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MULTI-STAGE GRAFTING AND SIMULTANEOUS CO- GRAFTING OF ACRYLIC ACID AND VINYL PYRROLIDONE ONTO POLYETHYLENE INDUCED BY γ-RAY IRRADIATION

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Multi-stage Grafting and Simultaneous Co-grafting of Acrylic Acid and Vinyl Pyrrolidone onto Polyethylene Induced by γ-ray Irradiation

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Studies on multi-stage grafting or simultaneous co-grafting of acrylic acid and vinyl pyrrolidone onto polyethylene have been carried out in an attempt to introduce different functional groups into polymers in some ordered states. In the multi-stage grafting, time conversion curves of grafting and graft percent of the second stage depend on the order of monomer to be grafted, and on graft percent of the first-stage grafting. In simultaneous co-grafting, time conversion curves of grafting reaction and graft percent of each monomer depend on monomer ratio in the solution. Effect of graft percent on electric resistance across the grafted film was also found to depend on the mode of multi-stage grafting or simultaneous co-grafting. Results of some trial experiments to establish the grafting conditions are also described.

Keywords: Gamma Ray, Radiation, Grafting, Multi-stage, Co-grafting, Acrylic Acid, Vinyl Pyrrolidone, Polyethylene, Electric Resistance

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γ線照射によるアクリル酸とビニルピロリドンのポリエチレン・フィルムへの多段・複合グラフト重合と同時共グラフト重合

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（1991年6月5日受理）

多種類の官能基をある種の規則性を以てポリマーに導入するために，アクリル酸とビニルピロリドンの多段・複合グラフト重合及び共グラフト重合の研究を行った。多段・複合重合においてはグラフト反応の時間－反応率曲線及び第二段階のグラフト重合のグラフト率は，多段・複合グラフト重合のモード（グラフト重合の順序，等），第一段階のグラフト率等に依存した。同時共グラフト重合ではモノマー溶液中のモノマー比に依存した。10％ KOH 電解溶液中でのグラフト膜の電気抵抗は，グラフト膜のグラフト率に依存するが，その挙動はグラフト重合のモードや第一段階のグラフト率等の条件によりによって異なることが分かった。本報告では，グラフト条件を決めるための幾つかの予備実験の結果についても詳しく述べた。

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1. Introduction

Radiation-induced modification of polymer is one of the most promising methods to give desired properties such as hydrophilic or hydrophobic property, or selective adsorption properties toward particular compound by introducing functional group in polymers. Most of the grafting studies were carried out using one monomer, and even studies on co-grafting in binary even ternary monomer mixture systems are reported[1]. Multi-stage grafting to increase graft percent is also studied[3] to prepare electrolytic membrane and reported that the multiple grafting gave low conductive membrane when compared at the same graft percent. However, little studies have yet reported except one[2] which concerned with additional grafting of styrene or methyl methacrylate onto polyester fiber which had been pre-grafted with acrylic acid.

Since it is interesting to develop methods to introduce different functional group to polymers in organized states in order to obtain membranes for selective permeation or adsorption, studies have been carried out in an attempt to know grafting behavior in the following systems, (1) multi-stage grafting of vinyl pyrrolidone onto polyethylene film pre-grafted with acrylic acid, (2) multi-stage grafting of acrylic acid onto polyethylene film pre-grafted with vinyl pyrrolidone, and (3) simultaneous grafting using monomer mixture of acrylic acid and vinyl pyrrolidone. Grafting of one of these monomers was also studied for comparison. Electric resistance in electrolyte was also measured for grafted membranes prepared by these different methods. This paper describes the results of these studies including those of some preliminary tests. In this report, multi-step grafting means repeated grafting in a monomer solution of same component which is renewed after each step of grafting, while multi-stage grafting is referred as the grafting of one monomer after the other.

2. Experimental

2.1 Kinetic Behavior of Grafting

Simultaneous grafting technique using gamma-rays from cobalt-60 source was employed.

2.1.1 Materials

Polyethylene film used in the present study was obtained from the following sources:

i) HDPE 13 μm thickness, Mitsubishi Petrochemical Company
ii) HDPE 100 μm thickness, Sholex
iii) LDPE 27 μm thickness, Yuasa

Acrylic acid (AA) of research reagent grade was obtained from Nakarai Chemicals Co. and used without purification, since it was reported that the same kinetic results were obtained with monomer without purification when the solution was used with inhibitor[4]. Vinyl pyrrolidone (VP) and Mohr's salt of reagent grade were obtained from Nakarai Chemicals Co. and used without purification. Distilled water was used.

Monomer solution was prepared according to recipe recommended by one of the authors of the following composition[5]: AA:water=50:50 by volume, concentration of Mohr's salt (inhibitor of homopolymerization of monomers) = 4×10^{-3} mol/l, except some solutions, where the concentration was 1×10^{-3} mol/l.

2.1.2 Grafting procedures

The films were cut to pieces of 15 × 50 ~ 15 × 70, 8 × 40 mm and put into a pyrex test tube with a screw cap on the top (30 mm inner diameter (NX50) for earlier experiments (No. 1-38), 13.5 mm inner diameter (N16) for most experiments (No. 39-175, 187-458), and 17 mm (NW20) for later experiments (No.176-186). The known amount of monomer solution (about 50 ml for N16, about 16ml for NX50, and about 30 ml for NW20, respectively) was added in the tube. The solution was deaerated for 3 - 15 min (later experiments revealed that 3 min of deaeration was not enough for complete deaeration as will be described later), and then sealed off carefully from air with the screw cap. The tube was then irradiated in a tube rack set in a cobalt gamma ray source either in natural air cooled condition or in water bath kept at constant temperature. The irradiation temperature was either room temperature from 9 to 15°C in natural air convection cooling condition unless otherwise specified, or 49 - 50°C in a thermostatted water bath. Dose rate in air cooled condition was estimated from data measured by a solid detector dosimeter in Oct. 1990, and that in the water bath was measured using the same dosimeter.

After irradiation, as soon as the film was taken out from the tube, the film was washed with cold water to stop polymerization, and then extracted homopolymer in a hot water bath (50°C) for 4 hrs, before drying in a vacuum oven at 50°C for 5 hrs. The film was sandwiched with filter paper and was stored in a desiccator before weighing.

Weight of the film was measured using a micro balance (Shimadzu) and graft percent at the ith step grafting, G(i), was calculated using following equation:

$$G(i) = (W(i)-W0)/W0 \times 100\ (\%) \qquad (1)$$

where W(i) is weight of film after the ith step grafting with AA, VP, or AA+VP, and W0 is the weight of the original film.

In the multi-stage grafting of one monomer after the other, the graft percent of the second stage grafting (the mth step) after the ith step of the first stage grafting, G(i+m), is defined as follows:

$$G(i+m) = (W(i,m,PE) - W(i,PE)) / W(i,PE) \qquad (2)$$

where W(i,m,PE) is weight after the second-stage grafting (of mth step), and W(i,PE) is weight after the first-stage grafting (of the ith step). To avoid confusion, here, we define "step" as a number of grafting procedures using one monomer, and "stage" as a series of steps of grafting procedures of one monomer.

We also use notations, G_1(AA) and G_2(VP), for graft percent of AA at the first stage and that of VP at the second stage grafting, respectively.

2.1.3 Moisture absorption test

The grafted film taken out from a vacuum oven was put on a weighing pan of the microbalance, and weight indication was taken intermittently. The relative humidity and temperature were kept constant (see Table 3.33), respectively.

2.2 Infrared Spectroscopy

In order to determine the amount of AA and VP separately in the grafted polymer, infrared spectra of PE, AA-grafted PE, VP-grafted PE films, PE film grafted with AA at first stage and then with VP at the second stage, PE film grafted with VP at first stage and then with AA at the second stage, and PE film grafted with AA and VP simultaneously were measured on an FTIR (Perkin-Elmer 1720X) and it was found that the absorption band of carbonyl group of AA appeared at 1716 cm^{-1} different from the bands due to carbonyl of VP (1674 cm^{-1}), indicating that the amount of one monomer grafted can be determined separately without interference due to absorption of carbonyl bands of the other. For IR measurements, only 13 μm film is suitable for obtaining spectra of appropriate absorption range. Accumulation of 10 times was enough to obtain spectrum of high S/N ratio.

2.3 Electric Resistance Measurement

Electric resistance across the grafted film was measured using an apparatus shown in Fig. 2.1. A cylindrical cell made of semi-transparent teflon was designed so that grafted film to be measured is placed at center to separate into two compartments, each equipped with a platinum electrode (7.5 mm in diameter)[6]. The distance to the membrane from the electrode can be varied from 2 mm to 67 mm. Electrolyte (10% KOH aqueous solution in a plastic container which is hanged above the cell) flows into the cell from an injection port placed at bottom of each compartment of the cell and comes out from an exit port equipped on top. This allows easy replacement of electrolyte with removal of air bubbles which may disturb the conductivity measurement. A Kohlrausch type bridge (Yanagimoto Co., MY-8 type) operated at 1000 Hz was used to obtain conductivity. This bridge allows to otain data from 1 to 10^6 ohms. During the measurement, the room was kept constant temperature at 24 ± 1.1°C using a conventional room air conditioner. The measurement was taken intermittently, to know the time required for equilibrium value. All electric conductivity measurements were carried out after stored for enough time in KOH solution to reach equilibrium. Distances between the electrodes were 2 mm and 30 mm, which give electric resistance without sample film (cell resistance, R_0) of 3.5 and 20-26 Ω, respectively. No subtractions of cell resistance were made for all experimental values.

3. Results and Discussion

3.1 Kinetics of Grafting Reaction

[Factors that may or may not affect the measurement of graft percent]

Table 3.1 summarizes the results of preliminary test of grafting using PE film (13 μm HDPE, 40 × 40 mm). In some experiments, it was observed that a part of film (about 10 - 15% of total area) was exposed to air on the solution surface, or a film floated up to a position just below the surface, even the film was not exposed to air. These parts were found to remain ungrafted by visual examination after dying. This indicates that air which may exist in the atmosphere above the liquid level inhibits grafting, and air dissolves into the solution and inhibits grafting of the part of film which is close to the level.

Grafting experiments of four types were carried out in order to know the effect of pre-irradiation and "after-treatments" on graft percent and the results are shown in Table 3.2 and in Fig. 3.1. In type A grafting, the film was irradiated (without solution) in nitrogen atmosphere, and then the irradiated film was taken out in air and then immersed in the monomer solution. In the grafting of B type, the film was irradiated in the deaerated monomer solution, and the film was taken out in air and then immersed in 50°C water for 4 hr. In the grafting of C type the film was irradiated in the deaerated monomer solution, and the film was taken out in air and then immersed in 10°C water for 4 hr. In the D type grafting, the film was irradiated in the deaerated monomer solution, and the film was taken out in air and then just washed and dried in an oven to determine graft percent.

As shown in Table 3.2 and Fig. 3.1, little grafting takes place in type A grafting, indicating that no post grafting occurs. The time conversion of graft percent is almost the same to those observed for graftings of types B, C, and D. This result indicates that little or only a little amount of post-grafting occur in 50°C water if some of monomer possibly remain on the surface of the film. Further, it is indicated that not much difference was observed in the case where careful removal of water soluble homopolymer which possibly exists in the grafting film.

Further experiments were carried out in order to know the effect of "after-treatments" on graft percent, and the results were shown in Table 3.3. There is a little difference among ① graft percents observed when the film after irradiation was not immersed in water bath, but just washed by running water to remove unreacted monomer and ② those when entire or half area of the film after irradiation was immersed in water for 4 hrs.

In Tables 3.4 and 3.5, results of the test in an attempt to know the effect of amount of oxygen present in the monomer solution. In Table 3.4, results were shown for graft percent obtained for ① bubbled nitrogen stream through the solution, ② air saturated (no gas bubbling), and ③ bubbled oxygen stream through the monomer solution. For all runs, gas bubbling time was 3 min. and irradiation time was 120 min. As shown in Table 3.4, little difference was observed among graft percents obtained these conditions. This indicates that (1) gas bubbling time of 3 min. is not enough to replace oxygen in the reaction tubing with nitrogen, and (2) that most oxygen initially dissolved in the tube was consumed during 120 min irradiation time. This was further supported by the results shown in Table 3.5 and Fig. 3.2 where gas bubbling time was extended to 15 min. and irradiation time was changed from 210 min. This time, no grafting took place when oxygen was bubbled, and retardation of the grafting was observed for grafting using air saturated (no gas bubbling) solution when compared with the results obtained for the runs where nitrogen stream was bubbled through the solution.

[Effect of ambient cooling fluid on graft percent]

It was noticed that the sample tubes became warm after grafting reaction due to heat of reaction generated during the reaction when the irradiation was carried out in air under natural convection cooling condition. More effective cooling will be expected when the irradiation is carried out in water at the same temperature. To prove this effect, the grafting experiments were carried out, one in natural air cooled condition and the other in a water bath, where the dose rate and temperature were adjusted as close as possible for the both cases. The results illustrated in Fig. 3.3 support the above possibility showing that higher graft percent was obtained when the irradiation was carried out in air.

[Effect of Mohr's salt concentration on graft percent]

It is known that the graft percent and distribution of grafted polymer in the film are dependent on the concentration of the inhibitor of homopolymerization[5]. The results of grafting under different concentration of Mohr's salt are compared in Fig. 3.4 where Mohr's salt concentrations were 1×10^{-3} mole/l and 4×10^{-3} mole/l while other conditions were the same. Grafting seems to proceed more smoothly by the presence of Mohr's salt concentration of 4×10^{-3} mole/l. Therefore most grafting experiments were done at this concentration of Mohr's salt. (see Table 3.11).

[Effect of storage time of Mohr's salt stock solution]

Mohr's salt is à little unstable and gradually oxidized to form ferric ion when dissolved in water containing oxygen. Therefore, storage of stock solution for at least one day is recommended for stable results. In order to test the range of scattering of data, grafting reactions on 27 μm LDPE and 100 μm HDPE films were carried out in monomer solutions which were freshly prepared just before using Mohr's stock solution (0.008 mole/l) which had been stored for within one hour, one day, and one week. The results were given in Table 3.20A and Fig. 3.5, and Table 3.20 and Fig. 3.6. For both types of films, the similar tendency was observed in that as the storage time becomes longer, higher graft percent was observed at earlier period of grafting reaction, indicating that ferrous ion was oxidized during storage and effective concentration became small. (see Fig. 3.4)

[Effect of storage time of monomer solution]

Grafting experiments were carried out in monomer solutions which were prepared just before grafting and were stored nine days after preparation. Time conversion curves are comparatively shown in Fig. 3.7 (Tables 3.20B, and 3.21), where higher graft percent was observed for the case where freshly prepared monomer solution was used.

[Effect of moisture gain on mass measurement of grafted film]

Grafted film becomes more or less hydrophilic and therefore, absorption of moisture in the grafted film may give larger value of graft percent than real value. Moisture gain of film (100 μ HDPE) is plotted as a function of time exposed in air at 19.9°C and relative humidity of 50% after taken out from a desiccator in which the vacuum dried grafting film had been stored. The result indicates that the weight increase due to moisture gain is only 0.18 percent when the reading of the weight is taken in 20 minutes. Of course, this error depends on the graft percent, but this error may be largest considering that most of our graft percents are lower than 50%. Most of the data were taken within this time. (Fig.

3.8, Table 3.33)

[Time conversion curves of grafting of AA on HDPE at different dose rates]

In order to establish condition of grafting of AA, grafting reactions on 13 μm HDPE film were carried out at two different dose rates, one at 1.35×10^5 rad/h and the other at 2.57×10^5 rad/h at 15°C. The results are shown in Fig. 3.9 (Tables 3.1 and 3.2), where graft percent increased with time, but small retardation period was observed for grafting at smaller dose rate.

[Time conversion curves of grafting of AA on LDPE at different Mohr's salt concentrations]

Grafting reactions on 27 μm LDPE film were carried out in monomer solutions containing Mohr's salt at two different concentrations, one at 1×10^{-3} mol/l at 50°C, and the other at 4×10^{-3} mol/l at 9°C. The results are shown in Fig. 3.10 (Table 3.19) and Fig. 3.11 (Table 3.20A), respectively. Both systems were found to give graft polymers of high graft percent, but the latter system is possibly suitable to obtain graft polymer in which cross sectional distribution of grafted polymer is more uniform.

[Time conversion curves of grafting of VP on HDPE]

Grafting reactions on 13 μm HDPE film were carried out in monomer solutions containing Mohr's salt at 4×10^{-3} mol/l at 50°C and at dose rate of 1.18×10^5 rad/h and graft percent is plotted as a function of irradiation time in Fig. 3.12 (see also Table 1.23). Although small retardation was observed at earlier period of grafting, graft percent of as high as 60% was successfully obtained by 1 hr irradiation.

Similar graftings were carried out at two different dose rates, one at 1.23×10^5 rad/h and the other at 2.23×10^5 rad/h at 13.8°C. The results are shown in Fig. 3.13 (Table 3.8A), where graft percent increased with time, but small retardation period was observed for grafting at two dose rates. The same data are shown in Fig. 3.14 (Table 3.8A) where dose in kGy is taken on abscissa. Higher graft percent was obtained for grafting carried out at lower dose rate when compared at the same dose, as is expected from general law of square root of dose rate.

[Comparison of reactivity of AA and VP]

Time conversion curves for grafting of AA and VP on 13 μ HDPE films are compared in Fig. 3.15 (Tables 3.6, 3.8 and 3.11) at the same conditions of grafting. Later retardation was observed for grafting of VP than that for AA, but rate of grafting of VP becomes higher after initial retardation period.

[Effect of temperature on time conversion curve for grafting of AA onto 100 μm HDPE film]

Grafting reactions onto 100 μm HDPE film were carried out in monomer solutions containing Mohr's salt at 4×10^{-3} mol/l at two different temperatures, one at 49°C and the other at 10.8°C in the thermostatted water bath, and at dose rate of 0.99×10^{5} rad/h, and graft percent is plotted as a function of irradiation time in Fig. 3.16 (see also Tables 3.16 and 3.18). Grafting at 10.8°C was not very promising because monomer solution became very viscous at 350 min. irradiation before graft polymer of high graft percent was obtained at this temperature.

[Effect of dose rate on time conversion curve for grafting of AA onto 100 μm HDPE film]

Grafting reactions on 100 μm HDPE film were carried out in monomer solutions containing Mohr's salt at 4×10^{-3} mol/l at 50°C at three different dose rates: 2.11×10^{3} rad/h, 2.66×10^{4} rad/h, and 1.18×10^{5} rad/h, and graft percent is plotted as a function of irradiation time in Fig. 3.17 (see also Tables 3.25, 3.26 and 3.20B). It is noted that the graft percent is very low to allow enough graft percent for conductivity measurements at dose rates below 2.11×10^{3} rad/h.

[Multi-step grafting and single-step grafting of AA onto PE film]

In Fig. 3.18 (Table 3.17), time conversion curves are compared for grafting of single step (circles) and for two-step grafting (square and triangle symbols). The time conversion curves obtained by these methods agree well with one another and this indicates that grafting with single step and that with multi-step proceed with same mechanism.

[Graft percent of VP at the second stage and that of AA previously grafted on AA in the first stage and vice versa]

The PE films of AA grafted at different graft percents were obtained at the first stage grafting by changing irradiation time while other reaction conditions were kept constant (Tables 3.10 and 3.11). The second stage grafting of VP onto the previously AA grafted PE film was carried out for 10, 20.3, 25, and 40.3 min. (Tables 3.9B and 3.12). The graft percent of VP was plotted as a function of graft percent of AA in Fig. 3.19 where the irradiation time at the second stage grafting is taken as parameter. It is noted that a peak appeared at about 20% graft percent of AA for all curves obtained. A qualitative explanation will be that at lower graft percent of AA, VP may easily diffuse into polymer to be grafted with polyethylene chain and graft percent of VP increased with increasing graft percent of AA, but at higher graft percent of AA, VP diffused into polymer may have difficulty to encounter ungrafted PE chain to graft (VP may not be grafted onto PAA chain in the grafted polymer).

Similar experiment to that mentioned above was carried out in which the order of grafting was reversed: VP grafted at the first stage (Tables 3.23, 3.24 and 3.8A) and then AA at the second stage (Tables 3.7 and 3.8B). The graft percent of AA was plotted as a function of graft percent of VP in Figs. 3.20 and 3.21 (in extended scale at the lower graft percent of VP below 25%) where the irradiation time at the second stage grafting is taken as parameter. It is noted that no peak appeared on all curves obtained and graft percent of AA monotonously increased with increasing graft percent of VP. An explanation for this will be that AA may easily diffuse into polymer resulting in increase of graft percent of AA as the graft percent of VP increased. This is possibly because grafted PVP chain has $-CH_2-$ units to be grafted with AA (AA may be grafted onto PVP chain in the grafted polymer).

[Multi-step grafting of AA as the first stage grafting and multi-step grafting of VP as the second stage grafting]

Multi-step grafting of AA was carried out onto 100 μm PE film to obtain film of high graft percents, each step being about 40 min grafting time just before the monomer solution becomes too viscous the film to be taken out (see Tables 3.13A and 3.13B, for details). Then, four-step grafting of VP initiated on the AA-grafted films, one, after the 4th, and the other, after the 5th step grafting (see Tables 3.14A and 3.14B, for details). Graft percent at each step grafting is plotted as a grafting time (irradiation time) in Fig. 3.22 (Table 3.15), where it is noted that grafting accelerates as the number of steps of VP grafting increases, and the shape of time-conversion curves are the same each other for the second stage grafting after the 4th step of the first stage grafting and that after the first stage of the 5th step. This is perhaps related with that number of $-CH_2-$ units of VP chain on which the grafting of VP occurs increases with increasing number of steps of the VP grafting.

[Simultaneous co-grafting of AA and VP onto 13 μm HDPE film]

In order to determine the graft percent of AA and VP separately in the simultaneous co-grafting using monomer solution containing both AA and VP, it is necessary to know the relation between amount of grafted monomer and optical density of characteristic bands in IR spectrum of each component. In Fig. 3.23 (Table 3.1), time conversion curve of AA grafting and change of O.D. at 1716 cm^{-1} are shown. It is clear from the figure, that graft percent increase is closely related to increase of O.D. indicating that simple correlation exists between graft percent of one component and O.D., which will be described in a later section. Graftings onto 13 μm HDPE film were carried out in monomer solutions containing AA and VP in the volume ratio (AA:VP) of 50:0, 40:10, 30:20, 20:30, 10:40, and 0:50 at 12°C (Table 3.10). Time conversion curves obtained in these monomer solutions are shown in Fig. 3.24 where rate of grafting increases with increasing content of AA in the monomer solution at higher concentration of AA. This is further illustrated in Fig. 3.25 where graft percents are plotted as a function of content of AA in the monomer solution, irradiation time being taken as parameter.

The graft percent of (AA+VP) obtained by gravimetric method can be separated into graft percent of each component using absorption of carbonyl bands of AA and VP, which appeared at different wave number, as mentioned in the later section. Graft percent of VP in the co-grafted film thus determined is plotted as a function of graft percent of AA determined by the same way in Fig. 3.26 where dotted lines indicate hypothetical relation between graft percent of VP and that of AA assuming that the both monomer graft with polymer with equal reactivity. All experimental points deviate lower from those of the corresponding ideal lines, indicating that AA is more reactive than VP.

Mole fraction of AA in the grafted film is plotted against that in monomer solution in Fig. 3.27 where experimental points deviate upward from the dotted line indicating that monomer reactivity ratios of AA and VP are about 2 and 1/2, respectively.

Similar co-grafting experiment was carried out on 100 μm HDPE film to obtain films to be used for conductivity measurements and graft percent of (AA+VP) is plotted as a function of irradiation time in Fig. 3.28 (Tables 3.29A and B), monomer ratio in the monomer solution being taken as parameter. Again higher rate of grafting was observed in the monomer solution containing AA in higher concentration. No separate determination of graft percent of each component is possible since films are too thick to obtain proper IR spectrum.

3.2 Infrared Spectra of Grafted Films

Infrared spectra of original PE film, AA grafted film, and VP grafted film are shown in Fig. 3.29, where it is noted that carbonyl bands appeared at 1716 cm^{-1} for AA grafted film and 1674 cm^{-1} for VP grafted film, as mentioned in the previous section. Therefore, it is possible to determine amount of monomer in the grafted film separately. Absorption intensities of carbonyl bands are listed in Tables 2.1 ~ 2.5 as a function of graft percent.

