.

LDPE Film

Commercial LDPE film (63 μm in thickness, transparency to UV light, 75%, $\lambda = 254 \text{ Nm}$) was cut into circular samples about 70 mm in diameter,

and then subjected to Soxhlet extraction with acetone for 5 h to exclude impurities and additives before use.

Solvent

Acetone, ethyl acetate and butanone, AR grade, were directly used without purification. Monomer: styrene (St), methyl acrylate (MA) and acrylic acid (AA), AR grade, were purified by distillation in advance to remove the inhibitor. Maleic anhydride (MAH) was purified by recrystallization from acetone. Acrylamide (AAM), AR grade, was used as received.

Grafting Procedure

The apparatus used to perform grafting polymerization and the setup of the film samples were reported in detail in the earlier paper.^[10] The main polymerization procedure was as follows.

One-Step Method

A pre-determined amount of monomer solution containing photo-initiator (pre-bubbled with nitrogen gas to remove oxygen dissolved in the solution) was deposited between two pieces of the film with a micro-syringe and appropriate pressure was given to make the reaction liquid a thin and even layer. Then the samples were placed on the holder and covered with a piece of quartz plate. The system was irradiated by UV radiation (UV lamp: high-pressure mercury lamp, 1000 W) at a given temperature controlled by a thermocouple thermometer. The polymerization degree was decided by varying the irradiation time.

Pre-irradiation Method

A certain amount of acetone solution containing BP (pre-purged with nitrogen gas) was deposited between the films, and made a thin and even layer with a certain pressure. The films were separated and stayed to dry at ambient temperature. After that, the films were irradiated by UV light in nitrogen atmosphere to prepare pre-irradiated film samples. By extraction with acetone, the residue of BP and pinacol formed on the films were eliminated. To complete the polymerization, two methods were applied, i.e., by heating or by UV radiation.

By UV Radiation

St was deposited between two pieces of the film samples pre-irradiated, and the system was irradiated by UV radiation at a given temperature.

By Heating

The main procedure was similar to that of by UV radiation, just differing in that a piece of aluminized foil was laid on the system to exclude the UV radiation totally. The polymerization was just conducted at the given temperature. The more detailed procedure is similar to that described above in the One Step Method section.

Pre-grafting Method

The main polymerization procedure is similar to that of applying the one-step method, just differing in that one kind of other monomers containing photoinitiator was grafted onto LDPE film samples at first, and then St (without photoinitiator) was added to let the polymerization continue. The polymerization of St was induced by UV radiation or by heating, which was introduced above.

Auto-initiating Method

The main procedure is similar to that of applying the one-step method, just differing in that St monomer solution without photoinitiator was used to carry out the grafting reaction.

To all of the polymerizations introduced above, after reaction, the polymerization systems were taken out, separated, dried to constant weight, and then subjected to Soxhlet extraction with suitable solvents for 8 h. to remove the homopolymer. After extraction, the samples were dried to constant weight again. Two parameters, percent conversion (CP) and grafting efficiency (GE) were determined according to the following formula.

$$CP = (W_P/W_M)100\%$$

$$GE = (W_G/W_P)100\%$$

where W_M is the weight of the monomer; W_P is the weight of polymer formed (the total weight of grafted polymer and homopolymer); and W_G is the weight of the grafted polymer.

RESULTS AND DISCUSSION

One-Step Method

In order to investigate the polymerization of grafting St onto LDPE substrates with one-step method, four kinds of photoinitiators, namely, BP, 651, ITX and BPO, were applied as the photoinitiator. All of them were dissolved in neat St. Both of the St solutions containing BP and 651 are colorless and transparent; the solution containing ITX seems to be slightly yellow; and the solution containing BPO seems to be colorless and slightly hazy, but there is no precipitate formed. The evolution of the photografting polymerization of St is presented in Figs. 1 and 2).

According to the curves in Figs. 1 and 2, it is shown that all of the initiators examined are not very effective in inducing grafting St onto LDPE film. The quantity of polystyrene (PS) formed is at the highest level when Irgacure 651 was used (CP is about 50%, when reaction proceeds for 180 sec); BP is the most unfit one, because only about 16% of the St took part in

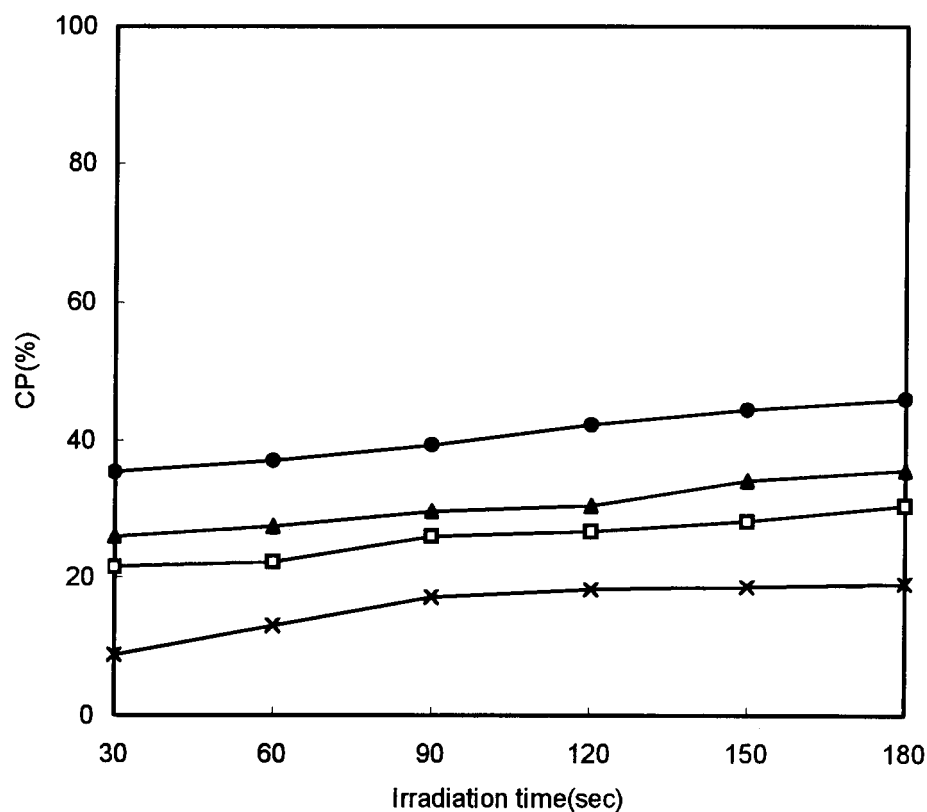


Figure 1. Effects of different initiators on CP ●, 651; □, ITX; ▲, BPO; ×, BP. Reaction temperature, 60°C; initiator/St, 3 wt%; intensity of UV radiation, 3900 $\mu\text{w}/\text{cm}^2$.

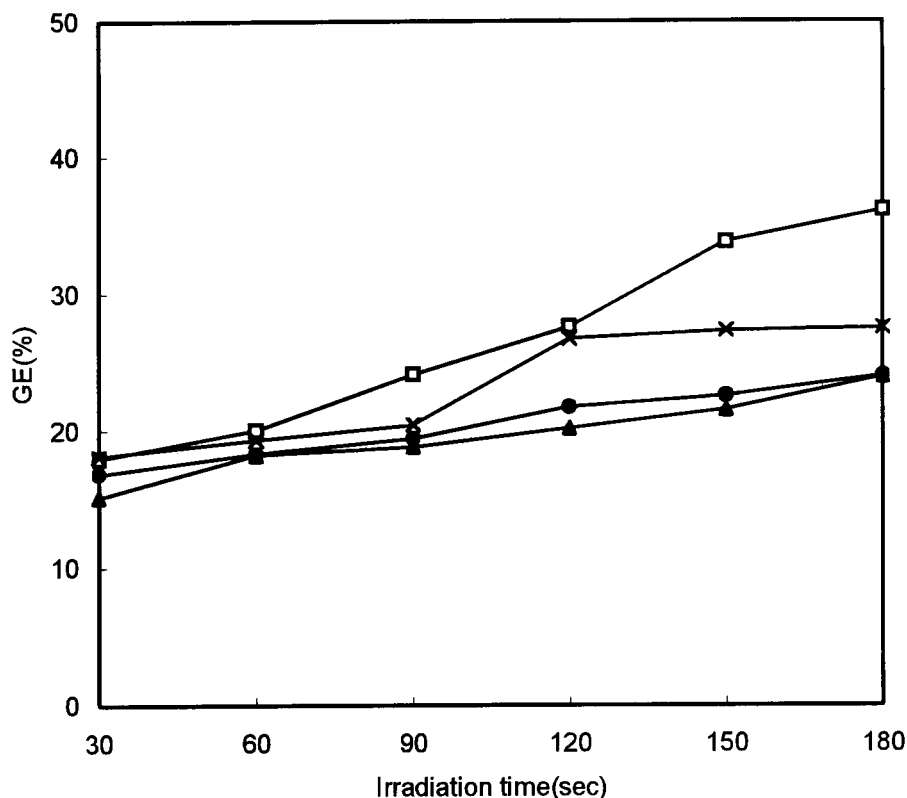


Figure 2. Effects of different initiators on GE ●, 651; □, ITX; ▲, BPO; ×, BP. Reaction temperature, 60°C; initiator/St, 3 wt%; intensity of UV radiation, 3900 $\mu\text{w}/\text{cm}^2$.

polymerization. As far as GE is concerned, ITX is the most suitable one. However, all of the GE's of the polymerization systems are too low, lower than 40%.