Figs. 3.30 and 3.31 show infrared spectra obtained for simultaneous co-grafted film of AA and VP in the monomer solution containing AA and VP at different ratios, (AA:VP), 40:10, 30:20, 20:30, 10:40. As the VP content in the monomer solution increases, the peak due to carbonyl group of AA decreased and the peak due to carbonyl of VP increased, and at the same time, peak position of carbonyl band of AA shifted toward higher wave number and that of VP shifted toward higher wave number assymptotically to the characteristic wave number of VP (1674 cm^{-1}).

This is more clearly seen in differential spectrum shown in Fig. 3.32 where spectra of original PE film, AA (AA grafted film - PE), and VP (VP grafted film - PE) are subtracted from spectrum of (AA+VP) co-grafted film. Remaining peaks at 1733 cm^{-1} and at 1631 cm^{-1} in the differential spectrum may indicate presence of carbonyl groups which interact one another.

In Fig. 3.33 through Fig. 3.36, infrared spectra of film grafted with AA at the first stage and then grafted with VP at the second stage are shown. The spectra were arranged in order of increasing ratio of graft percent of AA to that of VP: G1(AA)/G2(VP), but graft percents were not considered, because spectrum shape depends on graft ratio and is little affected by graft percent.

In Fig. 3.37 through Fig. 3.39, infrared spectra of film grafted with VP at the first stage and then grafted with AA at the second stage are shown. The spectra were shown in order of increasing ratio of graft percent of AA to that of VP: G(AA)2/G1(VP), but again graft percents were not considered.

Shape of carbonyl bands changed with increasing graft percent ratio, G(AA)/G(VP) as observed for simultaneous co-grafting of AA and VP (Figs. 3.30 and 3.30).

In Fig. 3.40 through Fig. 3.48, a pair of infrared spectrum of a film grafted with AA at the first stage and that of the film then grafted with AA at the second stage on the same film are shown. The spectra are shown in order of increasing ratio of graft percent of AA to that of VP: G1(AA)/G2(VP), but again graft percents were not considered. It is observed that intensity of the absorption of carbonyl bands due to AA grafted at the first stage decreased after the second grafting with VP. The position of the peak maximum also shifted toward higher wave number by the same procedure. The results seem to indicate that the size of the film expanded by the second stage grafting as confirmed by length measurements (Table 3.39) and that there is some interaction between the carbonyl groups of AA and VP introduced in the PE film.

Comparison of the spectra of the grafted film observed for AA-VP multi-stage grafting and those for VP-AA grafting, the spectra obtained by the former show three distinct small shoulders at 1460 - 1600 wavelength region (Figs. 3.34C and 3.37A). Subtractions of spectrum of original PE, of AA and of VP from the AA-VP grafting film gave complex spectrum which could not be identified at present (Fig. 3.49). Result of similar subtraction on the spectra of the film obtained for the latter grafting, (VP-AA), is shown in Fig. 3.50, where simpler residual structure of carbonyl bands is observed than that obtained for the former (AA-VP).

Optical density of AA grafted film at 1716 cm^{-1} (○) and that of VP grafted film at 1674 cm^{-1} (Δ) are plotted as a function of graft percent in Fig. 3.51 (Tables 3.34 ~ 3.38). In the same figure, sum of optical densities at ca. 1716 cm^{-1} and ca. 1674 cm^{-1} is plotted as a function of total graft percent for AA-VP grafted film (□), for VP-AA grafted film (∇), and for simultaneous co-grafted film with AA and VP (◇). The points are scattered along a straight line. The film was expanded or deformed by the grafting, and therefore, optical densities were corrected using characteristic band of methylene group, according to the following equation:

$$A' = A\ (B_0/B)$$

where A is the optical density, B_0 is optical density of $-CH_2-$ at 723 cm^{-1} before grafting (B_0 = 0.333 for 13 μ HDPE) and B, optical density of the same band after the grafting. The plots after the correction are shown in Fig. 3.52 where narrower scattering of points was obtained. The calibration line was used to determine graft percent of AA and that of VP in the simultaneously co-grafted films.

3.3 Electric Resistance Across Grafted Films

It was found that electric resistance decreased with time after the film was set in the cell containing KOH solution, and reached equilibrium value which is almost close to the electric resistance without film between the two compartments, due to swelling and salt formation with potassium ion. Two examples are shown in Fig. 3.53, and three in Fig. 3.54 where electric resistance was plotted as a function of swelling time (see also Tables 3.48 ~ 3.53). Rate of the decrease depends on graft percent. All data reported thereafter are electric resistance taken when it reached equilibrium value.

In Fig. 3.55, electric resistances across the grafted films are plotted as a function of graft percent for AA grafted film and VP grafted film (Tables 3.22 and 3.27). In the case of AA, electric resistance decreased with increasing graft percent reaching a plateau at 30%, and then decreased again when the graft percent increased further. On the other hand, in the case of VP grafted film, electric resistance decreased monotonously with increasing graft percent.

In Fig. 3.56, the same plots are shown for simultaneous co-grafted film in which the AA:VP in the monomer solution is altered from 20:30 to 45:5 (Tables 3.29A and 3.29B). The electric resistance decreased with increasing graft percent reaching plateau value and then again decreased with increasing graft percent. The graft percent at which sharp decrease after plateau occurs depends on composition of monomer solution and it increased with increase of AA content in the monomer solution.

In Fig. 3.57, the electric resistances across the grafted films of two types, one, VP-AA type (Tables 3.28A ~ 3.28C) and the other, AA-VP type (Tables 3.30A ~ 3.30D; Tables 3.31A, 3.31B and 3.32) are plotted as a function of graft percent of second stage. For grafted films of VP-AA type, five curves are shown for the film grafted with VP at graft percent of 1, 2, 5, 12, and 25%, graft percent at the first stage being taken as parameter. Except the last curve, almost similar curves observed for AA grafted film were obtained, where the graft percent decreased and reached a plateau value with increasing graft percent of VP, and after plateau value, the resistance decreased again with increasing graft percent. The graft percent at which the decrease of resistance after plateau value occurs depends on the graft percent of the first stage, and they are 35 and 40% for graft percent of VP of 1 and 2%, respectively, and they are about 65% for the film grafted with VP at 5 and 12%, respectively. No such irregular point was observed for the curve obtained for the film grafted with VP at 25%, but a monotonously decreasing curve with increasing graft percent was observed.

For grafted films of AA-VP type, only one curve was obtained for the film grafted with AA at graft percent of 5%. Marked difference was observed in shape of the curves between the AA-VP grafted and the VP-AA grafted films, when the curves of two types of films are compared at the same graft percent (5%) of the first stage. It is noted that for the film grafted with AA at the first stage (AA-VP type), resistance decreased monotonously, while resistance of the film grafted with VP (VP-AA type) decreased step wise. No explanation for this is proposed at present, but may be related with difference found between IR spectra of the two types, which may reflect structural change of the two.

Step wise decrease found for AA grafted film, grafted film co-grafted with AA and VP, and grafted film of VP-AA type (G1(VP) < 12%) is possibly related with that grafted zone with AA which exists in the surface region of the film at early stage of grafting penetrated into the film connecting the grafted zones of both surface regions when graft percent exceeds certain critical value.

4. Conclusions

In the multi-stage grafting of AA and VP, grafting behavior was found to depend on which monomer was grafted at the first stage and graft percent and rate of grafting at the second stage depends on graft percent of the first stage. It is also noted that shape of carbonyl bands appeared in the infrared spectra also depends on the mode of the multi-stage grafting suggesting that the structure of the graft layer is different among these grafted films prepared by the different modes of grafting. Difference in structure and depth distribution[3],[5] of the grafted chain of the two monomers may result in observed different dependence of resistance on graft percents both at the first stage and the later stage.

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Authors wish to express their sincere thanks to Prof. Okada of Oita University for his comments and encouragements on this work and to Mr. F. Murakami of Sanwa Kako Co. for giving us polyethylene film on which our grafting experiments can be carried out. We are also indebted to Mr. K. Matsuda of our Laboratory for his help in dosimetry in the gamma ray field. One of the authors (QGM) wishes to express his deepest appreciation to Science and Technology Agency of Japanese Government for support to study in JAERI, and also to Mr. Wu Ying Hua, vice director general, and Mr. Hong Yong Han, vice director, of South-West Center of Nuclear Reactor Design Research Institute for permission to leave for Japan.

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2) I. Sakurada, T. Okada, K. Kaji, and A. Tsuchiya, "Radiation-Induced Graft Copolymerization to Polyester XIX. Resistivity of Poly-

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Appendix

i) PEAA###, PEAV###T, PEVA###T, PEVA### are file names used for infrared spectrum recordings. Here, PE, A, and V mean polyethylene, acrylic acid, and vinyl pyrrolidone, respectively. T means the graftings were carried out in two stages, in the order of A and V as indicated in the file name. File name without T is for one stage grafting: for the name with A and V, simultaneous co-grafting in monomer mixture of A and V was carried out. ###SUBVAPE means residual spectrum obtained by subtraction of spectra of A, V, and PE from spectrum indicated by ###. Number ### is the sample number appeared in Tables in Chapter 1.

ii) f appeared in cations to infrared spectra means scale factor of the spectrum indicated by capital alphabet. f of spectrum A is always 1.000 showing absorbance scale on the ordinate is correct. Corrected absorbance for the other spectrum, B and C, is that multiplied by f-factor.

iii) When one piece of film is splitted into two fragments, and further experiments are carried out on these fragments, the sample number is indicated as ###($-&) where ### is original sample number, $ is step or stage number, and & indicates fragment number.

iv) VP-AA multi-stage grafting means graft procedure in which AA grafted at the first stage and VP at the second stage.

v) (AA+VP) grafting indicates simultaneous one stage co-grafting in monomer solution containing AA and VP.

vi) G1(AA) and G2(VP) indicate graft percent of AA at the first stage, and that of VP at the second stage, respectively.

Table 3.1 Graft percent as a function of irradiation time at different dose rates

(Film: 13 μm HDPE, 40×40cm)

Sample No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
1	30	1.35×10^5	15.2	no	$AA:H_2O+Fe^{2+}$	5.1	
2	60	"	"	no	$50:50+4\times10^{-3}$	15.3	①
3	90	"	"	no	"	26.1	②
4	120	"	"	no	"	47.1	③
5	60	"	15	no	"	15.4	
6	90	"	"	no	"	34.2	
7	120	"	"	no	"	45.3	④
8	180	"	"	a little	"	49.1	
9	240	"	"	a little	"	61.6	④
10	30	2.57×10^5	"	no	"	8.8	④
11	60.	"	"	no	"	26.1	④
12	90	"	"	no	"	30.0	④
13	120	"	"	a little	"	45.8	④
14	116	"	"	a little	"	34.6	④
15	176	"	"	viscous	"	50.0	
16	236	"	"	viscous	"	65.0	

① A part of film (about 15% of total area) was exposed to air on the solution surface, and was found to remain ungrafted by visual examination after dying.

② A part of film (about 10% of total area) was exposed to air above the solution surface.

③ A central part of the film (about 20% of total area) came close below the solution surface (not exposed to air) ungrafted.

④ A small part of a corner of the film came closer to the solution level, and this part was not grafted.

⑤ A small part of a corner of the film came closer to the solution level, and this part was not well grafted.

Table 3.2 Effect of pre-irradiation and after-treatments on graft percent

A; irradiation without solution in nitrogen atmosphere, the irradiated film was taken out in air and then immersed in the monomer solution.

B: irradiated in the deaerated monomer solution, and the film was taken out in air and then immersed in 50°C water for 4 hr.

C: irradiated in the deaerated monomer solution, and the film was taken out in air and then immersed in 10°C water for 4 hr.

D: irradiated in the deaerated monomer solution, and the film was taken out in air and then just washed and dried in an oven to determine graft(%).

	Sample No.	Irrad. Time (min)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
A	17	60	2.57×10^5	15	no	$AA:H_2O+Fe^{2+}$	0.27	
	18	120	"	"	no	$50:50+4\times10^{-3}$	-0.36	
	19	180	"	"	no	"	-0.35	
B	20	60	"	"	no	"	43.3	
	21	120	"	"	a little	"	59.2	
	22	180	"	"	viscous	"	67.8	
C	23	60	"	"	no	"	48.7	
	24	120	"	"	some	"	61.7	
	25	180	"	"	viscous	"	69.7	
D	26	60	"	"	no	"	45.4	
	27	120	"	"	some	"	59.2	
	28	180	"	"	viscous	"	67.7	

Table 3.3 Effect of after-treatments on graft percent

① was not immersed in water bath, and just washed by running water to remove unreacted monomer.
② total area of the film was immersed in water for 4 hr.
③ half of area of the film was immersed in water for 4 h before dried in an oven.

Sample No.	Irrad. Time (min)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
29	120	2.57×10^5	14	viscous	AA:H_2O+Fe^{2+}	61.8	①
30	"	"	"	"	50:50+4×10^{-3}	56.7	②
31	"	"	"	"	"	58.4	②
32	"	"	"	"	"	54.6	③
33	"	"	"	"	"	57.9	③

Table 3.4 Effect of amount of oxygen present in the monomer solution (1)

① bubbled nitrogen stream through the solution for 3 min.
② air saturated (no gas bubbling).
③ bubbled oxygen stream through the monomer solution for 3 min.

Sample No.	Irrad. Time (min)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
34	120	2.57×10^5	14	viscous	AA:H_2O+Fe^{2+}	60.9	①
35	"	"	"	"	50:50+4×10^{-3}	58.9	②
36	"	"	"	"	"	60.3	②
37	"	"	"	v.viscous	"	64.0	③
38	"	"	"	v.viscous	"	67.2	③

Table 3.5 Effect of amount of oxygen present in the monomer solution (2)

① bubbled nitrogen stream through the solution for 15 min.
② air saturated (no gas bubbling).
③ bubbled oxygen stream through the monomer solution for 15 min.

Sample No.	Irrad. Time (min)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
39	15	2.25×10^5	14.2	no	$AA:H_2O+Fe^{2+}$	3.7	①
40	30	"	"	no	$50:50+4\times10^{-3}$	9.7	①
41	60	"	"	some	"	41.6	①
42	120	"	"	viscous	"	68.6	①
43	180	"	"	highly viscous	"	85.8	①
44	210	"	"	can't take out	"		①
45	15	"	"	no	"	1.0	②
46	30	"	"	a little	"	5.5	②
47	60	"	"	a little	"	19.2	②
48	120	"	"	viscous	"	72.8	②
49	180	"	"	highly viscous	"	90.1	②
50	210	"	"	can't take out	"		②
51	15	"	"	no	"	-0.9	③
52	30	"	"	no	"	-0.7	③
53	60	"	"	a little	"	3.2	③
54	120	"	"	a little	"	0.8	③
55	180	"	"	can't take out	"		③
56	210	"	"	can't take out	"		③

Table 3.6 One-stage grafting of AA, VP, and mixture of AA and VP

(film, 13 μm HDPE, 15×50 mm)

Sample No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
57	15	2.23×10^5	22	a little	$VP:H_2O+Fe^{2+}$	2.0	
58	30	"	"	highly viscous	$50:50+1\times10^{-3}$	25.1	
59	45	"	"	very high	"		①
60	45	"	"	very high	"		①
61	45	"	"	very high	"		①
62	15	"	"	some	$AA:H_2O+Fe^{2+}$	23.2	
63	30	"	"	highly viscous	$50:50+1\times10^{-3}$	44.8	②
64	37	"	"	very high	"	45.8	
65	37	"	"	very high	"	48.3	
66	15	"	"	some	$VP:AA:H_2O+Fe^{2+}$	9.9	
67	30	"	"	highly viscous	$30:30:40+1\times10^{-3}$		③
68	37	"	"	very high	"		③
69	37	"	"	viscous	"	11.5	④

① No.59-61, monomer solution too viscous to take out film.

② after taken out from water both and dried in a vacuum oven, a small amount of homopolymer was found on the film.

③ No.67-68, films were taken out from the solution, but a small amount of homopolymer on the films can not be removed.

④ No.69, diameter of the sample tube is a little larger than that of holes of the tubing stand, and therefore, the tube had to be set for irradiation at possibly lower dose rate.

Table 3.7 Grafting of AA on PE film previously grafted with VP and that of VP on AA grafted PE (preliminary test)

(film 13 μm HDPE)

Sample No.	G_1 (%)	Sample No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
57	G(VP)=2.0	70	30	2.23×10^5	14.2	highly viscous	AA:H_2O+Fe^{2+}	46.1	
58	G(VP)=25.1	71	30	"	"	highly viscous	50:50+1×10^{-3}	83.2	
62	G(AA)=23.2	72	30	"	"	highly viscous	VP:H_2O+Fe^{2+}	20.0	
63	G(AA)=44.8	73	30	"	"	highly viscous	50:50+1×10^{-3}	1.8	①
64	G(AA)=45.8	74	8	"	"	no	"	0.9	
65	G(AA)=48.3	75	30	"	"	viscous	"	1.4	

① Homopolymer was found on the AA grafted film, but it was dissappeared after the second grafting.

Table 3.8 Grafting of AA on PE film previously grafted with VP
(film: 13 μm HDPE 15×50mm)

Relation between graft percent of AA and that of previously grafted VP

(A) Grafting of VP onto PE (the first stage grafting)

Sample No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
76	15	2.23×10^5	13.8	a little	$VP:H_2O+Fe^{2+}$	1.1	
77	10	"	"	no	$50:50+1\times10^{-3}$	0.8	
78	20	"	"	some	"	4.6	
79	30	"	"	highly viscous	"	21.2	
80	25	"	"	highly viscous	"	8.7	
81	40	1.23×10^5	"	highly viscous	"	21.2	
82	20	"	"	no	"	0.7	
83	30	"	"	some	"	2.9	
84	50	"	"	highly viscous	"	20.9	
85	70	"	"	very high	"		①

① The solution too viscous to take out film

(B) Grafting of AA onto VP grafted PE (the second stage grafting)

No.	G 1(VP) (%)	No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
76	1.1	86	40	1.23×10^5	12.5	viscous	$AA:H_2O+Fe^{2+}$	28.8	
77	0.8	87	20	1.23×10^5	"	viscous	$50:50+1\times10^{-3}$	7.5	
82	0.7	88	20.3	2.23×10^5	"	viscous	"	27.8	
79	21.2	89	40	1.23×10^5	"	highly viscous	"	84.6	
84	20.9	90	20.3	2.23×10^5	"	viscous	"	78.6	
78	4.6	91	40	1.23×10^5	"	viscous	"	52.1	
80	8.7	92	20.3	2.23×10^5	"	viscous	"	54.5	
81	21.2	93	20	1.23×10^5	"	some	"	34.8	
83	2.9	94	20	1.23×10^5	"	some	"	17.3	

Table 3.9 Grafting of VP on PE film previously grafted with AA
(film: 13 μm HDPE 15×50mm)

Relation between graft percent of VP and that of previously grafted AA

(A) Grafting of AA onto PE (the first stage grafting)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
95	10	1.23×10^5	12.5	very little	$AA:H_2O+Fe^{2+}$	2.5	
96	10	"	"	very little	$50:50+1\times10^{-3}$	3.6	
97	20	"	"	a little	"	8.4	
98	20	"	"	a little	"	5.8	
99	30	"	"	viscous	"	17.7	
100	30	"	"	viscous	"	20.3	
101	15	"	"	very little	"	1.0	
102	15	"	"	very little	"	0.6	
103	40	"	"	highly viscous	"	40.7	
104	40	"	"	highly viscous	"	43.2	
105	50	"	"	highly viscous	"	29.8	
106	50	"	"	very high	"	51.4	

(b) Grafting of VP onto AA grafted PE (the second stage grafting)

No.	G_1(AA)	No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
95	2.5	107	20.3	1.23×10^5	12	a little	$VP:H_2O+Fe^{2+}$	2.3	
97	8.4	108	"	"	"	"	$50:50+1\times10^{-3}$	2.8	
99	17.7	109	"	"	"	"	"	4.9	
101	1.0	110	"	"	"	"	"	2.3	
103	40.7	111	"	"	"	"	"	2.1	
105	29.8	112	"	"	"	"	"	2.7	
96	3.6	113	40.3	"	"	very high	"	9.1	
98	5.8	114	"	"	"	"	"	13.9	
100	20.3	115	"	"	"	"	"	17.0	
102	0.6	116	"	"	"	"	"	11.2	
104	43.2	117	"	"	"	"	"	4.2	
106	51.4	118	"	"	"	"	"	3.4	

Table 3.10 One-stage co-grafting of AA and VP
(film: 13 μm HDPE 15×50mm)
Composition of AA and VP in monomer and graft percent

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
119	10	1.23×10^5	12	no	$AA:H_2O+Fe^{2+}$	2.3	
120	20	"	"	no	$50:50+1\times10^{-3}$	9.6	
121	30	"	"	some	"	18.8	
122	40	"	"	viscous	"	33.2	
123	10	"	"	no	$AA:VP:H_2O+Fe^{2+}$	2.7	
124	20	"	"	no	$40:10:50+1\times10^{-3}$	7.1	
125	30	"	"	viscous	"	12.6	
126	40	"	"	viscous	"	14.2	
127	10	"	"	a little	$AA:VP:H_2O:Fe^{2+}$	1.0	
128	20	"	"	a little	$30:20:50+1\times10^{-3}$	-6.4	①
129	30	"	"	some	"	5.7	
130	40	"	"	viscous	"	12.2	
131	10	"	"	some	$AA:VP:H_2O+Fe^{2+}$	1.0	
132	20	"	"	highly viscous	$20:30:50+1\times10^{-3}$	4.0	
133	30	"	"	highly viscous	"	6.2	
134	40	"	"	very high	"	12.3	
135	10	"	"	a little	$AA:VP:H_2O+Fe^{2+}$	0.7	
136	20	"	"	very high	$10:40:50+1\times10^{-3}$	3.3	
137	30	"	"	very high	"	6.1	
138	40	"	"	v.v. high	"	10.3	
139	10	"	"	no	$VP:H_2O+Fe^{2+}$	-0.4	
140	20	"	"	a little	$50:50+1\times10^{-3}$	1.4	
141	30	"	"	a little	"	5.1	
142	40	"	"	highly viscous	"	9.5	

① No.128 was broken into three pieces when it was washed in water to remove homopolymer. A small part of the sample may be lost.

Table 3.11 Preparation of PE film grafted with AA at different graft percent to be used for the second stage grafting of VP (c.f. Table 12)

(film: 13 μm HDPE 15×50mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
143	5	1.23×10^5	10.5	no	$AA:H_2O+Fe^{2+}$	1.72	
144	10	"	"	a little	$50:50+1\times10^{-3}$	3.02	
145	15	"	"	some	"	6.09	
146	20	"	"	some	"	8.29	
147	30	"	"	viscous	"	18.54	
148	40	"	"	highly viscous	"	33.9	
149	5	"	"	no	"	2.48	
150	10	"	"	a little	"	5.26	
151	15	"	"	some	"	7.07	
152	20	"	"	some	"	6.19	
153	30	"	"	viscous	"	24.52	
154	40	"	"	highly viscous	"	36.94	
155	5	"	"	no	"	2.56	
156	10	"	"	a little	"	5.9	
157	15	"	"	a little	"	6.51	
158	20	"	"	some	"	9.12	
159	30	"	"	viscous	"	35.02	
160	40	"	"	highly viscous	"	35.02	

Table 3.12 Grafting of VP onto PE films previously grafted with AA at different graft percents (c.f. Table 11)

(film: 13 μm HDPE 15×50mm)

No.	G (AA) (%)	No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
143	1.72	161	35	1.23×10^5	10.5	highly viscous	$VP:H_2O+Fe^{2+}$	6.86	
144	3.02	162	"	"	"	"	$50:50+1\times10^{-3}$	9.23	
145	6.09	163	"	"	"	"	"	10.3	
146	8.29	164	"	"	"	"	"	12.9	
147	18.54	165	"	"	"	"	"	14.5	
148	33.9	166	"	"	"	"	"	4.92	
149	2.48	167	25	"	"	some	"	2.52	
150	5.26	168	"	"	"	"	"	3.33	
151	7.07	169	"	"	"	"	"	4.26	
152	6.19	170	"	"	"	"	"	3.76	
153	24.52	171	"	"	"	"	"	5.01	
154	36.94	172	"	"	"	"	"	1.94	
155	2.56	173	10	"	"	no	"	0.31	
156	5.9	174	"	"	"	"	"	-0.09	
157	6.51	175	"	"	"	"	"	0.79	
158	9.12	176	"	"	"	"	"	0.92	
159	35.02	177	"	"	"	"	"	1.40	
160	35.02	178	"	"	"	"	"	1.31	

Table 3.13A Preparation of AA grafted films of different graft percents using multi-stage grafting technique to be used for later stage grafting with VP

(film: 100 μm HDPE 15×60mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
179	40	1.22×10^5	10	viscous	AA:H_2O+Fe^{2+}	2.58	
180	"	"	"	"	50:50+1×10^{-3}	3.00	
181	"	"	"	"	"	*	
182	"	"	"	"	"	*	
183	"	"	"	"	"	*	
184	"	"	"	"	"	*	
185	"	"	"	"	"	*	
186	"	"	"	"	"	*	

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
181(2)**	44	1.22×10^5	10	highly viscous	AA:H_2O+Fe^{2+}	9.15	
182(2)	"	"	"	"	50:50+1×10^{-3}	10.09	
183(2)	"	"	"	"	"	10.05	
184(2)	"	"	"	"	"	9.86	
185(2)	"	"	"	"	"	9.70	
186(2)	"	"	"	"	"	8.74	

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
179(2)**	40.5	1.22×10^5	10	highly viscous	AA:H_2O+Fe^{2+}	*	
180(2)	"	"	"	"	50:50+1×10^{-3}	*	
181(3)	"	"	"	"	"	14.34	
182(3)	"	"	"	"	"	16.39	
183(3)	"	"	"	"	"	*	
184(3)	"	"	"	"	"	*	
185(3)	"	"	"	"	"	*	
186(3)	"	"	"	"	"	*	

* The film was used for later stage grafting without measuring graft percent at this stage.