As for 651, a typical Norrish I photoinitiator, whose reactivity is higher compared with those of others, more monomer was initiated to proceed polymerization (Fig. 1). Due to the fact that it belongs to Norrish type I, that is, when irradiated by UV radiation, 651 molecules absorb energy, jump to excited state, and further undergo cleavage reaction, causing primary free radicals generated, which initiate St molecules to polymerize. GE of LDPE/St-651 system is at a low level (Fig. 2), since H-abstraction reaction and transfer reaction of St macro-radicals to PE macro-molecules occur with much difficulty.

To BP, included in Norrish type II, is prone to abstract hydrogens from H-donors; ITX, absorbing UV light in a wide range of wavelengths, is also prone to proceed H-abstraction. GE of these two polymerization systems, i.e., LDPE/St-BP and LDPE/St-ITX, should be higher than that of

LDPE/St-651. In fact, however, the amounts of grafted polymer formed in LDPE/St-BP and LDPE/St-ITX systems are not remarkably more than that of LDPE/ST-651, despite that the GE of LDPE/St-ITX is about 36.1%.

As to BPO, which is not usually used to initiate photo-polymerization, but from this study, its efficiency in inducing grafting of St is comparable to those of 651, BP and ITX under the same reaction conditions. Ogiwara and co-workers^[12,13] also used BPO to initiate photografting polymerization of monomers on polyolefin films.

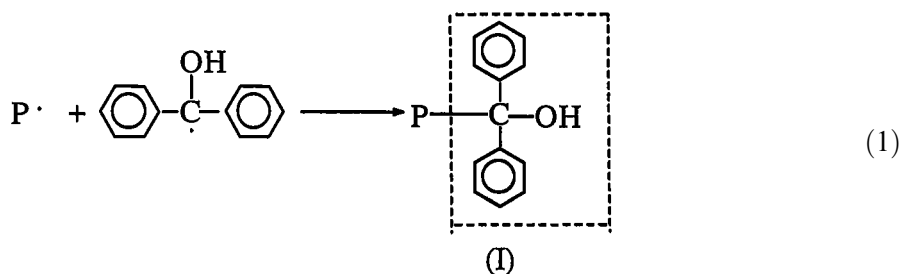
Our research results are in agreement with the studies of Ang et al.^[14] In their experiments, biacetyl (BAC), benzoin ethyl ether (BEE), BP and uranyl nitrate were used as initiators grafting St onto polypropylene (PP) film with simultaneous method. According to their research, BAC and BEE are the more effective initiators. In addition, similar results to ours were obtained in the report of W. T. Yang and colleague.^[11] Based on the research of Kuhlmann,^[15] it is suggested that BP photo-initiated polymerization of St is not feasible, since St molecules probably quench BP in triplet state (BP^T).

According to the above investigations, it is clearly shown that the one-step method, no matter which type of photoinitiators is used to initiate the grafting polymerization, is not suitable to be used in LDPE/St polymerization system.

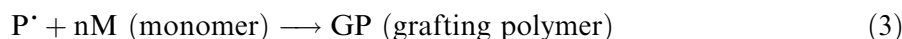
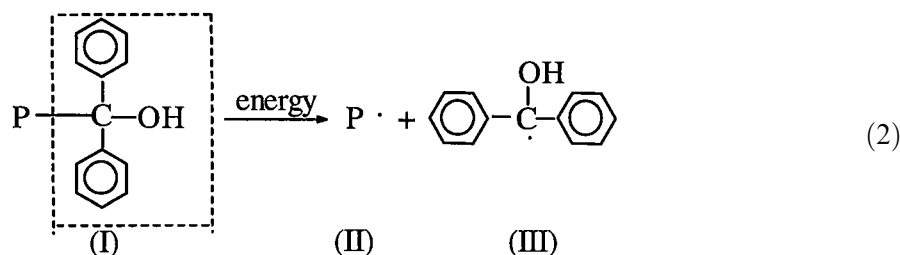
Pre-irradiation Method

To exclude the absorption of UV light by monomer St and the quenching effect of St on $[BP]^T$ or $[ITX]^T$, another approach i.e., the pre-irradiation method was explored. That is, BP pre-coated film samples are first prepared, then subjected to UV radiation to produce LDPE film samples containing dormant groups in surface; St is deposited and grafting polymerization occurs. The mechanism of this method can be briefly outlined as follows (Reactions 1~4).

During UV irradiation, coupling reactions between LDPE macromolecular free radicals and semi-benzopinacol free radicals take place (Reaction 1).



Product (I) of Reaction (1) may cleave under UV radiation or by heating, and then free radicals are produced again in the system, as described by Reaction (2). Product (II) and (III) formed in Reaction (2) initiate the homopolymerization and grafting polymerization, respectively (Reactions 3 and 4).



The results using pre-irradiation method are shown in Figs. 3–5.

In Fig. 3, the effects of concentration of BP on polymerization of the LDPE/St system are presented. From the curves of CP and GE, it is obviously shown that the pre-irradiation method is effective, by which 61.5% of St underwent polymerization and nearly 50% of the formed polymer is grafted co-polymer, remarkably higher than those of applying one-step method. In addition, the shape of the curves of CP and GE demonstrate that increasing BP concentration has a slight effect on the grafting polymerization of St. This phenomenon may be understood from two aspects as described below. Firstly, when a large amount of BP (e.g., 1.0 wt% of the film) is added on the films and irradiated by UV radiation, a great quantity of macro-radicals and semibenzopinacol free radicals are generated simultaneously, causing the possibility of the termination reaction between the free radicals to increase considerably. As a result, a slight drop for CP appears. On the other hand, the opportunity for St to homopolymerize or copolymerize remains the same, so GE remains nearly unchangeable. Secondly, the slight effects of increasing BP concentration on grafting polymerization may be ascribed to the auto-initiated grafting polymerization by St itself to some degree.

In Fig. 4, the concentration of BP is controlled equally for each sample, but the pre-irradiation time is lengthened progressively, which is indicated by the horizontal axis. From the curves of CP and GE, it is observed that increasing the pre-irradiation time is desired to grafting polymerization of St. Though CP increases to a small degree (from 35.6% when pre-irradiated

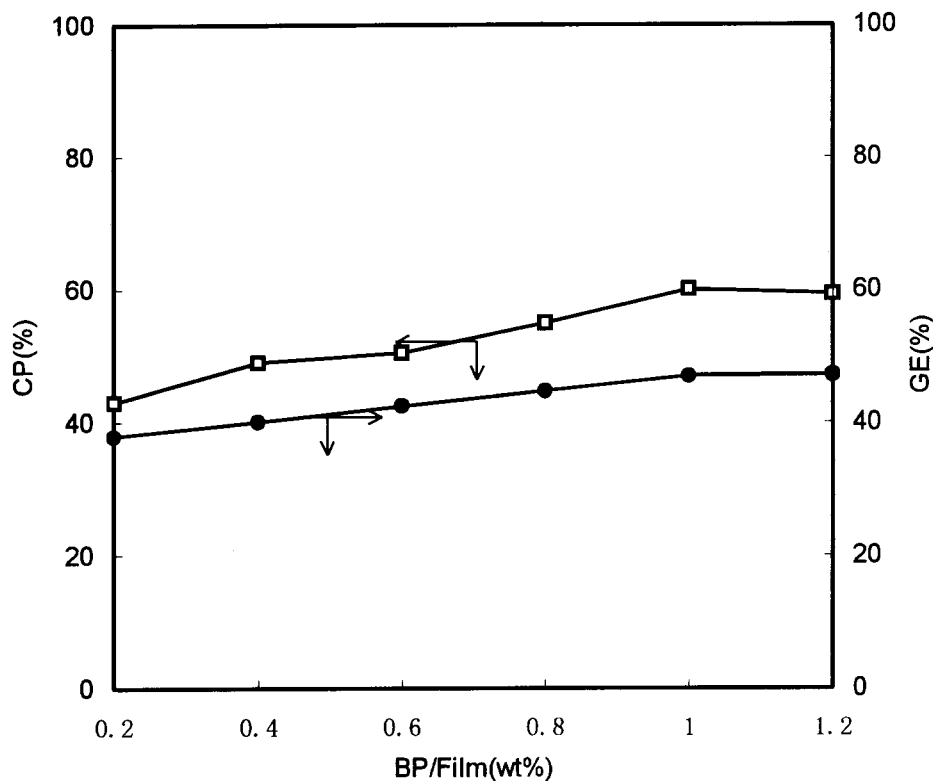


Figure 3. Effects of BP concentration on grafting polymerization ●, GE; □, CP. Reaction temperature, 60°C; intensity of UV radiation, 3880 $\mu\text{w}/\text{cm}^2$; pre-irradiation time, 90 sec; polymerization time, 120 sec.

for 15 sec to 46.7% for 90 sec), GE is elevated appreciably, namely, from 18.1% when the pre-irradiation time is 15 sec to 47.8% when it is lengthened to 90 sec. In order to understand the results, the quantities and activities of semibenzopinacol free radicals and macro-radicals are well worth considering.