** Numbers in parentheses indicate grafting stage at later stage grafting.

Table 3.13B Preparation of AA grafted films of different graft percents using multi-stage grafting technique to be used for later stage grafting with VP (Continued)

(film: 100 μm HDPE 15×60mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
179(3)**	39	1.22×10^5	10	highly viscous	$AA:H_2O+Fe^{2+}$	*	
180(3)	"	"	"	"	$50:50+1\times10^{-3}$	*	
183(4)	"	"	"	"	"	22.54	
184(4)	"	"	"	"	"	21.21	
185(4)	"	"	"	"	"	*	
186(4)	"	"	"	"	"	*	

179(4)	43	1.22×10^5	10	highly viscous	$AA:H_2O+Fe^{2+}$	16.51	
180(4)	"	"	"	"	$50:50+1\times10^{-3}$	17.48	
181(4)	"	"	"	"	"	20.19	
182(4)	"	"	"	"	"	23.69	
185(5)	"	"	"	"	"	29.12	
186(5)	"	"	"	"	"	25.77	

* The film was used for later stage grafting without measuring graft percent at this stage.

** Numbers in parenthese indicate grafting stage at later stage grafting.

Table 3.14A Grafting of VP onto PE previously grafted with AA at different graft percents prepared by the experiments in Table 13A and B.

(film: 100 μm HDPE 15×60mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
179(5-2)** ***	40	1.22×10^5	10	highly viscous	$VP:H_2O+Fe^{2+}$	1.10	
179(5-3)	"	"	"	"	$50:50+1\times10^{-3}$	*	
180(5-2)	"	"	"	"	"	*	
180(5-3)	"	"	"	"	"	*	
181(5-2)	"	"	"	"	"	1.49	
181(5-3)	"	"	"	"	"	*	
182(5-2)	"	"	"	"	"	*	
182(5-3)	"	"	"	"	"	*	
185(6-2)	"	"	"	"	"	2.07	
185(6-3)	"	"	"	"	"	*	
186(6-2)	"	"	"	"	"	*	
186(6-3)	"	"	"	"	"	*	

* The film was used for later stage grafting without measuring graft percent at this stage.

** Numbers in parentheses indicate grafting stage at later stage grafting.

*** Numbers after dash in parentheses indicate fragment piece number split from the original film.

Table 3.14B Grafting of VP onto PE previously grafted with AA at different graft percents prepared by the experiments in Table 13A and B.

(film: 100 μm HDPE 15×60mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
179(6-3)** *** *	40	1.21×10^5	10	highly viscous	$VP:H_2O+Fe^{2+}$	4.62	
180(6-2)	"	"	"	"	$50:50+1\times10^{-3}$		
180(6-3)	"	"	"	"	"	5.90	
181(6-3)	"	"	"	"	"		
182(6-2)	"	"	"	"	"		
182(6-3)	"	"	"	"	"		
185(7-3)	"	"	"	"	"	7.83	
186(7-2)	"	"	"	"	"		
186(7-3)	"	"	"	"	"		
180(7-2)** ***	40	1.21×10^5	10	highly viscous	$VP:H_2O+Fe^{2+}$	11.68	
180(7-3)	"	"	"	"	$50:50+1\times10^{-3}$		
182(7-2)	"	"	"	"	"	15.05	
182(7-3)	"	"	"	"	"		
186(8-2)	"	"	"	"	"	13.11	
186(8-3)	"	"	"	"	"		
180(8-3)** ***	41	1.21×10^5	10	highly viscous	$VP:H_2O+Fe^{2+}$	23.57	
182(8-3)	"	"	"	"	$50:50+1\times10^{-3}$	39.80	
186(9-3)	"	"	"	"	"	32.98	

* The film was used for later stage grafting without measuring graft percent at this stage.

** Numbers in parentheses indicate grafting stage at later stage grafting.

*** Numbers after dash in parentheses indicate fragment piece number split from the original film.

**** Graft percent of AA, G(AA), is calculated by (W(AA+PE)-W(PE))/W(PE). Graft percent of VP, G(VP), is calculated by (W(AA+VP+PE)-W(AA+PE))/W(AA+PE).

Table 3.15 Summary of graft percents of VP onto PE films previously grafted with AA

AA					VP						
No.	G_1	G_2	G_3	G_4	§	G_5	G_6	G_7	G_8	G_9	note
179	2.58	*	*	16.51	2	1.10					
					3	*	4.62				
180	3.00	*	*	17.48	2	*	*	11.68			
					3	*	*	*	23.57		
181	*	9.15	14.34	20.19	2	1.49					
					3	*	5.90				
182	*	10.09	16.39	23.69	2	*	*	15.05			
					3	*	*	*	39.80		
183	*	10.05	*	22.54	2						
					3						
184	*	9.86	*	21.21	2						
					3						
185	*	9.70	*	*		29.12					
					2		2.07				
					3		*	7.83			
186	*	8.74	*	*		25.77					
					2		*	*	13.11		
					3		*	*	*	32.98	
AA						VP					

1 Gi indicates graft percent at ith grafting stage;
Graft percent of VP onto PE grafted with AA is defined as:
G2(VP)=(W(AA+VP+PE)−W(AA+PE))/(W(AA+PE)).

2 §: numbers indicate fragment piece number split from the original film.

3 *: The film was used for later atage grafting without measuring graft percent at this stage.

Table 3.16 First stage grafting of AA to PE film in 49°C water bath

(film: 100 μm HDPE 8×40mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
187	30	9.93×10^{4}	49	no	$AA:H_2O+Fe^{2+}$	13.11	
188	60	"	"	"	$50:50+4\times10^{-3}$	26.44	
189	120	"	"	"	"	42.24	
190	180	"	"	"	"	39.19	
191	180	"	"	"	"	44.77	
192	180	"	"	"	"	42.51	
193	180	"	"	"	"	44.59	
194	839	"	"	v.v. high	"	96.76	
195	839	"	"	v.v. high	"	103.20	
196	90	"	"	no	"	32.50	
197	120	"	"	"	"	37.17	
198	120	"	"	"	"	37.10	
199	120	"	"	"	"	39.32	
200	150	"	"	"	"	41.30	
201	240	"	"	"	"	48.46	
202	299.2	"	"	viscous	"	49.53	
203	360	"	"	highly viscous	"	55.33	
204	439	"	"	v. high	"	63.36	

① irradiated in a water bath.

Table 3.17 Second step grafting of AA to PE film previously grafted with AA in 49°C water bath

(film: 100 μm HDPE 8×40mm)

No.	G_1(AA) (%)	No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)
189	42.24	189(2)	30	9.93×10^4	49	no	$AA:H_2O+Fe^{2+}$	1.47
197	37.17	197(2)	60	"	"	"	$50:50+4\times10^{-3}$	6.17
198	37.10	198(2)	120.4	"	"	"	"	8.15
199	39.32	199(2)	180	"	"	"	"	8.62
193	44.59	193(2)	30	"	"	"	"	1.74
192	42.51	192(2)	60	"	"	"	"	3.88
191	44.77	191(2)	120.4	"	"	"	"	4.45
190	39.19	190(2)	180	"	"	"	"	9.59

1) irradiated in a water bath.

Table 3.18 Grafting of AA onto PE film in a water bath (T=10.8°C) to be compared with that carried out in air at the same temperature

(film: 100 μm HDPE 8×40mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
205	30	9.93×10^4	10.8	no	$AA:H_2O+Fe^{2+}$	0.25	
206	60	"	"	no	$50:50+4\times10^{-3}$	0.38	
207	120	"	"	a little	"	1.45	
208	180	"	"	some	"	2.73	
209	47	"	"	no	"	0.82	
210	15	"	"	no	"	0.32	
211	240	"	"	viscous	"	4.40	
212	300	"	"	highly viscous	"	5.90	
213	330	"	"	highly viscous	"	6.40	
214	242	"	"	viscous	"	4.13	
215	300.3	"	"	highly viscous	"	4.83	
216	360	"	"	very high	"	5.78	
217	360	"	"	very high	"	6.16	
218	360	"	"	very high	"	5.79	
219	360	"	"	very high	"	6.34	

Table 3.19 Preparation of AA grafted film to be used for conductivity measurements

(film: 27 μm LDPE 15×40mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
220	5	1.21×10^5	9	no	AA:H_2O+Fe^{2+}	1.52	
221	10.1	"	"	no	50:50+1×10^{-3}	12.32	
222	15	"	"	no	"	11.55	
223	20	"	"	a little	"	15.68	
224	25	"	"	some	"	36.27	
225	30.1	"	"	viscous	"	38.87	
226	40	"	"	highly viscous	"	48.99	
227	5	"	"	no	"	2.12	
228	10.1	"	"	no	"	10.99	
229	15	"	"	no	"		
230	20	"	"	a little	"	22.03	
231	25	"	"	some	"	38.47	
232	30.1	"	"	viscous	"	31.94	
233	40	"	"	highly viscous	"	39.43	

Table 20A Grafting of AA onto PE film in solution prepared using Mohr's solution (0.008mol/1) of different stock times

(film: 27 μm LDPE 15×40mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
234	0.5	1.187×10^5	50	no	$AA:H_2O+Fe^{2+}$	-	last sample
235	1	"	"	"	$50:50+4\times10^{-3}$	59.47	
236	2	"	"	"	"	67.29	
237	3	"	"	"	"	71.05	
238	3	"	"	"	"	74.93	
239	3	"	"	"	"	74.77	
240	4	"	"	a little	"	80.70	
241	5.5	"	"	some	"	83.22	
242	7	"	"	viscous	"	93.77	
243	8.5	"	"	very high	"	105.56	
244	0.5	"	"	no	"	55.01	
245	1	"	"	"	"	61.33	
246	2	"	"	"	"	65.83	
247	3	"	"	"	"	77.39	
248	3	"	"	"	"	81.79	
249	3	"	"	"	"	78.60	
250	4	"	"	a little	"	84.86	
251	5.5	"	"	some	"	89.29	
252	7.32	"	"	highly viscous	"	102.34	
253	8.5	"	"	very high	"	104.18	
273	0.5	"	"	no	"	67.31	
274	1	"	"	"	"	67.89	
275	2	"	"	"	"	69.55	
276	3	"	"	"	"	74.71	
277	3	"	"	"	"	75.70	
278	3	"	"	"	"	69.64	
279	4	"	"	a little	"	84.86	
280	5.5	"	"	some	"	81.85	
281	7.12	"	"	viscous	"	88.33	
282	9.57	"	"	very high	"	105.36	

1) No.234-243, Mohr's solution was used just after preparation within 1 hr.

2) No.244-253, Mohr's solution was used just after preparation within 1 day.

3) No.273-282, Mohr's solution was used just after preparation within 1 week.

4) irradiated in a water bath.

Table 3.20B Grafting of AA onto PE film in solution prepared using Mohr's salt solutions (0.008mol/l) of different stock times

(film: 100 μm HDPE 15×50mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
317	5	1.18×10^5	50	no	AA:H_2O+Fe^{2+}	2.55	1
318	10	"	"	"	50:50+4×10^{-3}	6.19	1
319	20	"	"	"	"	12.26	1
320	30	"	"	"	"	17.95	1
321	60	"	"	"	"	31.29	1
322	120	"	"	"	"	45.34	1
323	287.6	"	"	viscous	"	56.54	1
324	5	"	"	no	"	2.79	2
325	10	"	"	"	"	0.24	2
326	20	"	"	"	"	12.86	2
327	30	"	"	"	"	18.12	2
328	60	"	"	"	"	31.90	2
329	120	"	"	"	"	45.50	2
330	274.1	"	"	viscous	"	53.48	2
351	5	"	"	no	"	2.87	3
352	10	"	"	"	"	6.74	3
353	30	"	"	"	"	17.37	3
354	20	"	"	"	"	10.93	3
355	65	"	"	"	"	37.66	3
356	70	"	"	"	"	40.28	3
357	134	"	"	"	"	44.24	3
358	240	"	"	some	"	51.23	3
359	423	"	"	viscous	"	64.91	3
360	614	"	"	v.v. high	"	19.68	3

1) No.317-323, Mohr's solution was used just after preparation within 1 hr.

2) No.324-330, Mohr's solution was used just after preparation within 1 day.

3) No.351-360, Mohr's solution was used just after preparation within 1 week.

Table 3.21 Grafting of AA onto PE film using monomer solution stored 9 days before use

(film: 100 μm HDPE 8×40mm)

No.	Irrad. Time (h)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
254	0.5	1.187×10^5	50	no	$AA:H_2O+Fe^{2+}$	11.14	
255	1	"	"	no	$50:50+4\times10^{-3}$	2.86?	reason is not clear.
256	1.5	"	"	no	"	28.20	
257	2	"	"	no	"	40.30	
258	3	"	"	no	"	46.66	
259	4	"	"	a little	"	46.54	
260	5.5	"	"	some	"	51.86	
261	7	"	"	viscous	"	55.54	
262	8.5	"	"	highly viscous	"	64.43	

1) irradiated in a water bath.

Table 3.22 Preparation of AA grafted PE film of high graft percents to be used for conductivity measurements

No.		Irrad. Time (h)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
100μ	263	14.18	2.77×10^4	50	little	$AA:H_2O+Fe^{2+}$	48.25	
HDPE	264	17.27	"	"	little	$50:50+4\times10^{-3}$	49.29	
	265	19.33	"	"	some	"	51.11	
	266	22.02	"	"	viscous	"	53.87	
	267	26.31	"	"	highly viscous	"	56.79	
	268	26.31	"	"	highly viscous	"	56.91	
27μ	269	26.31	"	"	highly viscous	"	109.33	
LDPE	270	22.02	"	"	viscous	"	99.65	
13μ	271	26.31	"	"	highly viscous	"	59.06	
HDPE	272	22.02	"	"	viscous	"	62.76	

1) No.263-268:film:100 μm HDPE 15×50mm

2) No.269,270:film: 27 μm LDPE 15×50mm

3) No.271,272:film: 13 μm HDPE 15×50mm

4) irradiated in a water bath.

Table 3.23 Preparation of VP grafted PE film to be used for the second stage grafting of AA
(film: 13 μm HDPE 15×50mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
283	5	1.18×10^5	50	no	$VP:H_2O+Fe^{2+}$	0	
284	15	"	"	no	$50:50+4\times10^{-3}$	5.72	
285	30	"	"	a little	"	19.75	
286	55	"	"	v.v. high	"	60.51	
287	60	"	"	v.v. high	"	69.63	
288	60	"	"	v.v. high	"	-	
289	60	"	"	v.v. high	"	45.58	
290	60	"	"	v.v. high	"	-	
291	5	"	"	no	"	1.15	
292	15	"	"	no	"	6.10	
293	30	"	"	a little	"	19.88	
294	55	"	"	v.v. high	"	61.09	
295	60	"	"	v.v. high	"	-	
296	60	"	"	v.v. high	"	-	
297	60	"	"	v.v. high	"	59.62	
298	60	"	"	v.v. high	"	64.14	

1) No.288,290,295, and 296; The monomer solution turned too viscous to take out film.

2) irradiated in a water bath.

Table 3.24 Grafting of AA onto PE film previously grafted with VP (see Table 23)

No.	G_1(VP)	No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
283	0	283(2)	15	1.12×10^5	11	a little	AA:H_2O+Fe^{2+}	6.55	
284	5.72	284(2)	"	"	"	"	$50:50+1\times10^{-3}$	16.33	
285	19.75	285(2)	"	"	"	"	"	32.45	
286	60.51	286(2)	"	"	"	"	"	86.49	
287	69.63	287(2)	"	"	"	some	"	105.82	
289	45.58	289(2)	"	"	"	a little	"	60.05	
		299	"	"	"	"	"	5.19	
291	1.15	291(2)	30	"	"	viscous	"	15.69	
292	6.10	292(2)	"	"	"	"	"	42.95	
293	19.88	293(2)	"	"	"	highly viscous	"	81.75	
294	61.09	294(2)	"	"	"	"	"	120.36	
297	59.62	297(2)	"	"	"	viscous	"	124.39	
298	64.14	298(2)	"	"	"	"	"	124.48	
		300	"	"	"	"	"	12.94	

Table 3.25 Preparation of AA grafted film of different graft percents to be used for conductivity measurements

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
301	10	2.66×10^{4}	50	no	$AA+H_2O+Fe^{2+}$	0.68	①
302	30	"	"	"	$50:50+4\times10^{-3}$	1.43	①
303	60	"	"	"	"	18.58	①
304	134	"	"	"	"	35.47	①
305	244	"	"	"	"	17.82	①④
306	480	"	"	"	"	43.08	①
307	10	"	"	"	"	8.14	②
308	30	"	"	"	"	39.48	②
309	60	"	"	"	"	36.21	②
310	244	"	"	"	"	49.44	②
311	5	1.12×10^{5}	11	"	"	0.15	③
312	10	"	"	"	"	0.52	③
313	20	"	"	"	"	0.66	③
314	45	"	"	"	"	1.28	③
315	60	"	"	"	"	2.22	③
316	134	"	"	can't take out	$AA:H_2O+Fe^{2+}$ $50:50+1\times10^{-3}$		③

1 film: 100 μm HDPE, 15×50mm in 50°C water bath

2 film: 13 μm HDPE, 15×50mm in 50°C water bath

3 film: 400 μm HDPE, 15×50mm in air at 11°C

4 No.305 about 50% of total area was not grafted.

Table 3.26 Preparation of AA grafted films to be used for conductivity measurements at different dose rates

(film: 100 μm HDPE 15×50mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
331	66	2.54×10^3	50	no	AA:H_2O+Fe^{2+}	2.08	
332	66	2.12×10^3	"	"	50:50+4×10^{-3}	2.37	
333	118	2.07×10^3	"	"	"	3.01	
334	118	1.92×10^3	"	"	"	-0.04	*
335	171	1.61×10^3	"	"	"	6.64	*
336	171	1.88×10^3	"	"	"	20.14	
337	912	1.52×10^3	"	"	"	34.43	
338	912	1.63×10^3	"	"	"	26.23	*
339	1188	1.50×10^3	"	"	"	32.08	
340	1188	1.35×10^3	"	"	"	29.18	*
341	40	1.18×10^5	"	"	"	22.98	
342	50	"	"	"	"	28.18	
343	60	"	"	"	"	33.01	
344	75	"	"	"	"	40.23	
345	90	"	"	"	"	41.65	
346	90	2.11×10^3	"	"	"	-0.09	
347	110	2.11×10^3	"	"	"	0.38	
348	130	2.15×10^3	"	"	"	2.02	
349	150	2.11×10^3	"	"	"	1.16	
350	180	2.11×10^3	"	"	"	4.44	

1) In films No.335, 338 and 340, a small non-grafted area (0.5-1.0 square cm) recognized visually due to possible contamination of the film surface. For No.334, the reason of this small graft percent is not known.

2) irradiated in a water bath.

Table 3.27 Preparation of AA grafted films to be used for conductivity measurements
(film: 100 μm HDPE 15×50mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
361	240	2.48×10^3	50	no	$AA:H_2O+Fe^{2+}$	27.00	*
362	300	2.11×10^3	"	"	$50:50+4\times10^{-3}$	13.20	*
363	360	2.11×10^3	"	"	"	20.11	*
364	423	2.15×10^3	"	"	"	4.63	*
365	482	2.11×10^3	"	"	"	-0.04	*
366	556	2.11×10^3	"	"	"	28.71	*
367	614	2.48×10^3	"	"	"	87.24	*

1) No.361-164, and 367 were not uniformly grafted due to possible contamination of the film surface before use.
2) irradiated in a water bath.

Table 3.28A Preparation of VP grafted PE film to be used for the second stage grafting using AA and for conductivity measurements

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
368	203	2.62×10^4	50	v.v. high	$VP:H_2O+Fe^{2+}$	24.14	
369	"	"	"	"	$50:50+4\times10^{-3}$	25.55	
370	"	"	"	"	"	25.26	
371	"	"	"	"	"	26.92	
372	"	"	"	"	"	27.34	
373	"	"	"	"	"	32.97	
374	"	"	"	"	"	28.05	
375	"	"	"	"	"	23.91	
376	"	"	"	"	"	22.31	
377	"	"	"	"	"	25.80	
378	"	"	"	"	"	27.43	
379	396	2.11×10^3	"	no	"	1.07	
380	"	2.11×10^3	"	no	"	0.95	
381	"	2.15×10^3	"	no	"	0.77	
382	"	2.11×10^3	"	no	"	0.29	
383	203	2.62×10^4	"	v.v. high	"	25.18	*
384	396	2.62×10^4	"	v.v. high	"	39.70	*
385	"	"	"	"	"	45.98	*
386	"	"	"	"	"	31.26	*
387	"	"	"	"	"	39.60	*

1) At this stage, grafting was carried out to obtain the grafted film whose cross section is uniform as possible.
2) No.383-387, the monomer solution became highly viscous, and films were taken out after immersing the film in cold water for two days.
3) irradiated in a water bath.

Table 3.28B Grafting of AA onto PE film previously grafted with VP, grafted film to be used for conductivity measurements

(film: 100 μm HDPE 15×70mm)

No.	G_1(VP) (%)	No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
368	24.14	368-(2)	10	1.17×10^5	50	no	AA:H_2O+Fe^{2+}	26.68	
372	27.34	372-(2)	20	"	"	"	50:50+4×10^{-3}	49.95	
373	32.97	373-(2)	40	"	"	"	"	74.02	
376	22.31	376 (2)	60	"	"	"	"	119.12	
379	1.07	379-(2)	15	"	"	"	"	10.20	
380	0.95	380-(2)	30	"	"	"	"	19.38	
381	0.77	381-(2)	60	"	"	"	"	34.57	
382	0.29	382-(2)	120	"	"	"	"	45.55	
371	26.92	371-(2)	4	"	"	"	"	15.86	
374	28.05	374-(2)	6	"	"	"	"	23.14	
377	25.80	377-(2)	8	"	"	"	"	27.93	
369	25.55	369-(2)	8	2.62×10^4	"	"	"	9.36	
370	25.26	370-(2)	15	"	"	"	"	15.96	
375	23.91	375-(2)	25	"	"	"	"	26.86	
378	27.43	378-(2)	40	"	"	"	"	30.82	

1) No.372(2), 373(2), and 376(2) became brittle and was easily broken when dried.

2) irradiated in a water bath.

Table 3.28C Preparation of VP grafted film to be used for second stage AA grafting and grafting of AA onto PE film previously grafted with VP, grafted film to be used for conductivity measurements

(film: 100 μm HDPE 15×70mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
404	20	1.17×10^5	50	no	$VP:H_2O+Fe^{2+}$	0.85	
405	"	"	"	"	$50:50+4\times10^{-3}$	0.93	
406	"	"	"	"	"	0.96	
407	"	"	"	"	"	1.03	
408	"	"	"	"	"	0.73	
409	"	"	"	"	"	0.86	

No.	G_1(VP)	No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
404	0.85	404(2-1)	5	1.17×10^5	50	no	$AA:H_2O+Fe^{2+}$	3.55	
405	0.93	405(2-1)	10	"	"	"	$50:50+4\times10^{-3}$	6.96	
406	0.96	406(2-1)	15	"	"	"	"	11.64	
409	0.86	409(2-1)	20	"	"	"	"	14.06	

1) irradiated in a water bath.