After film samples containing BP have been pre-irradiated, St is deposited and the polymerization reaction may proceed under the conditions of just being heated, without being irradiated by UV radiation. To investigate this possibility, further research was conducted. In order to control the reaction conditions equally with those of being irradiated by UV light, a piece of aluminized foil was placed on the top of the quartz plate to screen the UV radiation, leaving other reaction parameters unchangeable. The results are presented in Fig. 5. From Fig. 5, it is indicated that just heating, but without UV irradiation, is not desirable for grafting St (compared with Fig. 4). Under the equal reaction conditions, less polymer of St was produced in the absence of UV radiation than that in the presence of UV radiation ($t=90$ sec,

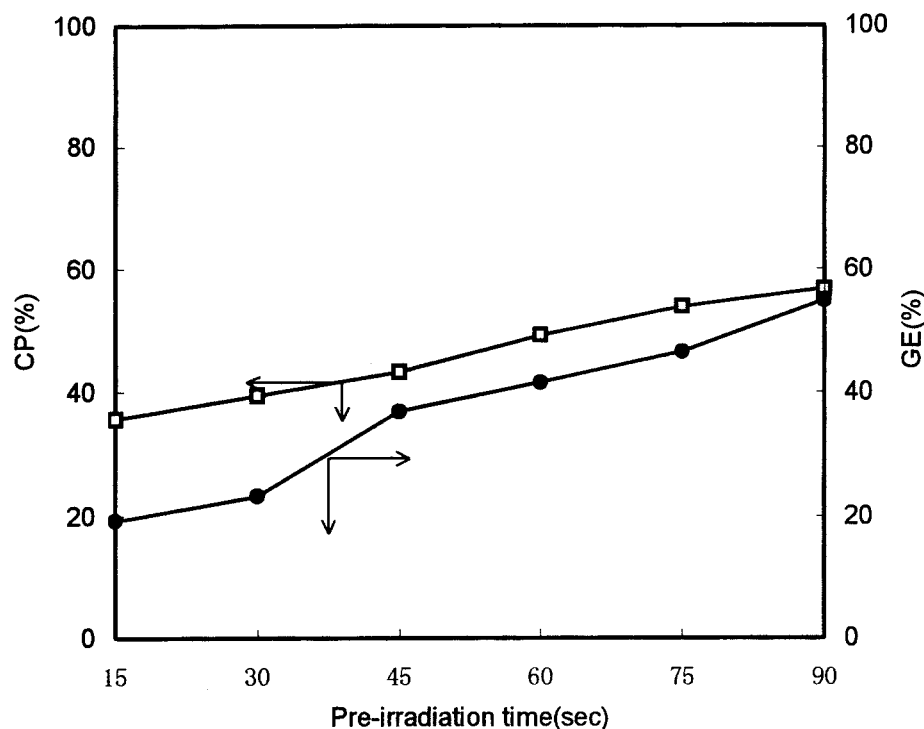


Figure 4. Grafting polymerization induced by UV radiation ●, GE; □, CP. Reaction temperature, 60°C; intensity of UV radiation, 3880 $\mu\text{W}/\text{cm}^2$; polymerization time, 120 sec; BP/Film, 0.4 wt%.

CP = 39.9%, in the absence of UV radiation; $t = 90$ sec, CP = 54.8%, in the presence of UV radiation). It seems that UV radiation facilitates the reactivation of the dormant groups and the polymerization of St.

Pre-grafting Method

Likewise, in order to get rid of the quenching effect of St on $[\text{BP}]^{\text{T}}$, the pre-grafting method was also employed. The monomers attempted are AA, MA and MAH. The results are listed in Table 1.

It is seen from Table 1 that after the polymerization of the first monomer, AA, MA or MAH, adding St made the reaction continue. The reason may be that, when St was added, there are dormant groups in the polymerization systems as discussed above in the pre-irradiation method. Polymerization reaction of St thus occurred, which was induced by free radicals produced during the process of reactivation of the dormant groups. To monomer AA, when its polymerization ended, most AA (95%) took part in the reaction, leading to a large amount of dormant groups and a large

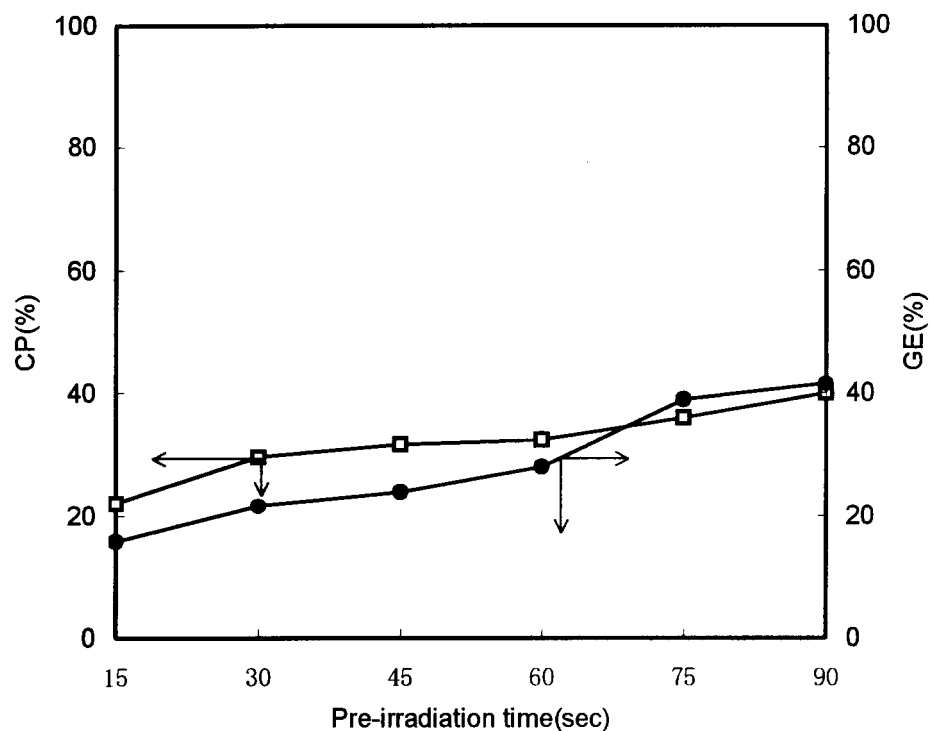


Figure 5. Grafting polymerization induced by heating ●, GE; □, CP. Reaction temperature, 60°C; intensity of UV radiation, 3880 $\mu\text{w}/\text{cm}^2$; polymerization time, 120 sec; BP/Film, 0.4 wt%.

Table 1. Grafted Polystyrene Applying Pre-grafting Method (mg)*

Volume of St (μL)	AA		MA		MAH	
	UV Radiation	Heating	UV Radiation	Heating	UV Radiation	Heating
0	1.9(2.0) ^a	/	0.4(2.0) ^a	/	0.7(2.0) ^a	/
5	2.3	1.7	0.8	0.4	1.4	0.8
10	3.6	2.6	1.1	0.6	1.6	1.2
15	4.8	3.3	1.3	0.7	1.8	1.3
20	5.8	3.6	1.3	0.8	1.9	1.5
25	6.5	3.7	1.4	0.9	2.0	1.7

*Obtained after extracting the homopolymer of St.

^aThe amount of grafted polymer of AA, MA, and MAH, respectively, when no St was added. Data in brackets, the amount of monomer (AA, MA, and MAH) used.

Reaction conditions: monomer (AA, MA, or MAH)/film, 0.5 wt% polymerization temperature, 60°C; BP concentration, 3 wt% of AA, MA, or MAH; intensity of UV radiation, 6250 w/cm^2 ; polymerization time for the first monomer, 30 sec; polymerization time for St, 90 sec.

amount of free radicals. Therefore, more grafted polymer of St was produced. Again from Table 1, it is obvious that MA is not a suitable monomer. The results may be attributed to the low quantity of grafted polymer of MA. It was found that irradiated for 30 sec, only 20% of MA polymerized to form grafting copolymer (Table 1). When MAH was used to perform the first step polymerization, the results are not very satisfactory either. According to the data in Table 1, for the first step polymerization, only 35% of MAH participated in grafting polymerization. Because St and MAH are inclined to copolymerize, and the amount of residual MAH and the homopolymer of MAH exceeds greatly that of grafted MAH, a small amount of grafting polymer of St was formed.

In addition, it is clear from Table 1 that grafting polymerization of St in the absence of UV radiation proceeds with much difficulty. After the first step, polymerization was completed, a thin layer of polymer of the first monomer was generated. When St was added, and irradiated by UV radiation, St molecules may abstract hydrogens from PE macro-molecular chains or from new-formed polymer chains of AA, MA and MAH. When UV radiation is absent, these reactions rarely occurs.

Auto-initiated Grafting Polymerization

As mentioned above, some phenomena performed in grafting St could not be explained with ease, for example, the effects of the concentration of BP on the grafting polymerization of St. Therefore, it is thought that auto-initiated grafting polymerization of St may contribute to them. Attempts were thus made to study it, as presented in Fig. 6.