Table 3.29A Preparation of PE film co-grafted in the monomer solutions containing different ratios of AA and VP, the grafted films of different graft percents to be used for conductivity measurements

(film: 100 μm HDPE 15×70mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
388	20.4	1.17×10^5	50	no	$AA:VP:H_2O+Fe^{2+}$	3.36	
389	40	"	"	"	$20:30:50+4\times10^{-3}$	7.26	
390	60	"	"	"	"	9.08	
391	91	"	"	"	"	12.38	
392	20.4	"	"	"	$AA:VP:H_2O+Fe^{2+}$	3.55	
393	40	"	"	"	$30:20:50+4\times10^{-3}$	9.61	
394	60	"	"	"	"	14.13	
395	91	"	"	"	"	21.43	
396	10	"	"	"	$AA:VP:H_2O+Fe^{2+}$	2.58	
397	19.6	"	"	"	$40:10:50+4\times10^{-3}$	7.02	
398	29.6	"	"	"	"	10.21	
399	70.6	"	"	"	"	26.65	
400	10	"	"	"	$AA:VP:H_2O+Fe^{2+}$	3.13	
401	19.6	"	"	"	$45:5:50+4\times10^{-3}$	8.73	
402	29.6	"	"	"	"	13.45	
403	70.6	"	"	"	"	32.35	

1) irradiated in a water bath.

Table 3.29B Preparation of PE film co-grafted in the monomer solutions containing different ratios of AA and VP, the grafted films of different graft percents to be used for conductivity measurements (continued)

(film: 100 μm HDPE 15×70mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
410	6	1.17×10^5	50	no	$AA:VP:H_2O+Fe^{2+}$	1.32	
411	100	"	"	no	$45:5:50+4\times10^{-3}$	38.74	
412	150	"	"	a little	"	42.97	
413	100	"	"	no	$AA:VP:H_2O+Fe^{2+}$	37.15	
414	150	"	"	a little	$40:10:50+4\times10^{-3}$	38.57	
415	10	"	"	no	$AA:VP:H_2O+Fe^{2+}$	1.25	
416	143	"	"	no	$30:20:50+4\times10^{-3}$	35.38	
417	213	"	"	some	"	37.83	
418	10	"	"	no	$AA:VP:H_2O+Fe^{2+}$	0.80	
419	120	"	"	no	$20:30:50+4\times10^{-3}$	19.10	
420	163	"	"	no	"	23.66	
421	240	"	"	a little	"	33.34	

1) irradiated in a water bath.

Table 3.30A Grafting of AA onto PE film previously grafted with VP, grafted film to be used for conductivity measurements

(film: 100 μm HDPE 15×65mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
422	40	1.17×10^5	50	v. high	$VP:H_2O+Fe^{2+}$	5.49	
423	"	"	"	"	$50:50+4\times10^{-3}$	5.22	
424	"	"	"	"	"	5.17	
425	"	"	"	"	"	3.32	
426	"	"	"	"	"	4.68	
427	"	"	"	"	"	5.65	
428	"	"	"	"	"	5.09	
429	"	"	"	"	"	5.21	

No.	G_1(VP)	No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
422	5.49	422(2-2)	3	1.17×10^5	50	no	$AA:H_2O+Fe^{2+}$	5.15	
423	5.22	423(2-2)	5	"	"	"	$50:50+4\times10^{-3}$	7.47	
424	5.17	424(2-2)	8	"	"	"	"	13.01	
422	5.49	422(2-1)	12	"	"	"	"	16.47	
423	5.22	423(2-1)	17	"	"	"	"	29.65	
424	5.17	424(2-1)	22	"	"	"	"	37.18	
425	5.32	425(2-1)	100	"	"	"	"	90.49	
426	4.68	426(2-1)	40	"	"	"	"	83.90	
427	5.65	427(2-1)	60	"	"	"	"	94.84	
428	5.09	428(2-1)	80	"	"	"	"	91.71	
429	5.21	429(2-1)	30	"	"	"	"	65.17	
425	5.32	425(2-2)	20	"	"	"	"	29.05	
426	4.68	426(2-2)	25	"	"	"	"	44.21	
427	5.65	427(2-2)	28	"	"	"	"	56.31	
428	5.09	428(2-2)	32.1	"	"	"	"	67.62	

1) irradiated in a water bath.

Table 3.30B Grafting of VP onto PE film previously grafted with AA, grafted film to be used for conductivity measurements

(film: 100 μm HDPE 15×65mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
430	8	1.17×10^5	50	no	$AA:H_2O+Fe^{2+}$	5.54	
431	"	"	"	"	$50:50+4\times10^{-3}$	3.00	
432	"	"	"	"	"	5.38	
433	"	"	"	"	"	4.72	
434	"	"	"	"	"	5.31	
435	"	"	"	"	"	5.19	
436	"	"	"	"	"	5.68	

No.	G_1(AA)	No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
430	5.54	430(2-1)	3.1	1.17×10^5	50	no	$VP:H_2O+Fe^{2+}$	0.24	
430	5.54	430(2-2)	5	"	"	"	$50:50+4\times10^{-3}$	0.29	
432	5.38	432(2-1)	8.1	"	"	"	"	0.62	
432	5.38	432(2-2)	12	"	"	"	"	0.98	
433	4.72	433(2-1)	17	"	"	"	"	1.94	
433	4.72	433(2-2)	25	"	"	a little	"	3.08	
434	5.31	434(2-1)	35	"	"	viscous	"	6.81	
434	5.31	434(2-2)	45	"	"	v. high	"	9.82	
435	5.68	435(2-1)	55	"	"	v.v. high	"	18.82	

1) irradiated in a water bath.

Table 30C Preparation of VP grafted PE film to be used for the second stage grafting using AA and for conductivity measurements

(film: 100 μm HDPE 15×65mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
437	55	1.15×10^5	50	v.v. high	$VP:H_2O+Fe^{2+}$	11.89	
438	"	"	"	"	$50:50+4\times10^{-3}$	9.34	
439	"	"	"	"	"	11.83	
440	"	"	"	"	"	11.60	
441	"	"	"	"	"	13.60	
442	"	"	"	"	"	14.31	
443	"	"	"	"	"	12.37	
444	"	"	"	"	"	14.00	
445	27.3	"	"	a little	"	2.26	
446	"	"	"	"	"	1.91	
447	"	"	"	"	"	2.31	
448	"	"	"	"	"	2.01	
449	"	"	"	"	"	1.63	
450	"	"	"	"	"	1.51	
451	"	"	"	"	"	2.24	
452	26.7	"	"	"	"	2.18	

1) irradiated in a water bath.

Table 3.30D Grafting of AA onto PE film previously grafted with VP, grafted film to be used for conductivity measurements

(film: 100 μm HDPE 15×60mm)

No.	G_1(VP)	No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
437	11.89	437(2-1)	3.1	1.15×10^5	50	no	$AA:H_2O+Fe^{2+}$	7.65	
437	11.89	437(2-2)	5	"	"	"	$50:50+4\times10^{-3}$	9.91	
439	11.83	439(2-1)	6.9	"	"	"	"	15.20	
439	11.83	439(2-2)	10	"	"	"	"	20.89	
440	11.60	440(2-1)	15	"	"	"	"	29.92	
440	11.60	440(2-2)	20	"	"	"	"	38.01	
441	13.60	441(2-1)	25	"	"	"	"	66.16	
441	13.60	441(2-2)	30	"	"	"	"	80.86	
443	12.37	443(2-1)	40	"	"	"	"	96.81	
443	12.37	443(2-2)	50	"	"	"	"	101.87	
444	14.00	444-2	70	"	"	"	"	108.17	
445	2.26	445(2-1)	3.1	"	"	"	"	4.82	
445	2.26	445(2-2)	6.9	"	"	"	"	12.68	
446	1.91	446(2-1)	10	"	"	"	"	18.87	
446	1.92	446(2-2)	15	"	"	"	"	18.43	
447	2.31	447(2-1)	20	"	"	"	"	26.57	
447	2.31	447(2-2)	25	"	"	"	"	30.57	
448	2.01	448(2-1)	30	"	"	"	"	34.11	
448	2.01	448(2-2)	40	"	"	"	"	39.24	
451	2.24	451(2-1)	60	"	"	"	"	47.05	
451	2.24	451(2-2)	80	"	"	"	"	49.35	
452	2.18	452(2-1)	100	"	"	"	"	48.39	
452	2.18	452(2-2)	116	"	"	"	"	51.43	

1) irradiated in a water bath.

Table 3.31A Grafting of VP onto PE film previously grafted with AA, grafted film to be used for conductivity measurements

(film: 100 μm HDPE 15×65mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
453	8	1.15×10^5	50	no	$AA:H_2O+Fe^{2+}$	1.35	
454	"	"	"	"	$50:50+4\times10^{-3}$	2.67	
455	"	"	"	"	"	3.88	
456	"	"	"	"	"	4.28	
457	"	"	"	"	"	4.45	
458	"	"	"	"	"	3.95	
459	"	"	"	"	"	3.88	
460	"	"	"	"	"	3.75	
431(2-1)	45	1.15×10^5	50	v.v. high	$VP:H_2O+Fe^{2+}$	8.71	①
431(2-2)	"	"	"	"	$50:50+4\times10^{-3}$	7.94	①
456(2-1)	"	"	"	"	"	13.44	①
456(2-2)	"	"	"	"	"	12.68	①
457(2-1)	"	"	"	"	"	11.49	①
457(2-2)	"	"	"	"	"	12.55	①
458(2-1)	"	"	"	"	"	11.89	①
458(2-2)	"	"	"	"	"	12.46	①
431(3-1)	45	1.15×10^5	50	v. high	$VP+H_2O+Fe^{2+}$	27.73	①
456(3-1)	"	"	"	"	$50:50+4\times10^{-3}$	*	②
456(3-2)	"	"	"	"	"	43.88	①
457(3-2)	"	"	"	"	"	*	②
458(3-1)	"	"	"	"	"	*	②
458(3-2)	"	"	"	"	"	*	②

① Graft percent of VP, G(VP), is calculated by
{(wt of VP in AA grafted PE)-(wt of AA grafted PE)}/
(wt of AA grafted PE)

② * The film was used for later stage grafting without measuring graft percent at this stage.

3) irradiated in a water bath.

Table 3.31B Grafting of VP onto PE film previously grafted with AA, grafted film to be used for conductivity measurements

(film: 100 μm HDPE 15×65mm)

No.	Irrad. Time (min.)	Dose Rate (rad/h)	T (°C)	Viscosity	Solution	G (%)	Note
456(4-1)	50	1.15×10^5	50	highly viscous	$VP:H_2O+Fe^{2+}$	121.91	①
457(4-2)	20	"	"	no	$50:50+4\times10^{-3}$	53.68	①
458(4-1)	50	"	"	highly viscous	"	119.87	①
458(4-2)	20	"	"	no	"	57.62	①

① Graft percent of VP, G(VP), is calculated by
{(wt of VP in AA grafted PE)-(wt of AA grafted PE)}/
(wt of AA grafted PE)

2) irradiated in a water bath.

Table 3.32 Summary of grafting of VP onto PE film previously grafted with AA, grafted film to be used for conductivity measurements

No.	G_1(AA)	No.	G_2(VP)	No.	G_3(VP)	No.	G_4(VP)	Note
453	1.35							
454	2.67							
455	3.88							
431	3.00	431(2-2)	7.94					
457	4.45	457(2-1)	11.49					
431	3.00	431(2-1)	8.71	431(3-1)	27.73			
456	4.28	456(2-2)	12.68	456(3-2)	43.88			
456	4.28	456(2-1)	13.44	456(3-2)	*	456(4-1)	121.91	
457	4.45	457(2-2)	12.55	457(3-2)	*	457(4-2)	53.68	
458	3.95	458(2-1)	11.89	457(3-1)	*	458(4-1)	119.87	
458	3.95	458(2-2)	12.46	458(3-2)	*	458(4-2)	57.62	

① Graft percent of VP, G(VP), is calculated by
{(wt of VP in AA grafted PE)-(wt of AA grafted PE)}/
(wt of AA grafted PE)

② * The film was used for later stage grafting without measuring graft percent at this stage.

Table 3.33 Moisture gain

t (min)	$w_1(t)$ (g)	G (%)
0	0.09604	0
1.42	0.09606	0.029
5	0.09609	0.074
16.25	0.09614	0.147
21.08	0.09616	0.177
24.08	0.09617	0.192
35.83	0.09620	0.236
41.92	0.09622	0.265
51	0.09625	0.310
62.58	0.09626	0.324
87	0.09629	0.369
106	0.09632	0.413

1) No.345, 100μ HDPE, G(AA)=41.65%, w_0=0.06780g, T=19.9°C, humility=50%

2) $$G = \frac{w_1(t) - w_1(t=0)}{w_0} \times 100$$

3) after vacuum oven dry at 50°C, the film was put into a desiccator immediately. Then waited 3 min. for cooling down before weighing.

Table 3.34 Optical density and graft perecent of AA-grafted film

No.	62	64	65	119	120	121	122	144
G(AA) (%)	23.2	45.9	48.3	2.3	9.6	18.8	33.2	3.02
A	0.9695	1.839	1.627	0.1449	0.3826	0.6425	1.3006	0.178
B	0.3069	0.2972	0.2526	0.2808	0.3056	0.3043	0.2941	0.3212
A'	1.0516	2.060	2.144	0.1718	0.4168	0.7029	1.472	0.1845

No.	145	146	147	148	150	151	152	
G(AA) (%)	6.09	8.29	18.54	33.9	5.26	7.07	6.19	
A	0.301	0.381	0.828	1.343	0.280	0.337	0.293	
B	0.3308	0.3136	0.2956	0.2833	0.2856	0.2972	0.3308	
A'	0.3029	0.4044	0.9325	1.578	0.3264	0.3775	0.2949	

No.	153	154	156	157	158	159	160	
G(AA) (%)	24.52	36.94	5.9	6.51	9.12	35.02	35.02	
A	0.922	1.534	0.286	0.345	0.370	1.215	1.322	
B	0.2871	0.2562	0.3256	0.3107	0.2872	0.2438	0.2615	
A'	1.069	1.993	0.2924	0.3697	0.4289	1.659	1.683	

Table 3.35 Optical density and graft perecent of VP-grafted film

No.	57	58	140	141	142
G(VP) (%)	2.0	25.1	1.4	5.1	9.5
A	0.0615	0.9035	0.1291	0.2825	0.5267
B	0.3026	0.2529	0.3179	0.2987	0.3211
A'	0.0676	1.189	0.1352	0.3148	0.5461

Table 3.36 Optical density and graft percent of VP-AA-grafted film

No.	70	71	87	89	90	91	92	93	94
G_1(VP) (%)	2.0	25.1	0.8	21.2	20.9	4.6	8.7	21.2	2.9
G_2(VP) (%)	46.1	83.2	7.5	84.6	78.6	52.1	54.5	34.8	17.3
G (%)	49.1	129.2	8.4	123.7	115.9	59.1	67.9	63.4	20.7
A	1.9428	4.953*	0.3672	4.886*	4.952*	2.0027	3.354	2.196	0.766
B	0.2765	0.1584	0.3199	0.2298	0.2173	0.2087	0.2442	0.2446	0.2888
A'	2.339	10.409	0.3821	7.078	7.586	3.195	4.572	2.989	0.8830

Table 3.37 Optical density and graft percent of AA-VP-grafted film

No.	72	75	109	114	161	162	163	164
G_1(AA) (%)	23.2	48.3	17.7	5.8	1.72	3.02	6.09	8.29
G_2(VP) (%)	20.0	1.4	4.9	13.9	6.86	9.23	10.3	12.9
G (%)	47.8	50.4	23.5	20.5	8.70	12.5	17.0	22.3
A	1.863	1.479	0.738	0.856	0.369	0.548	0.586	0.745
B	0.2701	0.2373	0.3179	0.3325	0.2811	0.3062	0.3121	0.2912
A'	2.296	2.075	0.7728	0.8570	0.4370	0.5958	0.6251	0.8517

No.	165	166	168	169	170	171	172	173
G_1(AA) (%)	18.54	33.9	5.26	7.07	6.19	24.52	36.94	5.9
G_2(VP) (%)	14.5	4.92	3.33	4.26	3.76	5.01	1.94	-0.09
G (%)	35.7	40.5	8.77	11.6	10.0	30.75	39.6	5.8
A	1.321	1.123	0.266	0.301	0.276	0.769	1.429	0.285
B	0.2718	0.2747	0.2761	0.2842	0.3308	0.2659	0.2549	0.3311
A'	1.618	1.361	0.3207	0.3526	0.2778	0.9628	1.866	0.2865

No.	175	176	177	178
G_1(AA) (%)	6.51	9.12	35.02	35.02
G_2(VP) (%)	0.79	0.92	1.4	1.31
G (%)	7.35	10.1	36.9	36.8
A	0.320	0.327	1.159	1.157
B	0.3136	0.2869	0.2208	0.2554
A'	0.3397	0.3794	1.747	1.508

Table 3.38 Optical density and graft percent of co-grafted film with AA and VP

No.	69	123	125	126	134	136	137	138
G (%)	11.5	2.7	12.6	14.2	12.3	3.3	6.1	10.3
A	0.4906	0.1334	0.563	0.6176	0.558	0.2055	0.2888	0.548
B	0.2944	0.3148	0.3127	0.2794	0.3015	0.3325	0.2965	0.3239
A'	0.5548	0.1411	0.5994	0.7359	0.6161	0.2057	0.3243	0.5632

Table 3.39 Change of film area before and after grafting
13 μ HDPE, DR: 2.57×10^5rad/h, T: 14°C

No.	G (%)	original area (mm^2)	area after irradiation (mm^2)	area increase (%)	$\frac{\Delta S}{G}\times 100$ (%)
29	61.8	1590	2019.3	27.0	43.69
31	58.4	1560	1955.3	25.3	43.32
33	57.9	1540.3	1920.8	24.5	42.31

Table 3.40 Electric resistance of AA-grafted film
13 μm HDPE;
monomer solution, AA:water, Mohr's salt=50:50,
0.001 mol/1 electrolyte, 10% KOH aqueous solution

No.	Film	Solution	Dose Rate (rad/h)	Irrad. Time (min.)	G (%)	R (Ω)	Swelling Time(hr)	R_0 (Ω)	T (°C)
299	13μ HDPE	AA:H_2O+Fe^{2+}	1.12×10^5	15	5.19	1.7×10^4	0.25	~20	~25
300	"	50:50+1×10^{-3}mol/1	"	30	12.94	2.91×10^3	0.083	~20	"
307	"	"	2.66×10^4	10	8.14	over range	0.383	~20	"
308	"	"	"	30	39.48	22.2	0.055	~20	"
309	"	"	"	60	36.21	21.2	0.0383	~20	"
310	"	"	"	244	49.44	21.3	0.0667	~20	"
336	100μ HDPE	"	1.88×10^3	171	20.14	280	5.75	~20	"
338	"	"	1.63×10^3	912	26.23	26	1.45	~20	"
340	"	"	1.35×10^3	1188	29.18	26	0.433	~20	"
339	"	"	1.50×10^3	1188	32.08	26	0.40	~20	"

Table 3.41 Electric resistance of AA-grafted film
100 μm HDPE;
monomer solution, AA:water, Mohr's salt=50:50,
0.004 mol/l electrolyte, 10% KOH aqueous solution

No.	Film	Solution	Dose Rate (rad/h)	Irrad. Time (min.)	G (%)	R (Ω)	Swelling time(hr)	R (Ω)	T (°C)
435b	100μ HDPE	AA:H_2O+Fe^{2+}	1.17×10^5	8	5.19	1.035×10^4	0.53	~20	~25
327 ①	"	50:50+4×10^{-3}mol/l	"	30	18.12	2.245×10^3	2.32	~3.5	~24
436a②	"	"	"	8	5.68	8.274×10^3	0.4	~3.5	~24
326a	"	"	"	20	12.86	3.26×10^3	15.37	~20	~25
314	"	"	1.12×10^5	45	1.28	over range	4.72	~20	~25
315	"	"	"	60	2.22	5.28×10^5	4	~20	~25
341	"	"	1.18×10^5	40	22.98	2.6×10^3	19.95	~20	14
342	"	"	"	50	28.18	1.82×10^3	19.57	~20	19
343③	"	"	"	60	33.01	2.51×10^5	21.72	~20	19
344	"	"	"	75	40.23	24	0.28	~20	19
345	"	"	"	90	41.65	25	0.32	~20	19
328④	"	"	"	60	31.90	5.52×10^4	7.52	~20	~25
329	"	"	"	120	45.55	22	1.4	~20	~25
355	"	"	"	65	37.66	27	0.32	~20	17.5
356	"	"	"	70	40.28	21	0.35	~20	18.5
354	"	"	"	20	10.93	over range	15.9	~33	15
353	"	"	"	30	17.37	4.3×10^5	16.9	~33	15
302	"	"	2.66×10^4	30	1.43	over range	12.55	~20	~25
303	"	"	"	60	18.58	"	1.77	~20	~25
304	"	"	"	104	35.47	24.2	0.32	~20	~25
306	"	"	"	480	43.08	27	0.62	~20	~25
263	"	"	2.77×10^4	14.2	48.25	19	1.8	~20	~25
264	"	"	"	17.3	49.29	20	0.27	~20	~25
265	"	"	"	19.3	51.11	21	0.67	~20	~25
266	"	"	"	22	53.87	21	0.22	~20	~25
267	"	"	"	26.3	56.79	21	0.17	~20	~25
268	"	"	"	26.3	56.91	14	0.18	~12	~25

Table 3.42 Electric resistance of VP-grafted film
100 μm HDPE; measurement temperature, 24.1±1.1°C
monomer solution, VP:water,
Mohr's salt=50:50, 0.004 mol/l
cell resistance, 3.5Ω.
electrolyte, 10% KOH aqueous solution

No.	VP	Dose Rate (rad/h)	Irrad. Time (min.)	G (%)	R (Ω)	Swelling time(hr)
376	"	2.62×10^4	203	22.31	over range	23.33
383	"	"	203	25.18	9.69×10^5	8.48
386	"	"	396	31.26	8.96×10^3	3.8
387	"	"	396	39.60	600	3.13
372	"	"	203	27.34	4.39×10^5	14.08
385	"	"	396	45.98	349	12.23
4046	"	1.17×10^5	20	0.85	over range	24.33

Table 3.43 Electric resistance of AA-VP-multi-stage grafted film
100 μm HDPE; measurement temperature, 24.1±1.1°C
monomer solution, VP:water, Mohr's salt=50:50, 0.004 mol/l
(for second stage grafting)
cell resistance, 3.5 Ω.
electrolyte, 10% KOH aqueous solution

No.	Dose Rate (rad/h)	Irrad. Time (min.)	G_1(AA) (%)	R (Ω)	No.	Dose Rate (rad/h)	Irrad. Time (min.)	G_2(VP) (%)	R (Ω)	Swelling Time (hr.)
435	1.17×10^5	8	5.19	1.035×10^4	435(2-1)	1.17×10^5	55	18.82	8.563×10^3	10.67
434	"	"	5.31		434(2-2)	"	45	9.82	1.128×10^4	21.93
434	"	"	5.31		434(2-1)	"	35	6.81	1.11×10^4	23.4
433	"	"	4.72		433(2-2)	"	25	3.08	1.262×10^4	24.28
433	"	"	4.72		433(2-1)	"	17	1.94	1.20×10^4	24.72
432	"	"	5.38		432(2-2)	"	12	0.98	over range	24.98
432	"	"	5.38		432(2-1)	"	8.1	0.62	8.96×10^3	25.37
430	"	"	5.54		430(2-2)	"	5	0.29	over range	25.63
430	"	"	5.54		430(2-1)	"	3.1	0.24	7.7×10^3	26.93
456	1.15×10^5	"	4.28		456(4-1)	1.15×10^5	50	121.91	30	40.07
458	"	"	3.95		458(4-1)	"	50	119.87	32	40.38
458	"	"	3.95		458(4-3)	"	20	57.62	1.792×10^2	40.62
457	"	"	4.45		457(4-2)	"	20	53.68	1.966×10^2	40.87
456	"	"	4.28		456(3-2)	"	45	43.88	5.624×10^2	41.08
431	1.17×10^5	"	3.0		431(3-1)	"	45	27.73	1.657×10^4	41.67
457	1.15×10^5	"	4.45		457(2-1)	"	45	11.49	1.69×10^4	41.98
431	1.17×10^5	"	3.0		431(2-2)	"	45	7.94	5.08×10^4	45

Table 3.44 Electric resistance of VP-AA-multi-stage grafted film
100 μm HDPE; measurement temperature, 24.1±1.1°C
monomer solution, AA:water, Mohr's salt=50:50, 0.004 mol/l
(for second stage grafting)
cell resistance, 3.5 Ω.
electrolyte, 10% KOH aqueous solution