It may be seen from Fig. 6 that at lower temperatures, such as 50°C, only homo-polymerization of St occurred. When the reaction temperature was elevated to 70°C, not only a homopolymer was formed, a grafting copolymer was also formed. Furthermore, the content of the grafting copolymer was remarkable (GE = 30.1%, when reaction proceeded for 4 min). If the temperature continues to be elevated to 80°C, more grafting copolymer was produced, and GE went up to 44.9% (t = 4 min). Nevertheless, CP fell off slightly as the temperature was elevated, which may be assigned to the volatilization of St. The higher the reaction temperature, the greater the volatilization of St.

There have been no similar studies reported up to now, the mechanism of the polymerization of St initiated by itself under UV radiation may be briefly proposed as below. When molecules of St are irradiated by UV radiation, they absorb energy and then are excited to single state $[St]^S$ (Reaction 5). Through intersystem crossing (ISC), $[St]^S$ transforms to more a stable triplet state $[St]^T$, which may exist in $Ph-CH\cdot-CH_2\cdot$, containing two free radicals (Reactions 6, 7).^[16] $Ph-CH\cdot-CH_2\cdot$ probably attacks monomer

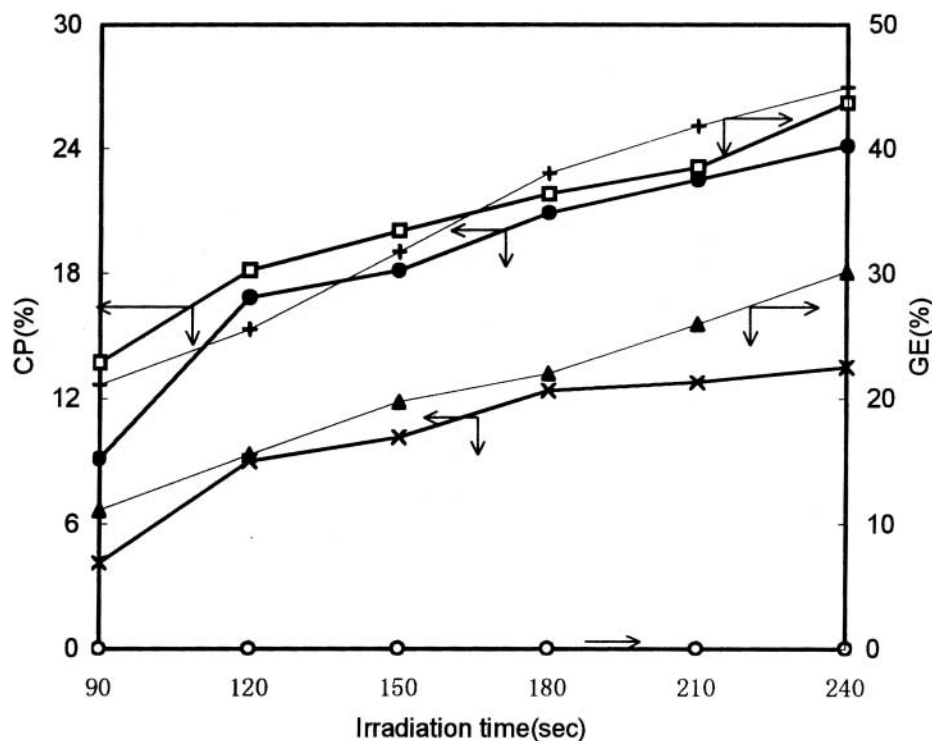
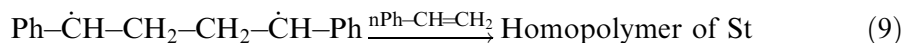
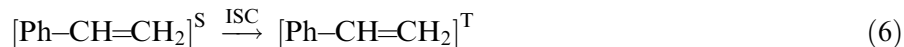
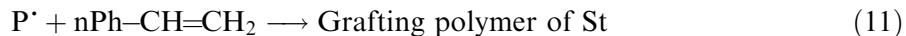


Figure 6. Results of polymerization induced by auto-initiating of St ○, GE(50°C); □, CP(50°C); ▲, GE (70°C); ●, CP(70°C); ×, CP(80°C); +, GE(80°C). St/film, 4 wt%; intensity of UV radiation, 5010 $\mu\text{w}/\text{cm}^2$

molecules of St (Reaction 8), and the product of Reaction (8) may initiate polymerization of St (Reaction 9). In the meantime, $\text{Ph-CH}\cdot\text{-CH}_2\cdot$ may also attack the macromolecules of LDPE, and abstract hydrogen atoms, resulting in macro-radicals formed, which finally induce grafting polymerization of St (Reactions 10, 11). During the reaction, the macroradicals of PS may proceed coupling reactions with LDPE macro-radicals, e.g., $\text{P}\cdot$ or $\text{P}\sim\cdot$, also causing grafting polymer produced (Reaction 12).