No.	Dose Rate (rad/h)	Irrad. Time (min.)	G_1(VP) (%)	R (Ω)	No.	Dose Rate (rad/h)	Irrad. Time (min.)	G_2(AA) (%)	R (Ω)	Swelling Time (hr.)
382	2.11×10	396	0.29		382-2	1.17×10^5	120	45.55	5.7	0.28
381	2.15×10	"	0.77		381-1	"	60	34.57	9.53×10^2	2.75
379	2.11×10	"	1.07		379-2	"	15	10.20	3.31×10^3	8
380	2.11×10	"	0.95		380-2	"	30	19.38	1.97×10^3	56
409	1.17×10	20	0.86		409(2-1)	"	20	14.06	2.022×10^3	7.75
406	"	"	0.96		406(2-1)	"	15	11.64	2.33×10^3	8.17
405	"	"	0.93		405(2-1)	"	10	6.96	4.16×10^3	8.45
404	"	"	0.85	over range	404(2-1)	"	5	3.55	1.361×10^4	22.58
		"								
428	1.17×10	40	5.09		428(2-1)	"	80	91.71	~6	16.27
427	"	"	5.65		427(2-1)	"	60	94.84	~5	16.9
426	"	"	4.68		426(2-1)	"	40	83.90	~5	23.33
429	"	"	5.21		429(2-1)	"	30	65.17	336	24.47
425	"	"	5.32		425(2-1)	"	100	90.49	~5	24.7
424	"	"	5.17		424(2-1)	"	22	37.18	544	24.95
423	"	"	5.22		423(2-1)	"	17	29.65	698	25.17
424	"	"	5.17		424(2-2)	"	8	13.01	2.15×10^3	25.87
423	"	"	5.22		423(2-2)	"	5	7.47	5.61×10^3	26.43
422	"	"	5.49		422(2-2)	"	3	5.15	1.50×10^4	26.78
422	"	"	5.49		422(2-1)	"	12	16.47	1.473×10^3	25.57
428	"	"	5.09		428(2-2)	"	32.1	67.62	3.19×10^2	14.35
427	"	"	5.65		427(2-2)	"	28	56.31	340	14.12
426	"	"	4.68		426(2-2)	"	25	44.21	443.2	14.63
425	"	"	5.32		425(2-2)	"	20	29.05	780	14.95

Table 3.45 Electric resistance of VP-AA-multi-stage grafted film (continued)
100 μm HDPE; measurement temperature, 24.1±1.1°C
monomer solution, AA:water, Mohr's salt=50:50, 0.004 mol/l (for second stage grafting)
cell resistance, 3.5 Ω.
electrolyte, 10% KOH aqueous solution

No.	Dose Rate (rad/h)	Irrad. Time (min.)	G_1(VP) (%)	R (Ω)	No.	Dose Rate (rad/h)	Irrad. Time (min.)	G_2(AA) (%)	R (Ω)	Swelling Time (hr.)
452	1.15×10^5	26.7	2.18		452(2-2)	1.15×10^5	116	51.43	~5	20.72
452	"	26.7	2.18		452(2-1)	"	100	48.39	~6	20.95
451	"	27.3	2.24		451(2-2)	"	80	49.35	~7	21.73
451	"	"	2.24		451(2-1)	"	60	47.05	~6	22.07
448	"	"	2.01		448(2-2)	"	40	39.24	5.08×10^2	22.8
448	"	"	2.01		448(2-1)	"	30	34.11	1.267×10^3	23.02
447	"	"	2.31		447(2-2)	"	25	30.57	9.79×10^2	23.43
447	"	"	2.31		447(2-1)	"	20	26.57	1.061×10^3	25.65
446	"	"	1.91		446(2-2)	"	15	18.43	2.31×10^3	25.92
446	"	"	1.91		446(2-1)	"	10	18.83	2.10×10^3	26.25
445	"	"	2.26		445(2-2)	"	6.9	12.68	1.95×10^3	26.5
445	"	"	2.26		445(2-1)	"	3.1	4.82	9.74×10^3	62.

Table 3.46 Electric resistance of VP-AA-multi-stage grafted film (continued)
100 μm HDPE; measurement temperature, 24.1±1.1°C
monomer solution, AA:water, Mohr's salt=50:50, 0.004 mol/l (for second stage grafting)
cell resistance, 3.5 Ω.
electrolyte, 10% KOH aqueous solution

No.	Dose Rate (rad/h)	Irrad. Time (min.)	G_1(VP) (%)	R (Ω)	No.	Dose Rate (rad/h)	Irrad. Time (min.)	G_2(AA) (%)	R (Ω)	Swelling Time (hr.)
372	2.62×10^4	203	27.34	4.39×10^5	372-2	1.17×10^5	20	49.95	10	0.98
373	"	"	32.97		373-2	"	40	74.02	~5	1.03
376	"	"	22.31	over range	376-2	"	60	119.12	6	0.98
368	"	"	24.14		368-2	"	10	26.68	10	2.72
377	"	"	25.80		377-2	"	8	27.93	8.9	12.62
374	"	"	28.05		374-2	"	6	23.14	9.9	13.1
371	"	"	26.92		371-2	"	4	15.86	17	13.48
378	"	"	27.43		378-2	2.62×10^4	40	30.82	8.5	14.17
375	"	"	23.91		375-2	"	25	26.86	8.6	14.6
370	"	"	25.26		370-2	"	15	15.96	10.8	15.08
369	"	"	25.55		369-2	"	8	9.36	27	15.28
444	1.15×10^5	55	14.00		444-2	1.15×10^5	70	108.17	~5	14.5
443	"	"	12.37		443(2-2)	"	50	101.87	~6	14.88
443	"	"	12.37		443(2-1)	"	40	96.81	~22	15.32
441	"	"	13.60		441(2-2)	"	30	80.86	~7	15.55
441	"	"	13.60		441(2-1)	"	25	66.16	384.2	16.07
440	"	"	11.60		440(2-2)	"	20	38.01	597.3	17.05
440	"	"	11.60		440(2-1)	"	15	29.92	914	17.58
439	"	"	11.83		439(2-2)	"	10	20.89	1.552×10^3	19.08
439	"	"	11.83		439(2-1)	"	6.9	15.20	2.097×10^3	19.52
437	"	"	11.89		437(2-2)	"	5	9.91	5.32×10^3	20.05
437	"	"	11.89		437(2-1)	"	3.1	7.65	7.12×10^3	20.43

Table 3.47 Electric resistance of simultaneously co-grafted film
100 μm HDPE; measurement temperature, 24.1±1.1°C
monomer solution, monomer:water,
Mohr's salt=50:50, 0.004 mol/1
cell resistance, 3.5 Ω.
electrolyte, 10% KOH aqueous solution

No.	AA:VP	Dose Rate (rad/h)	Irrad. Time (min.)	G (%)	R (Ω)	Swelling Time(hr.)
403	45:5	1.17×10^5	70.6	32.35	845	5.17
402	"	"	29.6	13.45	2.485×10^3	17.75
401	"	"	19.6	8.73	4.12×10^3	18.28
400	"	"	10	3.13	1.6×10^4	6
410	"	"	6	1.32	4.35×10^4	24.33
411	"	"	100	38.74	62	3.38
412	"	"	150	42.97	~5	0.37
399	40:10	"	70.6	26.65	1.495×10^3	16.38
398	"	"	29.6	10.21	2.76×10^3	16.72
397	"	"	19.6	7.02	7.97×10^3	17.37
396	"	"	10	2.58	over range	5.5
413	"	"	100	37.15	~6	3.72
414	"	"	150	38.57	~5	0.35
395	30:20	"	91	21.43	1.82×10^3	17.63
394	"	"	60	14.13	2.42×10^3	17.77
393	"	"	40	9.61	4.85×10^3	18.08
392	"	"	20.4	3.55	1.74×10^4	4.98
415	"	"	10	1.25	2.66×10^5	23.8
416	"	"	143	35.38	~5	4.42
417	"	"	213	37.83	~5	14.92
391	20:30	"	91	12.38	3.438×10^3	19.22
390	"	"	60	9.08	4.65×10^3	19.97
389	"	"	40	7.26	6.07×10^3	20.48
388	"	"	20.4	3.36	1.41×10^4	4.65
418	"	"	10	0.80	5.00×10^5	9.87
419	"	"	120	19.10	2.492×10^3	6.63
420	"	"	163	23.66	1.76×10^3	10.53
421	"	"	240	33.34	"	0.4

Table 3.48 Electric resistance as a function of swelling time (1)
--VP grafted film
electrolyte, 10% KOH aqueous solution

No.385, 100μ HDPE, G(VP)=45.98%; DR=2.62×10^4rad/h;
measurement temperature of solution: 24.1±1.1°C, $R_0 \approx 3.5\Omega$

Swelling time (min.)	3.25	5.0	10.0	14.37	21.0	27.0	734
Resistance (Ω)	664	579	499	460	445	433	449

Table 3.49 Electric resistance as a function of swelling time (2)
--film prepared by VP-AA multi-stage grafting
electrolyte, 10% KOH aqueous solution

No.381-2, 382-2, 100μ HDPE; DR=1.17×10^5rad/h;
measurement temperature of solution: 24.1±1.1°C, $R_0 \approx 3.5\Omega$

No.381-2, G_1(VP)=0.77%, G_2(AA)=34.5%

Swelling time (min.)	11.5	12.5	14	18	36	64	84	112	165	221
Resistance (Ω)	2.81×10^4	1.11×10^4	8.9×10^3	7.41×10^3	5.23×10^4	3.7×10^4	1.75×10^3	1.33×10^3	1.07×10^3	9.53×10^2

No.382-2, G_1(VP)=0.29%, G_2(AA)=45.55%

Swelling time (min.)	8.08	8.72	9.5	10.25	12	17
Resistance (Ω)	9.31×10^3	710	110	21	5.7	5.7

Table 3.50 Electric resistance as a function of swelling time (3)
--AA grafted film
electrolyte, 10% KOH aqueous solution

No.328, G(AA)=31.90%; measure T: ~25°C, R_0≈20

Swelling time (min.)	27.08	27.5	28.38	29.33	31.5	34	38	46.25
Resistance (Ω)	1.1×10^6	8.1×10^5	5.4×10^5	4×10^5	3×10^5	2.6×10^5	2.27×10^5	1.67×10^5
Swelling time (min.)	62	84	119	198	264	425	451	
Resistance (Ω)	1.343×10^5	1.135×10^5	9.55×10^4	7.99×10^4	7.23×10^4	5.62×10^4	5.52×10^4	

No.329, G(AA)=45.50%; measure T: ~25°C, R_0≈20Ω

Swelling time (min.)	7.17	9.17	9.93	10.58	11.17	11.97	12.83	21.17	109.17
Resistance (Ω)	over range	4.1×10^4	1.6×10^3	3.2×10^2	112	30	22	22	22

No.343, G(AA)=33.01%; measure T: 19°C, R_0≈20Ω

Swelling time (min.)	338	1074	1091	1155	1181	1219	1303
Resistance (Ω)	over range	3.5×10^5	3.41×10^5	3.03×10^5	2.92×10^5	2.77×10^5	2.51×10^5

No.344, G(AA)=40.23%; measure T: 19°C, R_0≈20Ω

Swelling time (min.)	10	10.17	10.67	11.45	12	12.5	13.33	14.5	17
Resistance (Ω)	over range	1.1×10^6	3.1×10^4	1.1×10^3	211	41	25	24	24

No.345, G(AA)=41.65%; measure T: 19°C, R_0≈20Ω

Swelling time (min.)	9	9.67	10.45	11.45	12	12.67	15	19
Resistance (Ω)	over range	1.1×10^6	2.1×10^3	101	30	26	25	25

Table 3.51 Electric resistance as a function of swelling time (4)
--AA grafted film
electrolyte, 10% KOH aqueous solution

No.355, G(AA)=37.66%; measure T: 17.5°C, $R_0 \approx 20\Omega$

Swelling time (min.)	10	10.83	11.25	11.83	12.67	13.08	13.83	16	19
Resistance (Ω)	over range	1.1×10^6	1.11×10^5	1.11×10^4	2.1×10^3	171	31	28	27

No.356, G(AA)=40.28%; measure T: 18.5°C, $R_0 \approx 20\Omega$

Swelling time (min.)	12	12.17	12.25	14	17	21
Resistance (Ω)	over range	1.11×10^6	1.7×10^3	111	21	21

Table 3.52 Electric resistance as a function of swelling time (5)
--AA grafted film
electrolyte, 10% KOH aqueous solution

No.264, G(AA)=49.29%; measure T: ~25°C, R_0≈20Ω

Swelling time (min.)	1	4	6	7.5	16
Resistance (Ω)	over range	1.6×10^4	300	33	20

No.265, G(AA)=51.11%; measure T: ~25°C, R_0≈20Ω

Swelling time (min.)	2	3	4	5	6	7	40
Resistance (Ω)	over range	1.1×10^4	4.1×10^2	1.4×10^2	40	23	21

No.266, G(AA)=53.87%; measure T: ~25°C, R_0≈20Ω

Swelling time (min.)	3	3.5	4.33	4.83	6	6.17	6.83	7.58	8.17	10	13
Resistance (Ω)	1.11×10^6	4.1×10^5	6.9×10^3	2.37×10^3	570	210	100	40	25	21	21

No.267, G(AA)=56.79%; measure T: ~25°C, R_0≈20Ω

Swelling time (min.)	3.67	4	4.58	5	5.75	6.5	6.83	7.58	8.33	9	10
Resistance (Ω)	over range	over range	1.11×10^6	3.51×10^5	3.5×10^4	4×10^3	1.14×10^3	240	53	28	21

No.268, G(AA)=56.91%; measure T: ~25°C, R_0≈12Ω

Swelling time (min.)	5	5.58	6.25	6.92	7.67	8.42	9.25	11
Resistance (Ω)	1.11×10^6	2.71×10^5	5.3×10^4	6.52×10^3	6.2×10^2	112	23	14

Table 3.53 Electric resistance as a function of swelling time (6)
--AA grafted film
electrolyte, 10% KOH aqueous solution

No.336, G(AA)=20.14%, DR=1.88×10^3rad/h, irrad. T: 50°C;
measure T: 25°C, $R_0\approx20\Omega$

Swelling time (min.)	24	50	58	81	110	167	204	240
Resistance (Ω)	4.75×10^3	1.176×10^3	951	682	548	434	395	366
Swelling time (min.)	255	267	297	325	257	380	420	465
Resistance (Ω)	355	348	329	314	303	297	288	280

No.338, G(AA)=26.23%, DR=1.63×10^3rad/h, irrad. T: 50°C;
measure T: 25°C, $R_0\approx20\Omega$

Swelling time (min)	13	15	16.33	17.75	19	25.33	26	27
Resistance (Ω)	over range	1.6×10^4	1.4×10^3	85	30	26	26	26

No.339, G(AA)=32.08%, DR=1.50×10^3rad/h, irrad. T: 50°C;
measure T: 25°C, $R_0\approx20\Omega$

Swelling time (min.)	13	14.08	14.63	15.58	16.5	17.25	18	18.58	21	24
Resistance (Ω)	over range	1.1×10^6	1.11×10^4	5.1×10^3	910	251	51	31	26	26

No.340, G(AA)=29.18%, DR=1.35×10^3rad/h, irrad. T: 50°C;
measure T: 25°C, $R_0\approx20\Omega$

Swelling time (min.)	13	14.5	15.25	16.17	17.17	18	19	26
Resistance (Ω)	over range	1.2×10^4	1.5×10^3	380	111	28	26	26

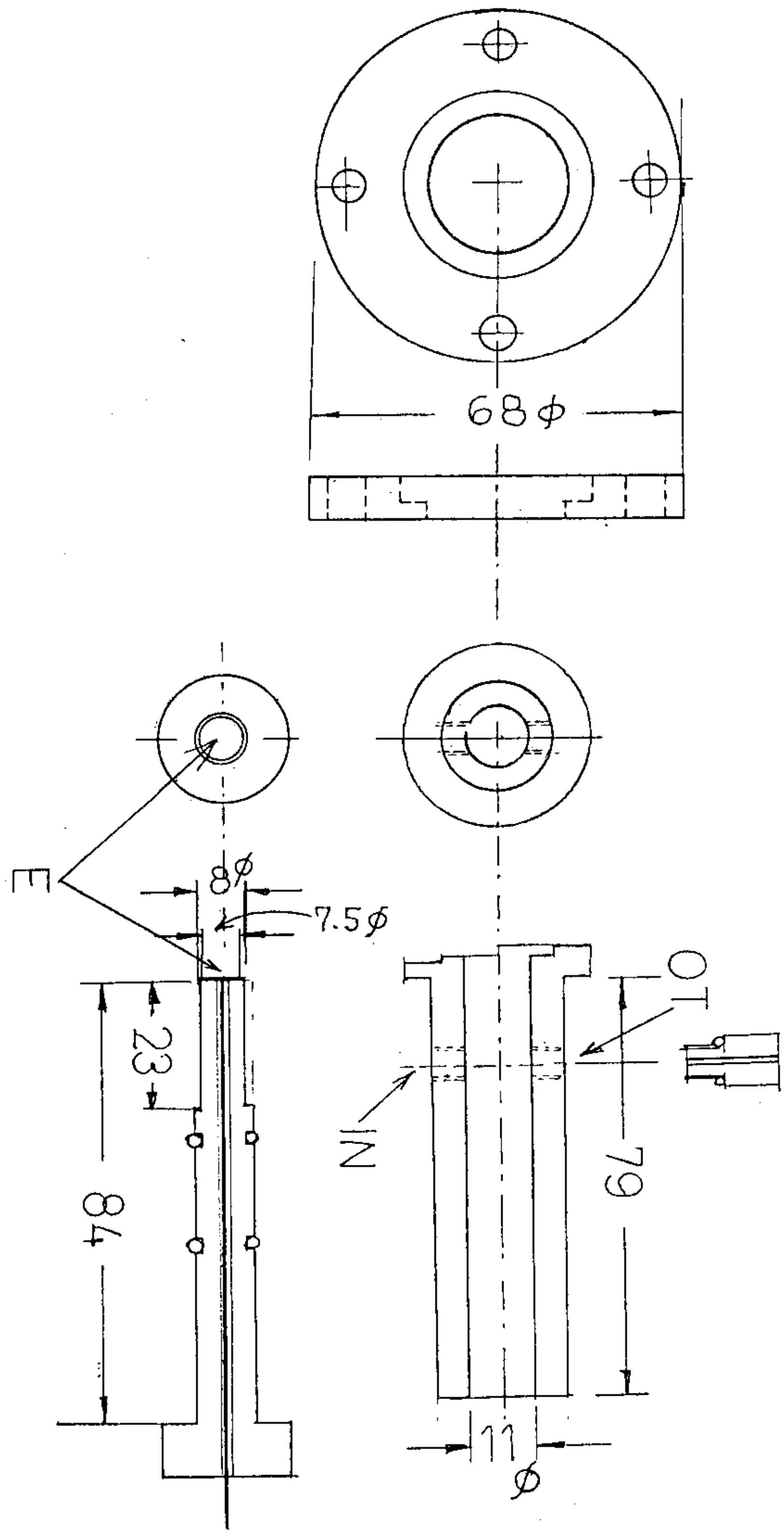

Fig. 2.1 Apparatus for electric resistance across grafted films; E, platinum electrode (7.5mmϕ); IN, entrance of electrolyte; OT, exit of electrolyte. Area of grafted film is 1 cm^2.

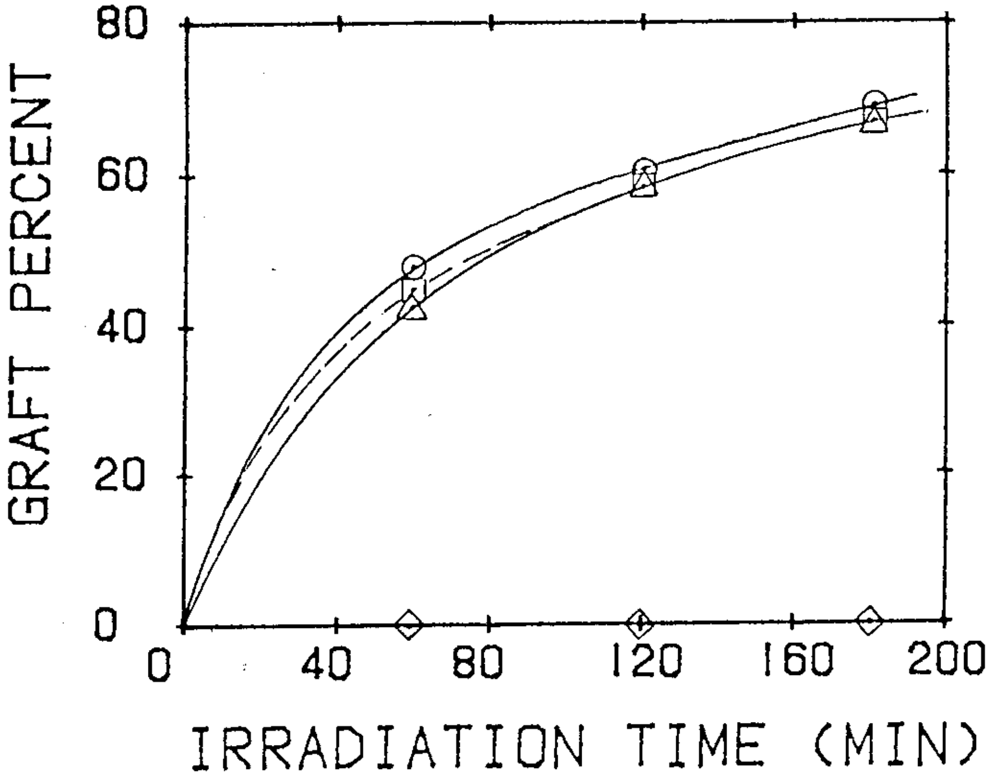

Fig. 3.1 Graft percent as a function of irradiation time; 13 μm HDPE, dose rate: 2.57×10^5rad/h; temperature: 15°C in air; monomer solution: AA:water, Fe^{2+}=50:50 by vol.,0.004mol/l; ◇#17-19, irradiated in nitrogen atmosphere, after irradiation, immersed in monomer solution; △ #20-22, irradiated in deaerated monomer solution, and the film was taken out in air and then immersed in 50°C for 4 hr; ○ #23-25, irradiated in the deaerated monomer solution, and then the film was taken out in air and then immersed in 10°C water for 4 hr; □ #26-28, irradiated in the deaerated monomer solution, and the film was taken out in air and then just washed and dried in an oven to determine graft percent.

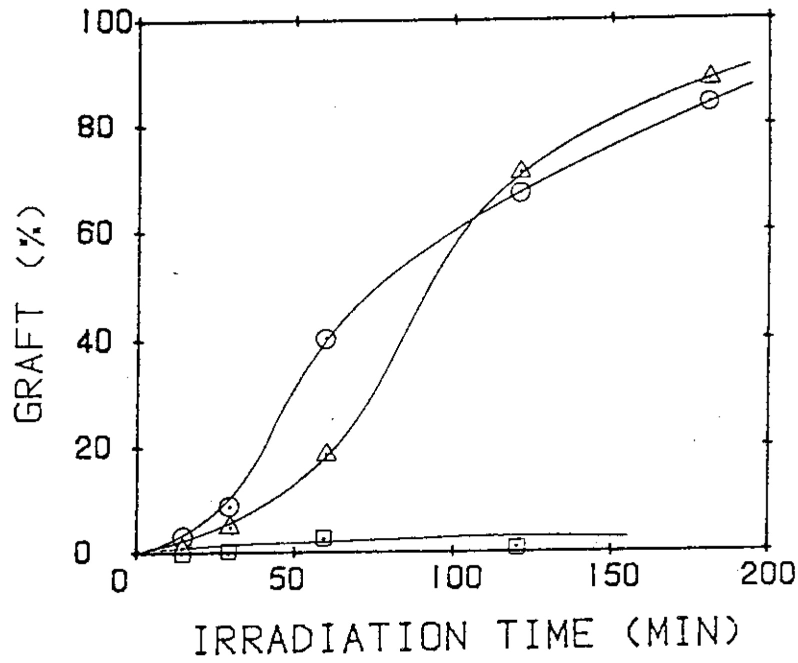

Fig. 3.2 Graft percent as a function of irradiation time; 13 μm HDPE, dose rate: 2.25×10^5rad/h; temperature: 14.2°C in air; monomer solution: AA:water, Fe^{2+}=50:50 by vol.,0.004mol/l; ○ #39-44, irradiated in monomer solution deaerated by bubbling nitrogen for 15 min; △ #45-50, irradiated in air saturated monomer solution, □ #51-56, irradiated in the monomer solution bubbled with oxygen for 15 min. After irradiation, films were taken out in air and then immersed in 50°C water for 4 hr., and the film was dried in an oven to determine graft percent.

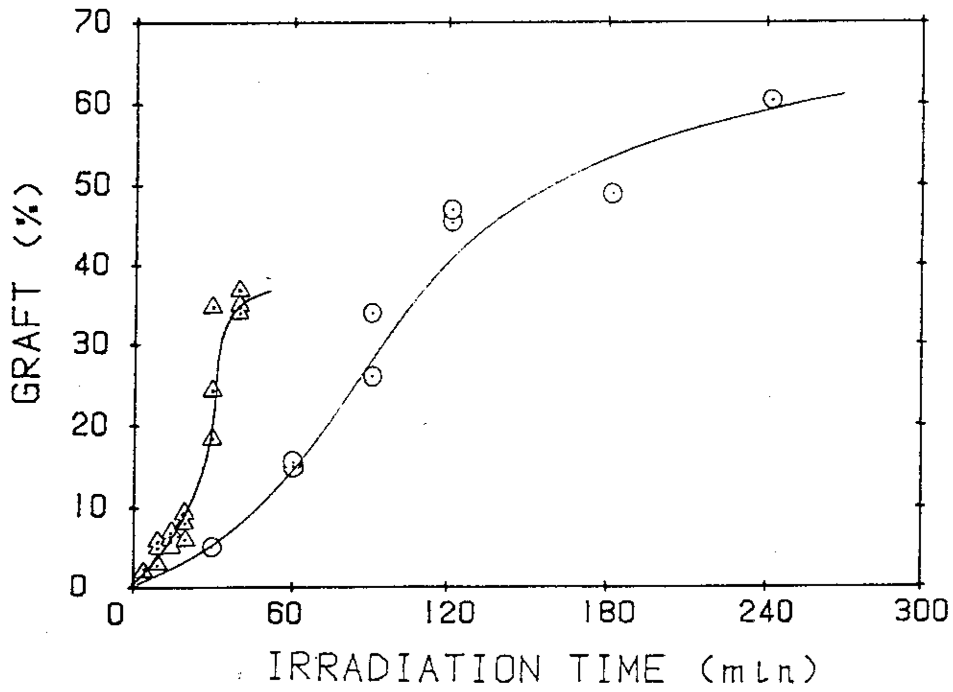

Fig. 3.4 Graft percent as a function of irradiation time--Effect of Mohr's salt concentration; 100 μm HPDE,; ○ #1-9, monomer solution: AA:water, Fe^{2+}=50:50 by vol.,0.004mol/l; dose rate: 1.35×10^5rad/h; temperature: 15.1°C in air; △ #143-160, monomer solution: AA:water, Fe^{2+}=50:50 by vol.,0.001mol/l; dose rate: 1.23×10^5rad/h; temperature: 10.5°C in air.

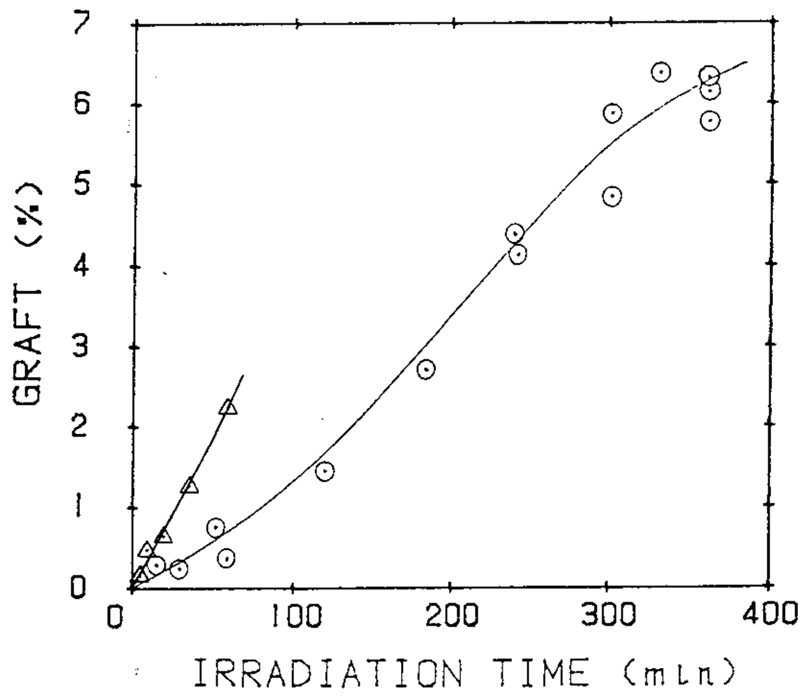

Fig. 3.3 Graft percent as a function of irradiation time; 100 μm HDPE, monomer solution: AA:water, Fe^{2+}=50:50 by vol.,0.004mol/l; ○ #205-219, dose rate: 0.993×10^5rad/h; temperature: 10.8°C in water bath; Δ #311-316, dose rate: 1.12×10^5rad/h; temperature: 11.0°C in air.

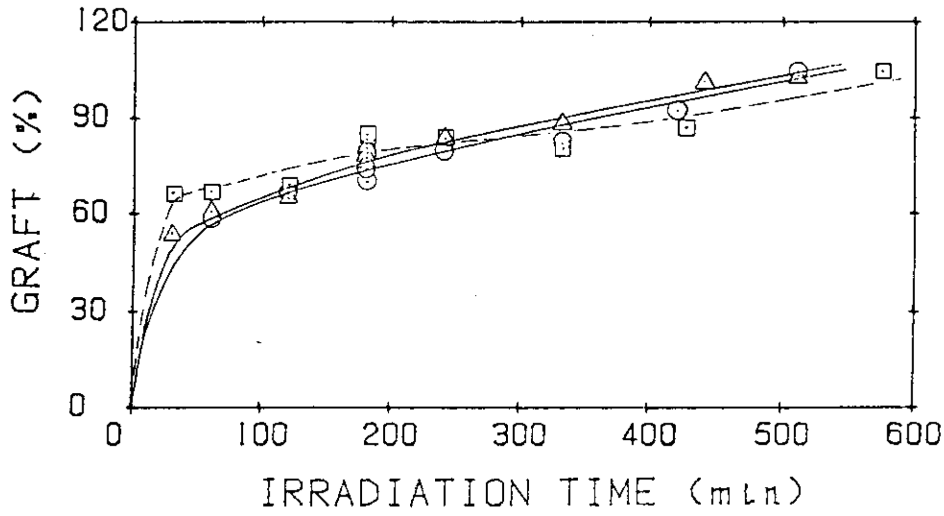

Fig. 3.5 Graft percent as a function of irradiation time--Effect of storage time of Mohr's salt stock solution(0.008mol/l); 27 μm LPDE,; dose rate: 1.19×10^5rad/h; temperature: 50.0°C; monomer solution: AA:water, Fe^{2+}= 50:50 by vol.,0.004mol/l; stock time: ○ #235-243, with one hour; △ #244-253, one day; □ #273-282, one week.

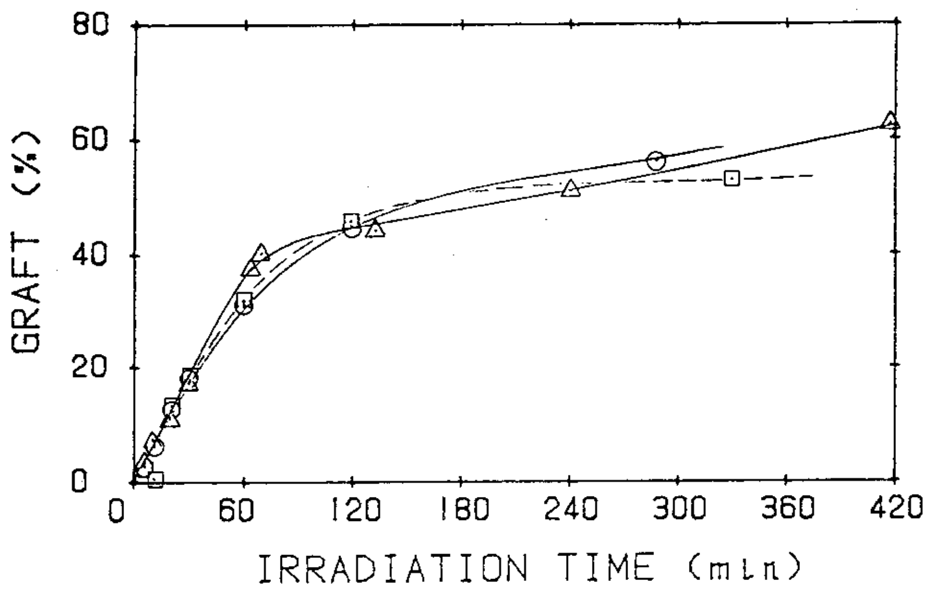

Fig. 3.6 Graft percent as a function of irradiation time--Effect of storage time of Mohr's salt stock solution; 100 μm HPDE,; dose rate: 1.18×10^5rad/h; temperature: 50.0°C; monomer solution: AA:water, Fe^{2+}=50:50 by vol., 0.004mol/l; stock time: ○ #317-323, with one hour; △ #324-330, one day; □ #351-359, one week.

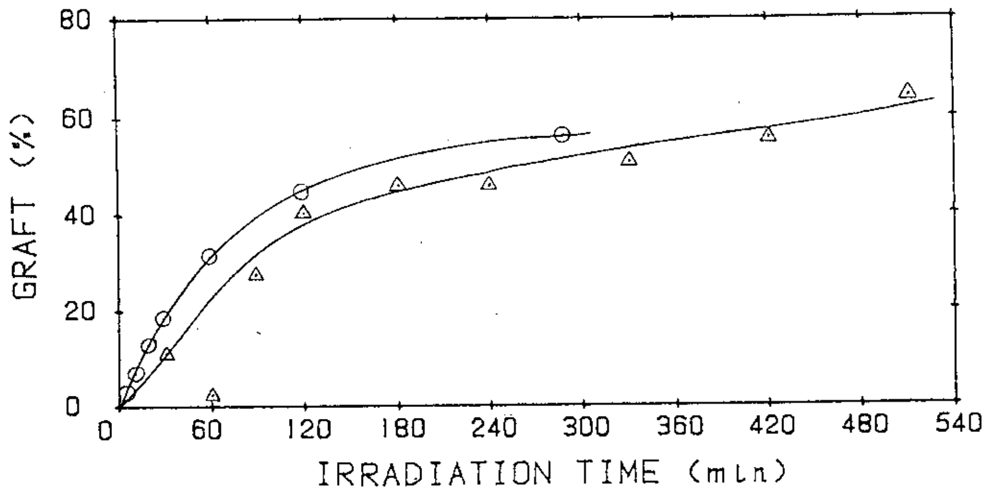

Fig. 3.7 Graft percent as a function of irradiation time--Effect of storage time of monomer solution; 100 μm HPDE,; dose rate: 1.18×10^5 rad/h; temperature: 50.0°C; monomer solution: AA:water, Fe^{2+}=50:50 by vol.,0.004mol/l; stock time: ○ #317-323, with one hour; △ #254-262, nine days.

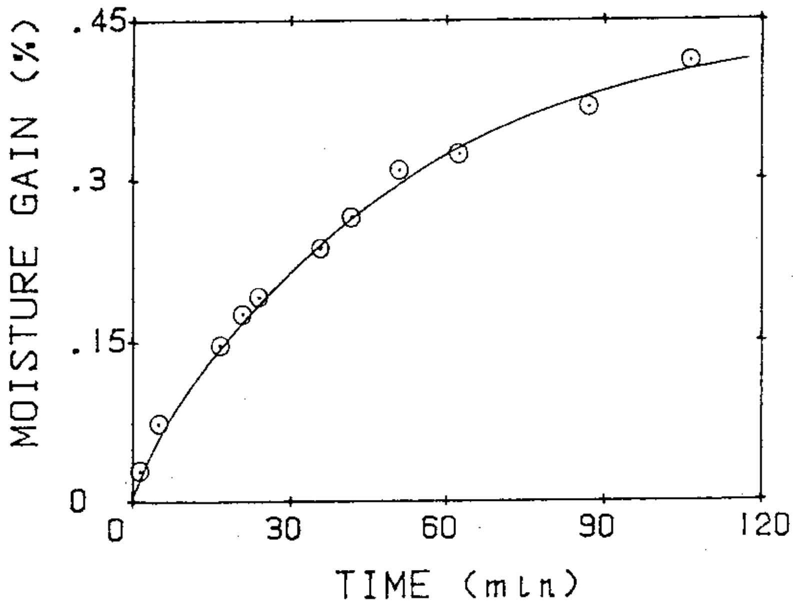

Fig. 3.8 Moisture gain as a function of time exposed to air; 100 μm HPDE, #345, graft percent of AA, 41.65%; temperature 19.9°C; humidity, 50%.

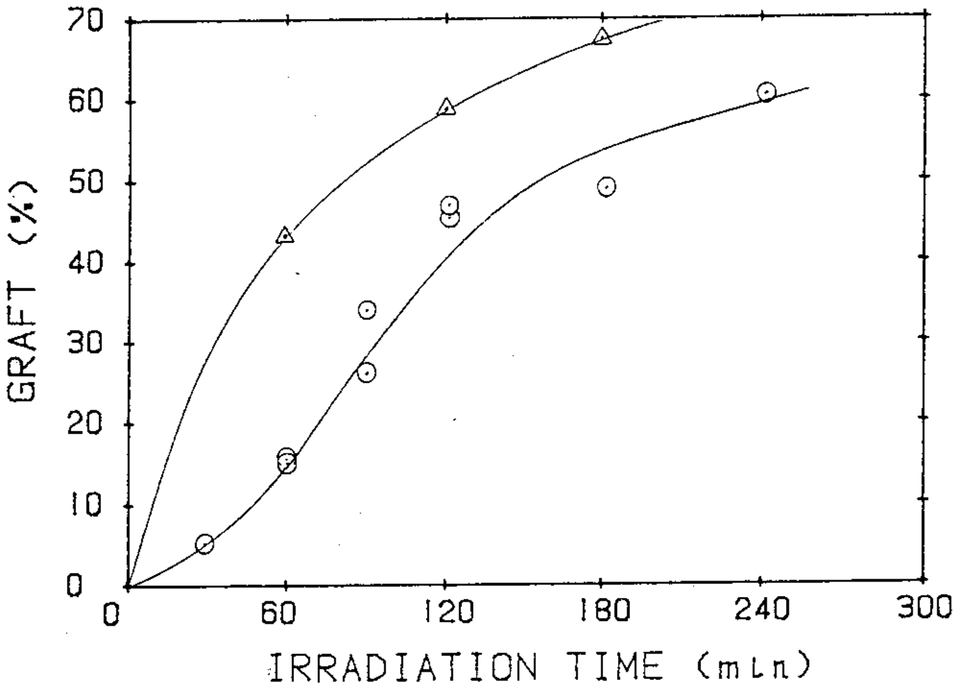

Fig. 3.9 Graft percent as a function of irradiation time--Effect of dose rate: 13 μm HPDE; monomer solution: AA:water, Fe^{2+}=50:50 by vol.,0.004mol/l; ○ #1-9, dose rate: 1.35×10^5 rad/h; temperature: 15.0-15.2°C; Δ #20-22, dose rate: 2.57×10^5rad/h; temperature: 15.0°C.

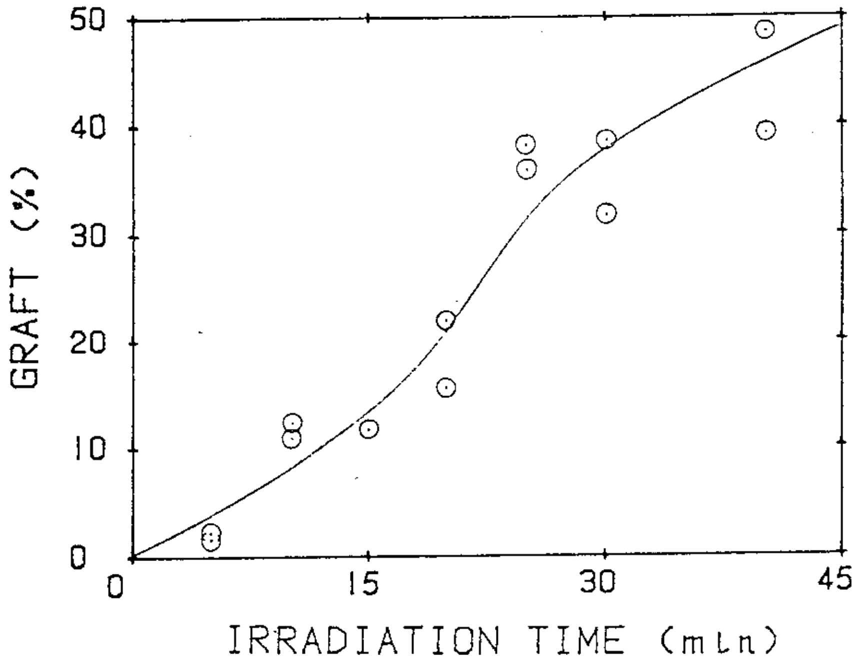

Fig. 3.10 Graft percent as a function of irradiation time; 27 μm LDPE; monomer solution: AA:water, Fe^{2+}=50:50 by vol.,0.001mol/l; O #220-233, dose rate: 1.21×10^{5}rad/h; temperature: 9°C.

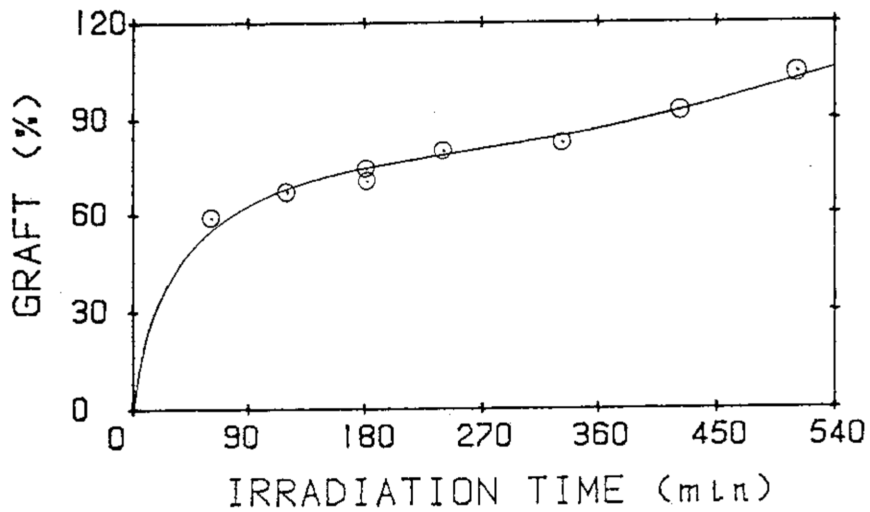

Fig. 3.11 Graft percent as a function of irradiation time; 27 μm LDPE; monomer solution: AA:water, Fe^{2+}=50:50 by vol.,0.004mol/l; O #235-243, dose rate: 1.19×10^{5}rad/h; temperature: 50°C.

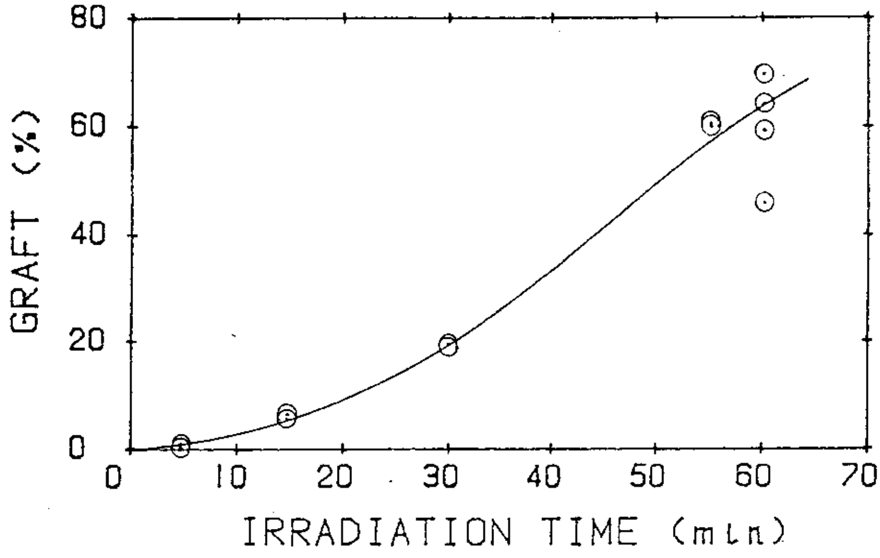

Fig. 3.12 Graft percent as a function of irradiation time; 13 μm HPDE; monomer solution: VP:water, Fe^{2+}=50:50 by vol.,0.004mol/l; ○ #283-298, dose rate: 1.18 × 10^5rad/h; temperature: 50°C.

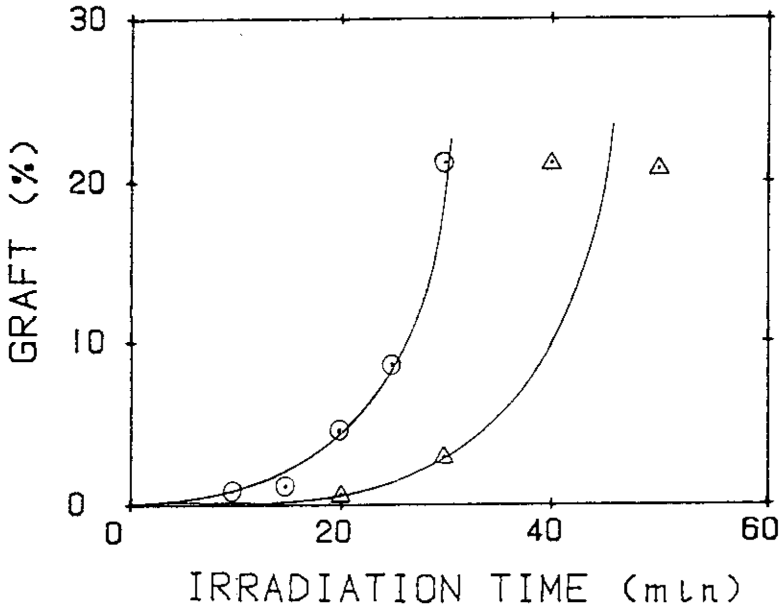

Fig. 3.13 Graft percent as a function of irradiation time; 13 μm HPDE; monomer solution: VP:water, Fe^{2+}=50:50 by vol.,0.001mol/l; temperature: 13.8°C; ○ #76-80, dose rate: 2.23 × 10^5rad/h; Δ #81-84, dose rate: 1.23 × 10^5rad/h.

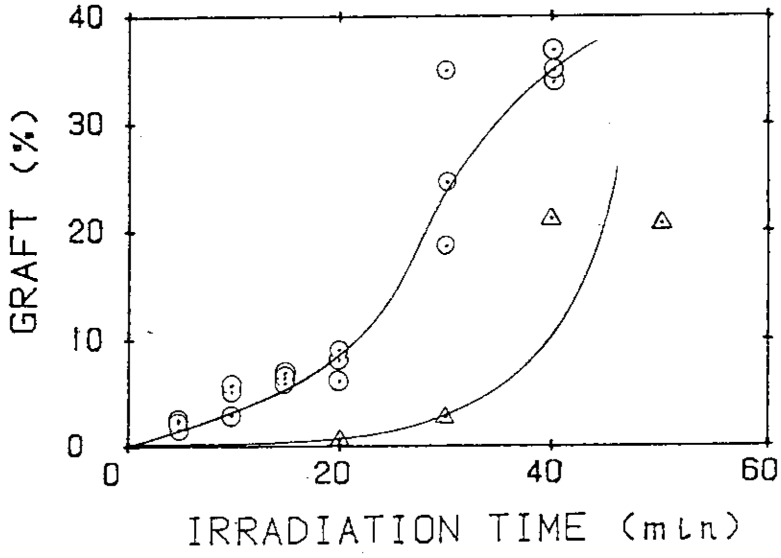

Fig. 3.14 Graft percent as a function of dose; 13 μm HPDE; monomer solution: VP:water, Fe^{2+}=50:50 by vol.,0.001mol/l; temperature: 13.8°C; ○ #76-80, dose rate: 2.23×10^5rad/h; △ #81-84, dose rate: 1.23×10^5rad/h.