The efficiency of St initiating grafting polymerization of other monomers onto substrates was investigated, which is presented in Fig. 7.

In view of the fact that the efficiency of St to abstract hydrogens may be lower than that of general photoinitiators, a large amount of St was thus used to induce photografting polymerization of other monomers, including AA and AAM. From Fig. 7, it can be seen that when no St was added, the amount of grafted PAA is just 1.2 mg when the polymerization temperature is 80°C (the total amount of monomer AA is 5.4 mg. Along with the increase of St added, the amount of polymer grafted (PAA + PS) goes up also, definitely indicating that St participated in the grafting polymerization. Figure 7 also shows the similar results of AAM (the total amount of AAM used is 2.1 mg to those of AA. Based on the results introduced above, it is confirmed

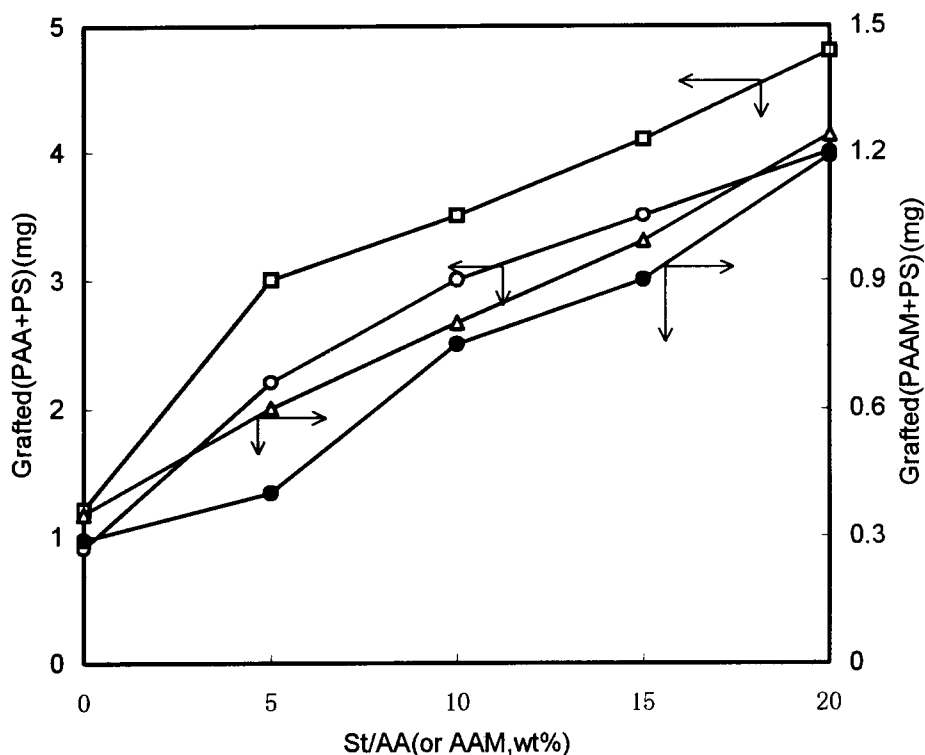


Figure 7. Results of grafting polymerization of AA and AAM initiated by St □, AA, acetone as solvent; ○, AA, ethyl acetate as solvent; △, AAM, acetone as solvent; ●, AAM, ethyl acetate as solvent; AA concentration, 5 mol/L; AAM concentration, 2 mol/L; polymerization time, 2 min; polymerization temperature, 80°C; intensity of UV radiation, 5150 $\mu\text{w}/\text{cm}^2$.

indirectly that when it is irradiated by UV radiation, St can abstract hydrogens from LDPE substrates.

However, the increase of the quantity of grafted polymer caused by adding St may be regarded as the contribution of the solvent acetone, a photosensitizer to some degree. In order to clarify this situation, complementary experiments were conducted, i.e., using ethyl acetate as the solvent instead of acetone. Again, from the data in Fig. 7, it is clearly observed that similar results were achieved to those using acetone as the solvent. Accordingly, it is further ratified that St can abstract hydrogens under UV radiation.

CONCLUSION

Based on the investigations and discussion presented above, some conclusions can be drawn. The one-step method is not suitable to photograft styrene onto LDPE film. Compared with it, the pre-irradiation method and pre-grafting method seem to be more applicable. When irradiated by UV radiation, styrene itself can abstract hydrogens from H-donors and act as a photoinitiator, which is of important significance to develop photoinitiator-free grafting polymerization systems.

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