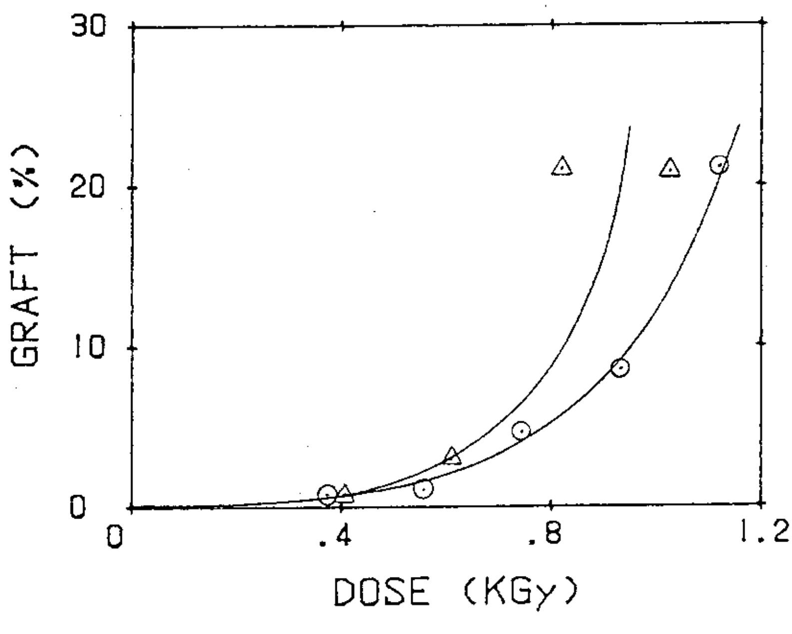

Fig. 3.15 Graft percent as a function of irradiation time--Comparison of time conversion curves of AA and VP graft; 13 μm HPDE; monomer solution: monomer:water, Fe^{2+}=50:50 by vol.,0.001mol/l; dose rate: 1.23×10^5rad/h; ○ #143-160; AA; temperature: 10.5°C; △ #81-84, temperature: 13.8°C.

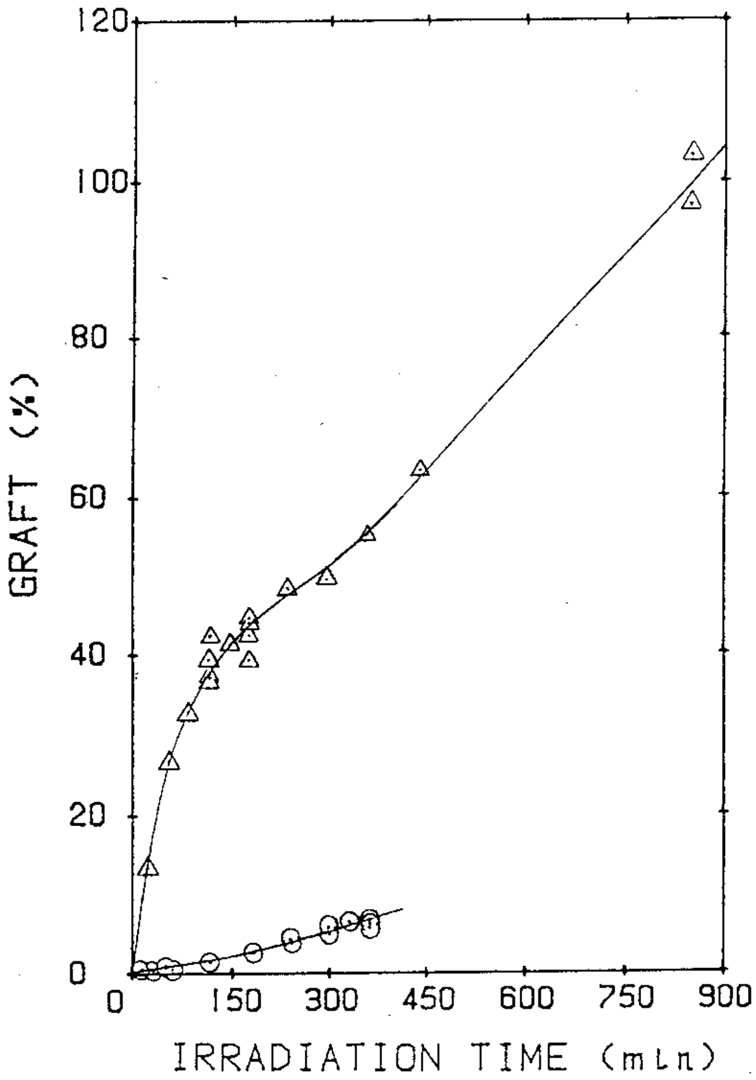

Fig. 3.16 Graft percent as a function of irradiation time--Effect of temperature; 100 μm HPDE; monomer solution: AA:water, Fe^{2+}=50:50 by vol.,0.004mol/l; dose rate: 0.993×10^5rad/h; ○ #205-219; temperature: 10.8°C in water bath; △ #81-84, temperature: 49°C in water bath.

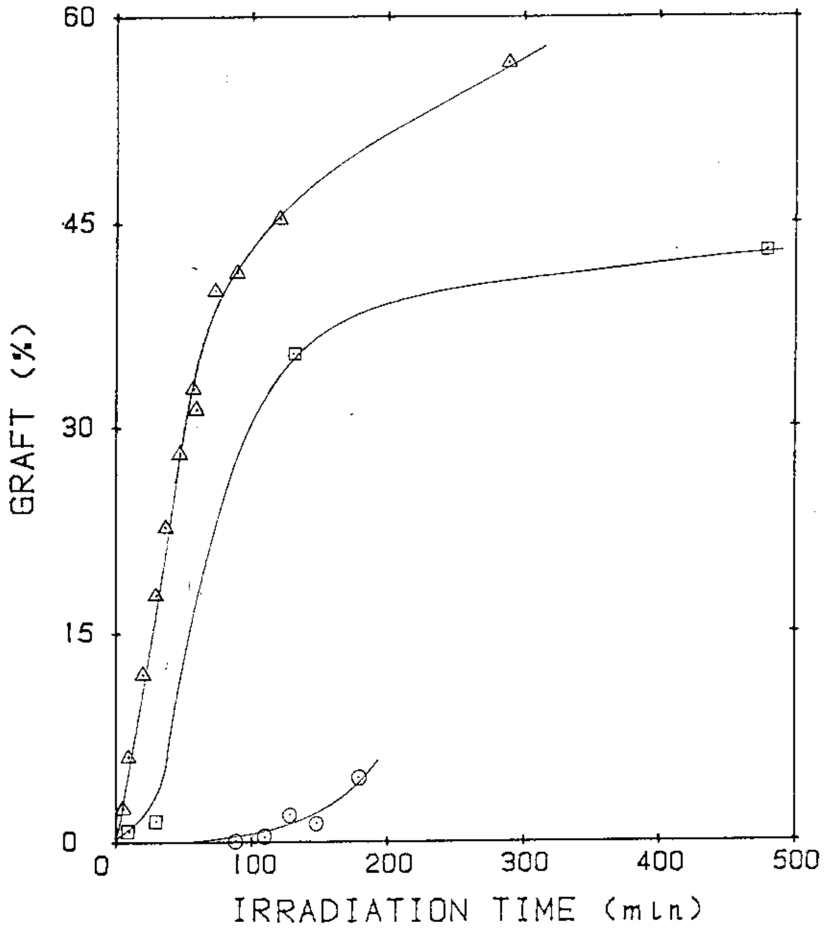

Fig. 3.17 Graft percent as a function of irradiation time--Effect of dose rate; 100 μm HPDE; monomer solution: AA:water, Fe^{2+}=50:50 by vol.,0.004mol/l; temperature, 50°C;
○ #346-350; AA; dose rate: 2.11×10^3rad/h;
□ #301-304,306; dose rate: 0.266×10^5rad/h;
△ #317-323,341-345, dose rate: 1.18×10^5rad/h.

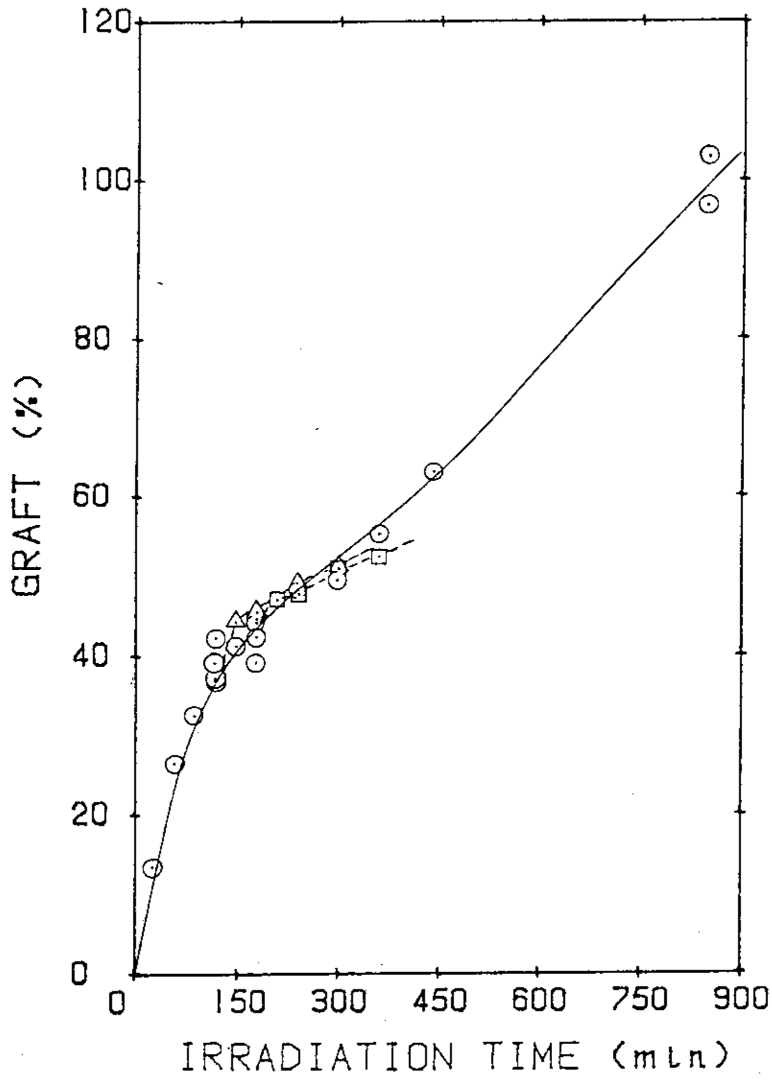

Fig. 3.18 Graft percent as a function of irradiation time--Effect of multi-step grafting; 100 μm HPDE; monomer solution: AA:water, Fe^{2+}=50:50 by vol.,0.004mol/l; temperature, 49°C; dose rate: 0.993×10^5rad/h; ○ #187-204; □ the second stage grafting initiated after 180 min of the first grafting, #190(2)-193(2); △ the second stage grafting initiated after 120 min of the first grafting, #189(2),197(2)-199(2); Number in parentheses indicate number of graft steps.

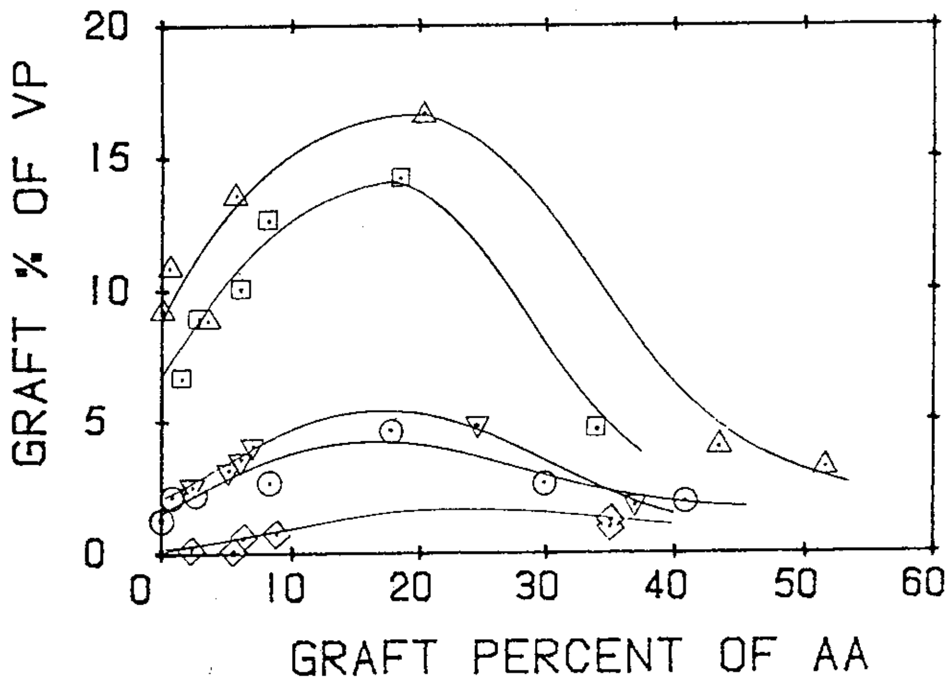

Fig. 3.19 Graft percent of VP at the second stage and that of AA at the first stage grafting; 13 μm HPDE; dose rate: 1.23 × 10^5rad/h; monomer solution: VP:water, Fe^{2+}=50:50 by vol., 0.001mol/l; △ #113-118,142; irradiation time: 40.3 min, temperature: 12°C, □ #161-166; irradiation time: 35 min, temperature: 10.5°C; ▽ #167-172; irradiation time: 25 min, temperature: 10.5°C; #107-112,140; irradiation time: 20.3 min, temperature: 12°C; ◇ #139,173-178; irradiation time: 10 min, temperature: 10.5°C.

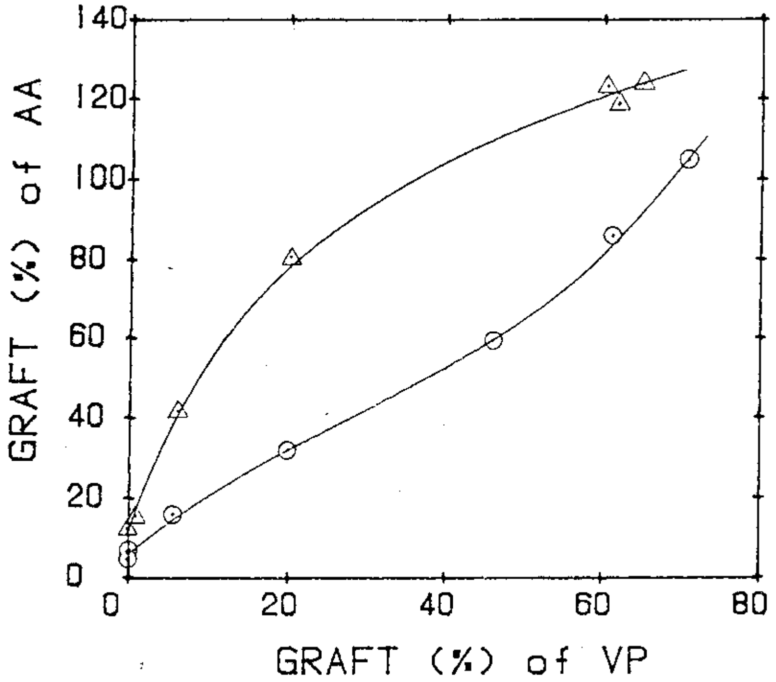

Fig. 3.20 Graft percent of AA at the second stage and that of VP at the first stage grafting; 13 μm HPDE; dose rate: 1.12×10^5rad/h; monomer solution: AA:water, Fe^{2+}=50:50 by vol., 0.001mol/l; temperature: 11°C; ○ #283(2)-287(2),289(2),299; irradiation time: 15 min; Δ #291(2)-294(2),297(2)-298(2),300: irradiation time: 30 min.

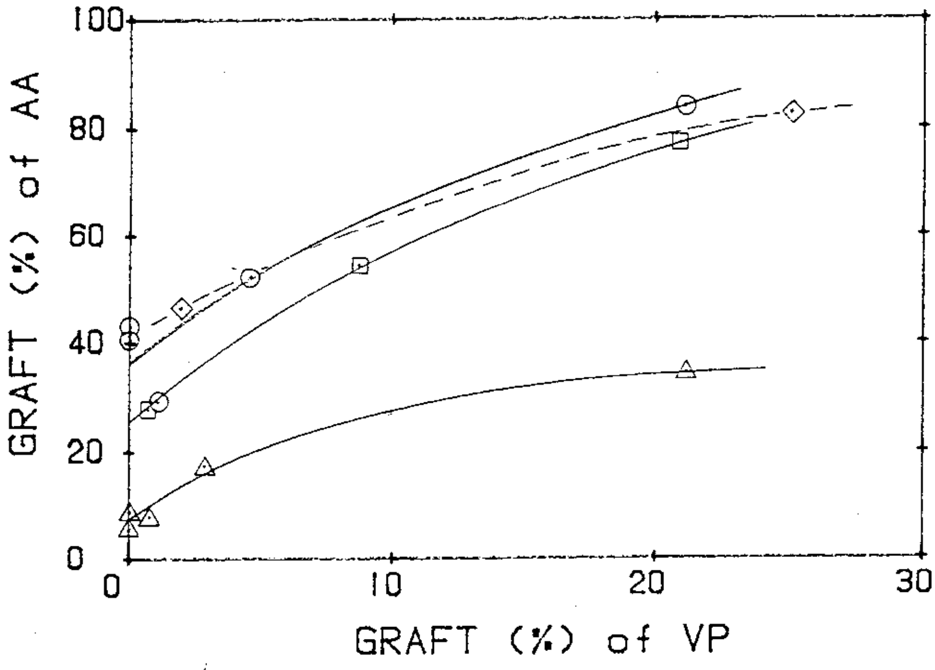

Fig. 3.21 Graft percent of AA at the second stage and that of VP at the first stage grafting at lower graft percent of VP; 13 μm HDPE; monomer solution: AA:water, Fe^{2+}=50:50 by vol.,0.001mol/l; ◇ #70,71; dose rate: 2.23 × 10^5rad/h; temperature: 14.2°C; □ #88,90,92; dose rate: 2.23 × 10^5rad/h; temperature: 12.5°C; ○ #86,89,91,103,104; dose rate: 1.23 × 10^5rad/h; temperature: 12.5°C; △ #87,93,94,97,98; dose rate: 1.23 × 10^5rad/h; temperature: 12.5°C.

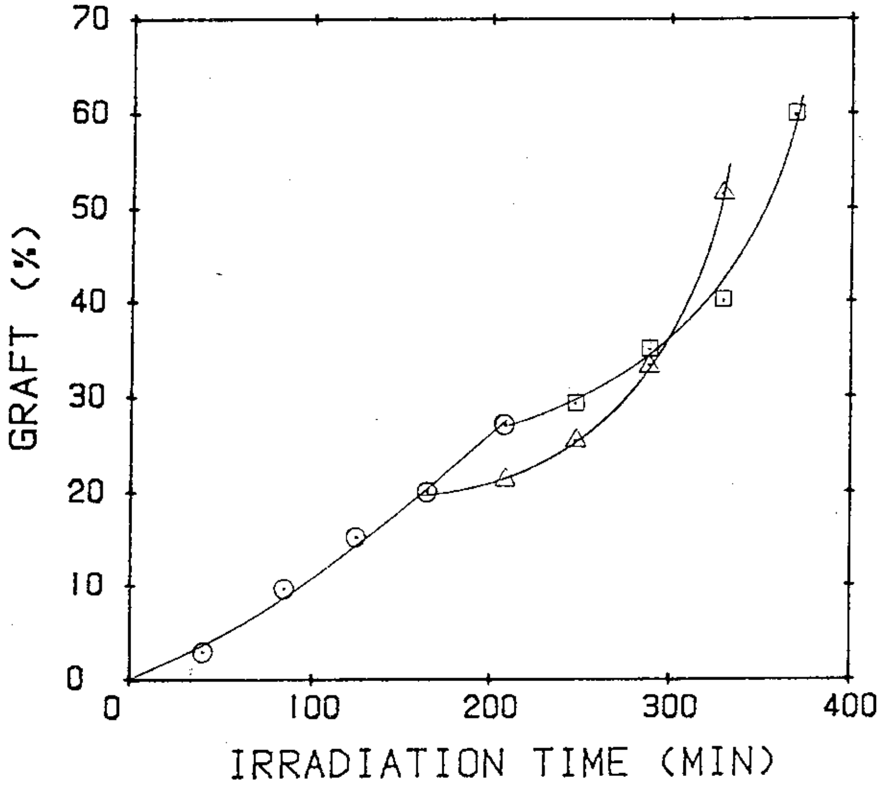

Fig. 3.22 Graft percent as a function of irradiation time in the multistep/stage grafting; 100 μm HDPE; monomer solution: monomer:water, Fe^{2+}= 50:50 by vol.,0.001mol/l; temperature: 10°C in air; dose rate: 1.22×10^5rad/h; ○ the first stage grafting with AA; □ second stage grafting with VP; △ the second stage grafting with VP; Only average of the data plotted (see Table 1.15).

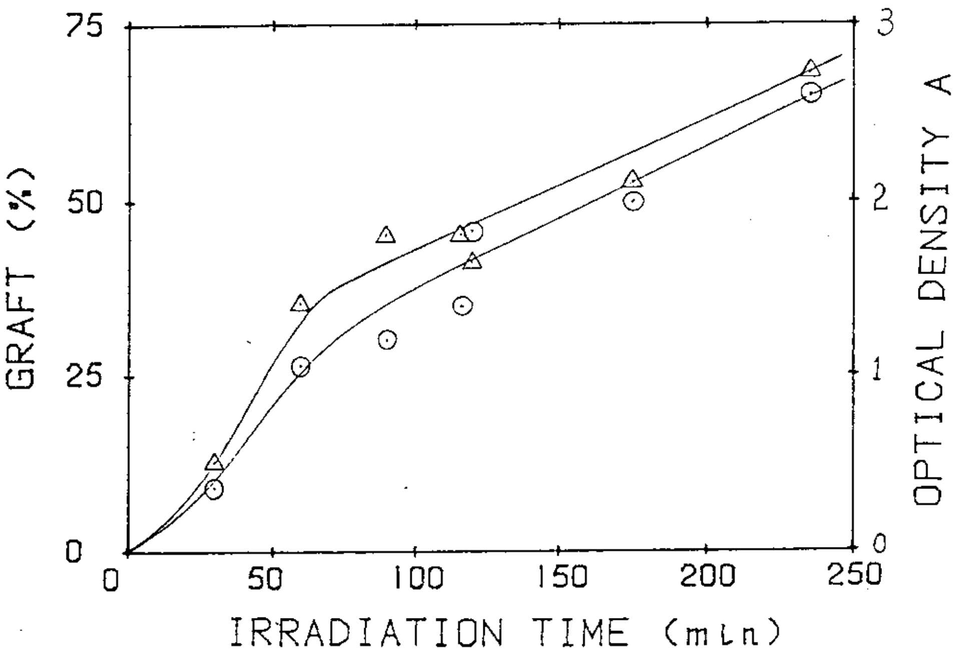

Fig. 3.23 Graft percent and O.D. of carbonyl bands as a function of irradiation time; 13 μm HDPE; monomer solution: monomer:water, Fe^{2+}=50:50 by vol.,0.004mol/l; temperature: 15°C in air; dose rate: 2.57×10^{5}rad/h.

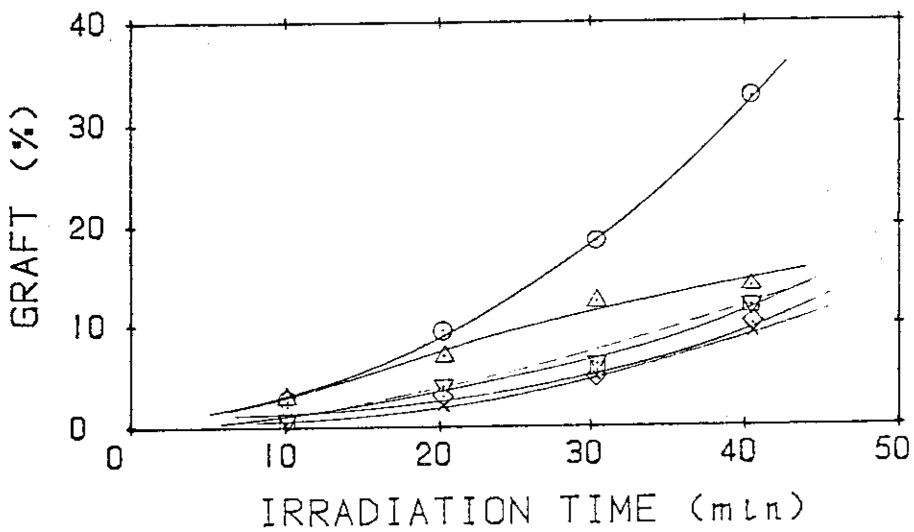

Fig. 3.24 Graft percent as a function of irradiation time in simultaneous co-grafting using monomer solution containing different amounts of AA and VP; 13 μm HDPE; monomer solution: monomer:water, Fe^{2+}=50:50 by vol.,0.001mol/l; temperature: 12°C in air; dose rate: 1.23×10^{5}rad/h; #119–142; AA:VP ratio, ○ 50:0, △ 40:10, □ 30:20, ▽ 20:30, ◇ 10:40, × 0:50.

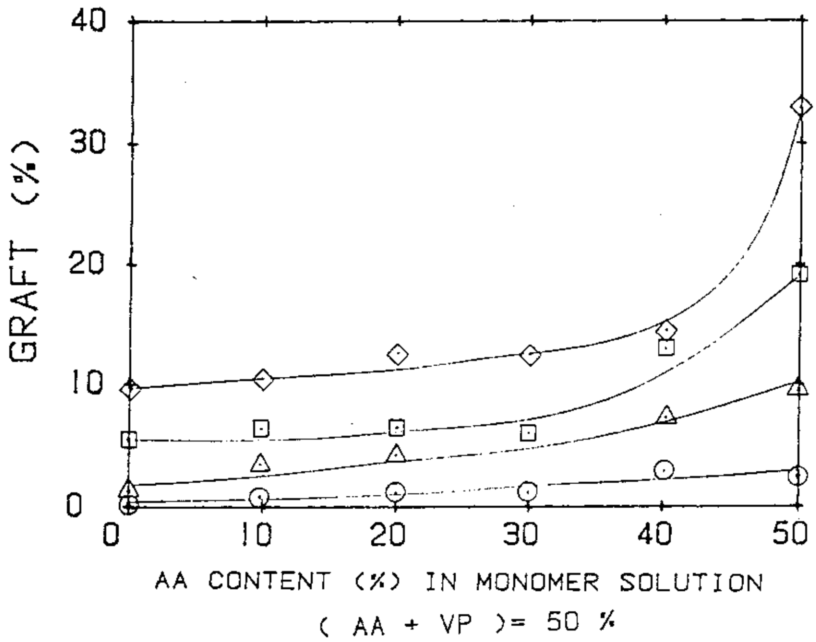

Fig. 3.25 Graft percent as a function of AA content in the monomer solution in simultaneous co-grafting using monomer solution containing different amounts of AA and VP; 13 μm HDPE; monomer solution: monomer:water, Fe^{2+}=50:50 by vol.,0.001mol/1; temperature: 12°C in air; dose rate: 1.23×10^5rad/h; #119-142; irradiation time, ○ 10 min, △ 20 min, ◇ 30 min, 40 min.

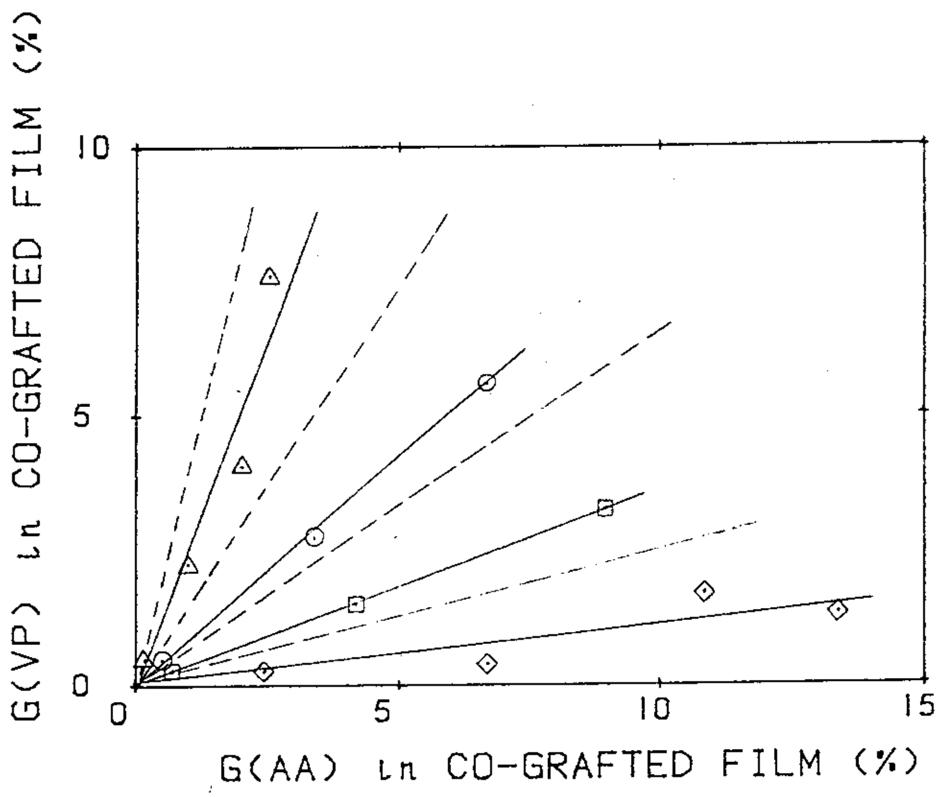

Fig. 3.26 Graft percent of VP and that of AA in simultaneous co-grafting film using monomer solution containing different amounts of AA and VP; 13 μm HDPE; monomer solution: monomer:water, Fe^{2+}=50:50 by vol.,0.001mol/l; temperature: 12°C in air; dose rate: 1.23 × 10^5rad/h; #119-142; AA:VP ratio, △ 40:10, ○ 30:20, □ 20:30, ◇ 10:40.

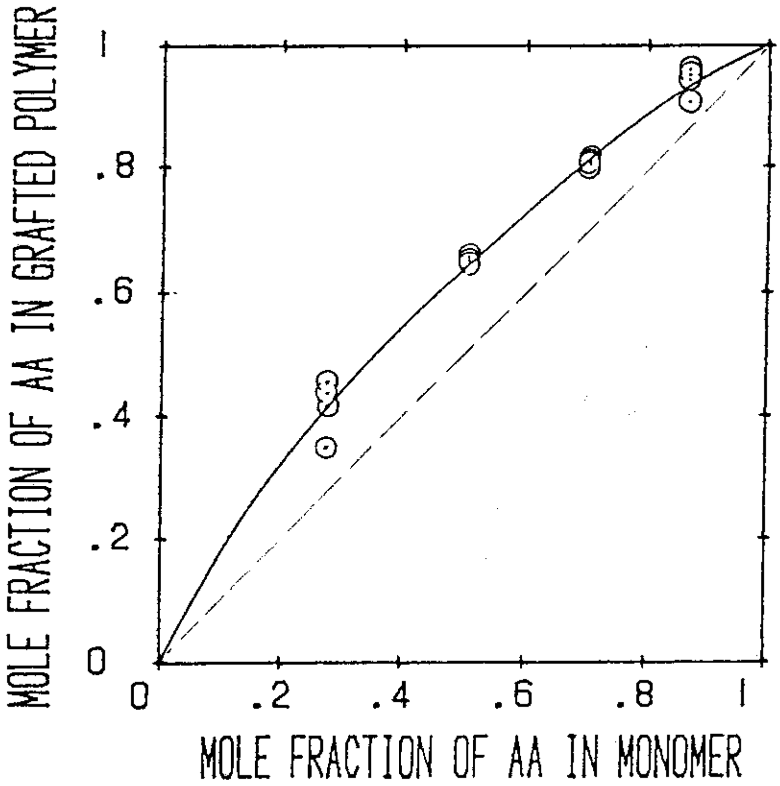

Fig. 3.27 Mole fraction of AA in co-grafted film and that in monomer solution; monomer solution: monomer:water, Fe^{2+}=50:50 by vol.,0.001mol/1; AA+VP=50%; temperature: 12°C in air; dose rate: 1.23×10^5rad/h; #119-142.

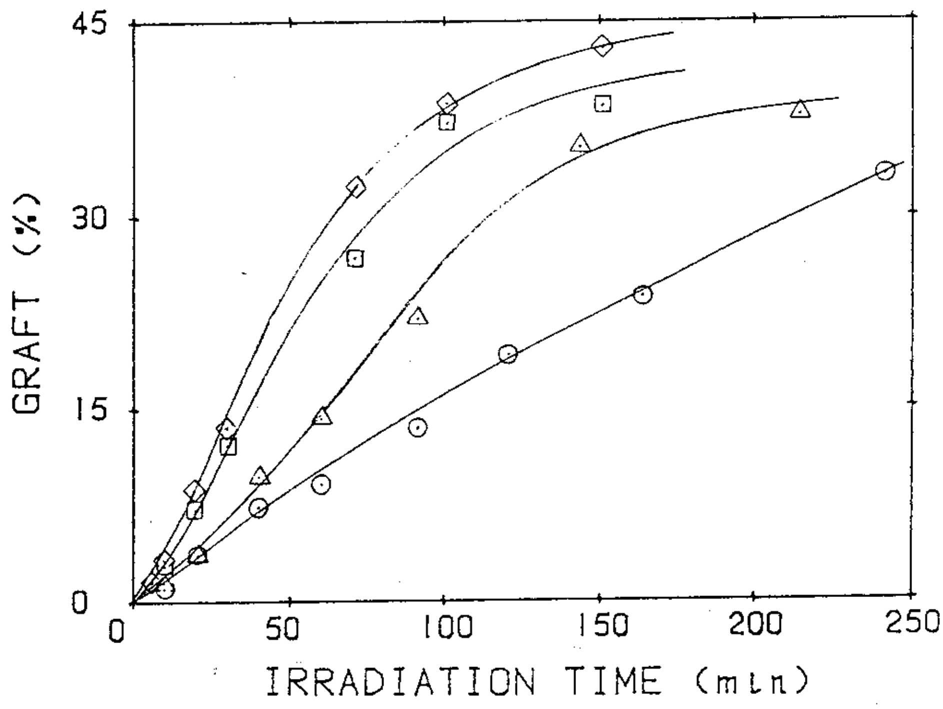

Fig. 3.28 Graft percent as a function of irradiation time in simultaneous co-grafting using monomer solution containing different amounts of AA and VP; 100 μm HDPE; monomer solution: monomer:water, Fe^{2+}=50:50 by vol.,0.004mol/l; temperature: 50°C in water bath; dose rate: 1.17×10^5rad/h; #388-403, 410-421; AA:VP ratio, ○ 20:30, △ 30:20, □ 40:10, ◇ 45:5.

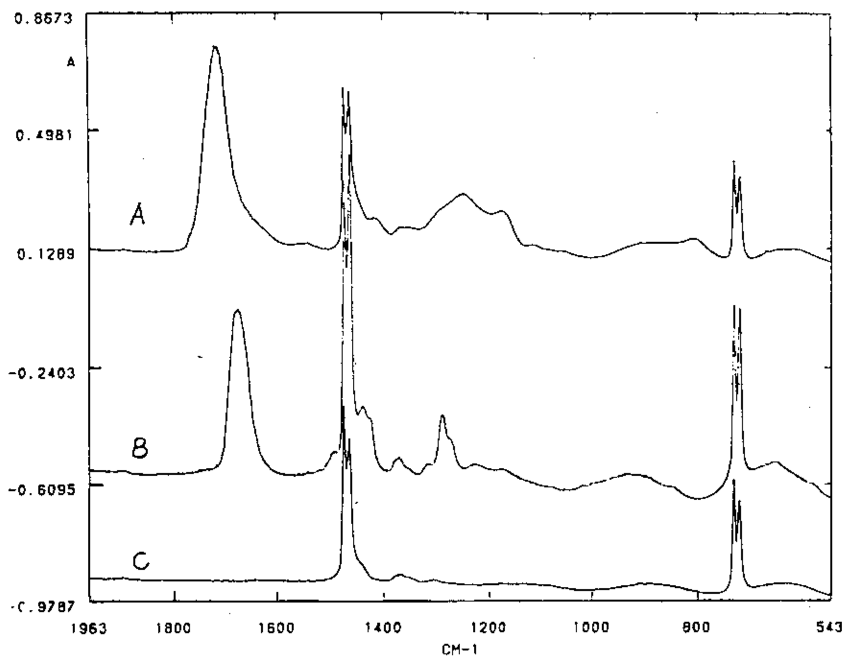

Fig. 3.29 Infrared spectra of (A) AA grafted PEAA121, (B) VP grafted PEVP141, f=0.414, and (C) original PE films, PEO, f=0.991.

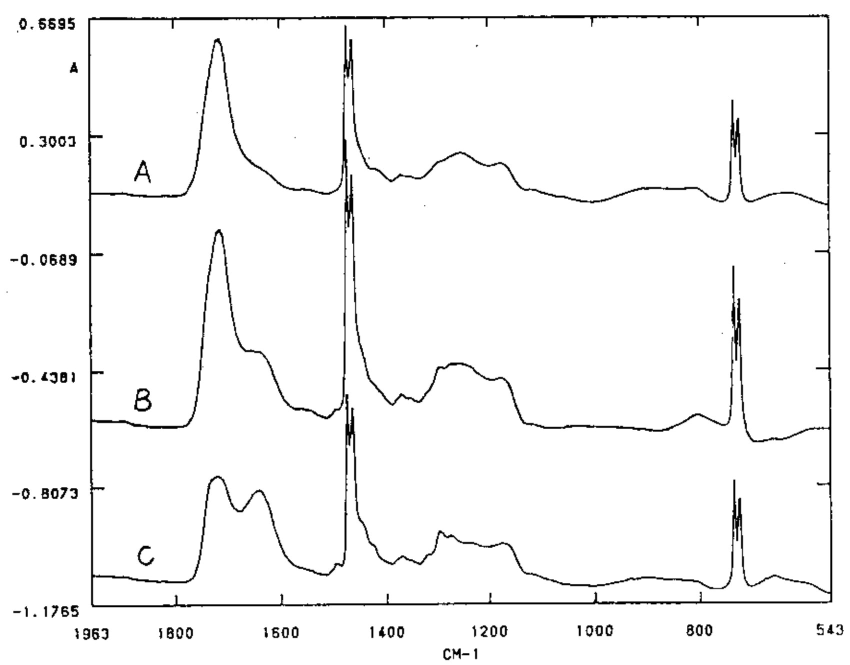

Fig. 3.30 Infrared spectra of simultaneous co-grafted films obtained from monomer solution of different AA contents; AA:VP, (A) 40:10 PEAV125, (B) 30:20, PEAV130, f=0.662, (C) 20:30, PEAV134, f=0.912.

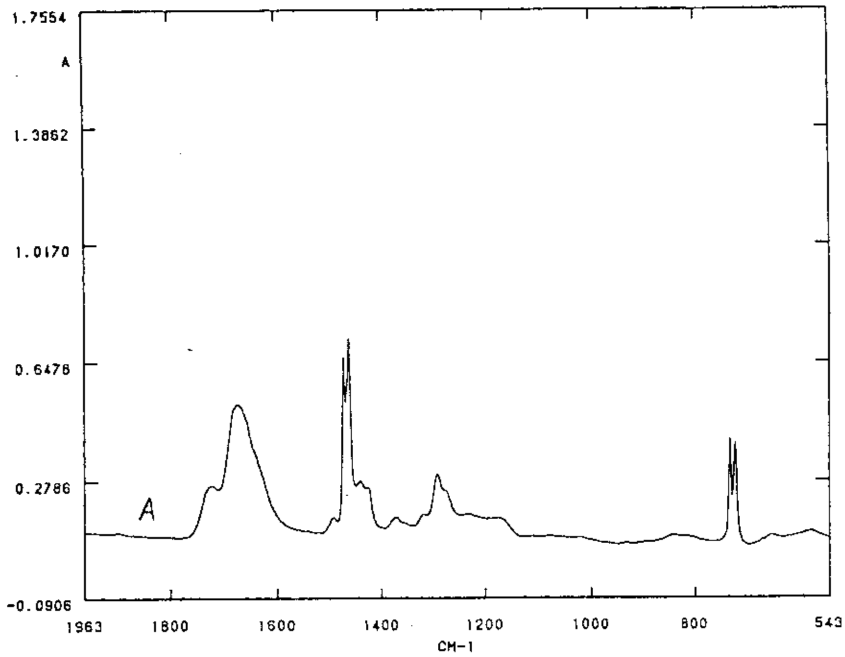

Fig. 3.31 Infrared spectra of simultaneous co-grafted films obtained from monomer solution of different AA contents; AA:VP=10:40, PEAV138.

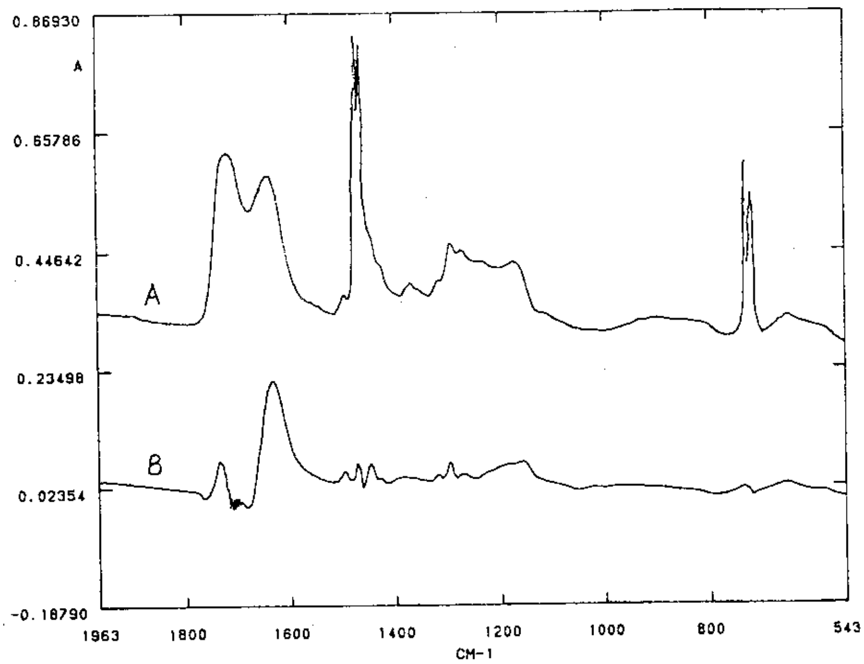

Fig. 3.32 Infrared spectrum (A) of simultaneous co-grafted films obtained from monomer solution (AA:VP=20:30) PEAV134 and differential spectrum (B) obtained by subtraction of AA and VP spectra from A, 134SUBAV (f=0.996) and spectrum (V) of original PE (f=1.007).

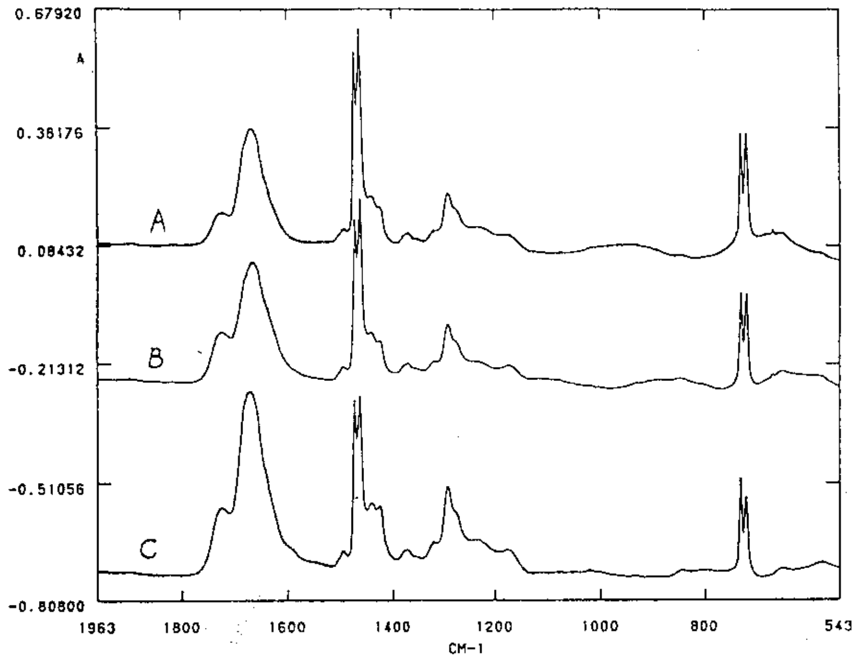

Fig. 3.33 Infrared spectra of PE film grafted with AA followed by the second grafting with VP; G1(AA)/G2(VP) (A) 0.251, PEAV161T, f=1.00, (B) 0.327, PEAV162T, f=1.291, (C) 0.417, f=1.383, PEAV114T.

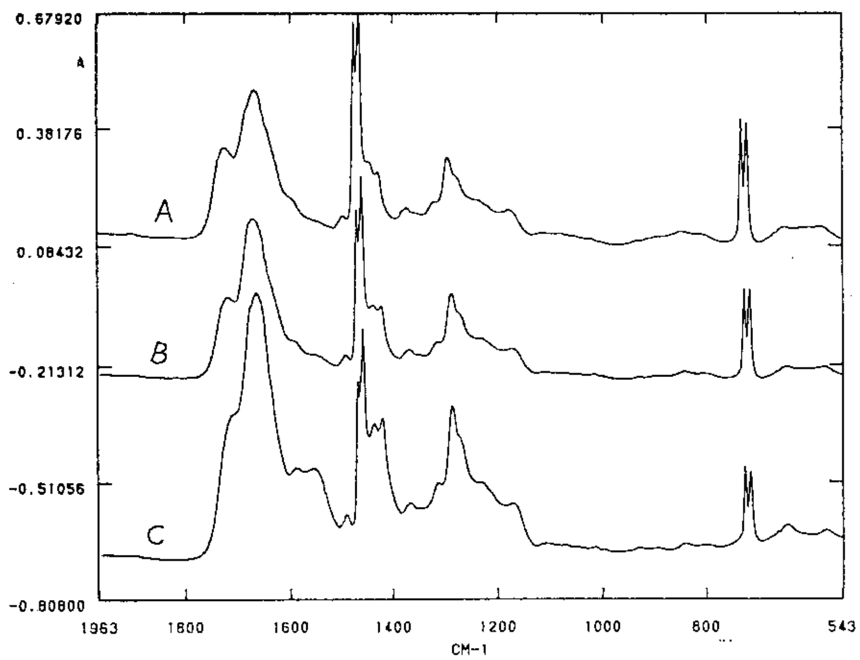

Fig. 3.34 Infrared spectra of PE film grafted with AA followed by the second grafting with VP; G1(AA)/G2(VP) (A) 0.591, PEAV163T, f=1.00, (B) 0.643, PEAV164T, f=1.291, (C) 1.16, f=1.383, PEAV72T.

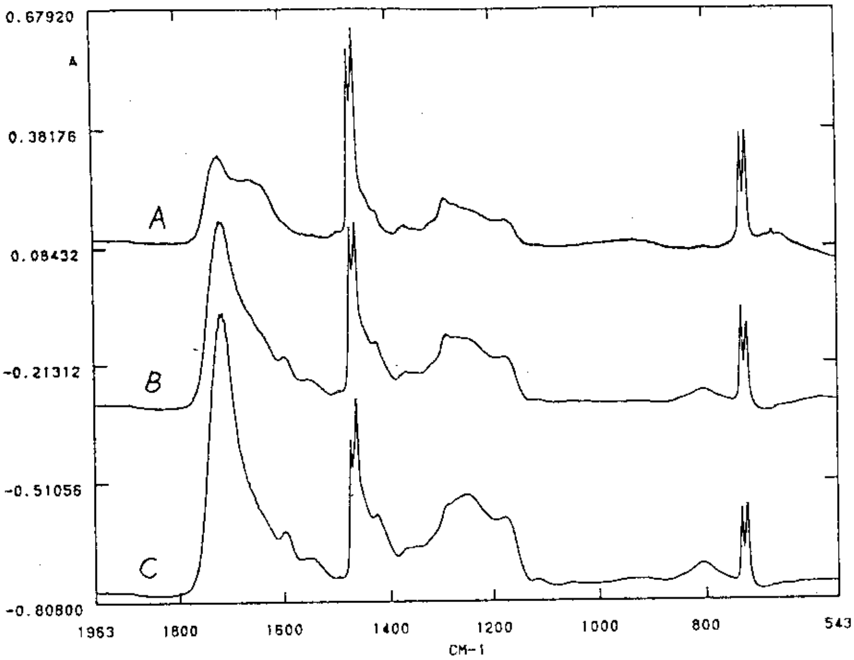

Fig. 3.35 Infrared spectra of PE film grafted with AA followed by the second grafting with VP; G1(AA)/G2(VP) (A) 1.579, PEAV168T, f=1.00, (B) 3.61, PEAV109T, f=1.291, (C) 6.89, f=1.474, PEAV166T.

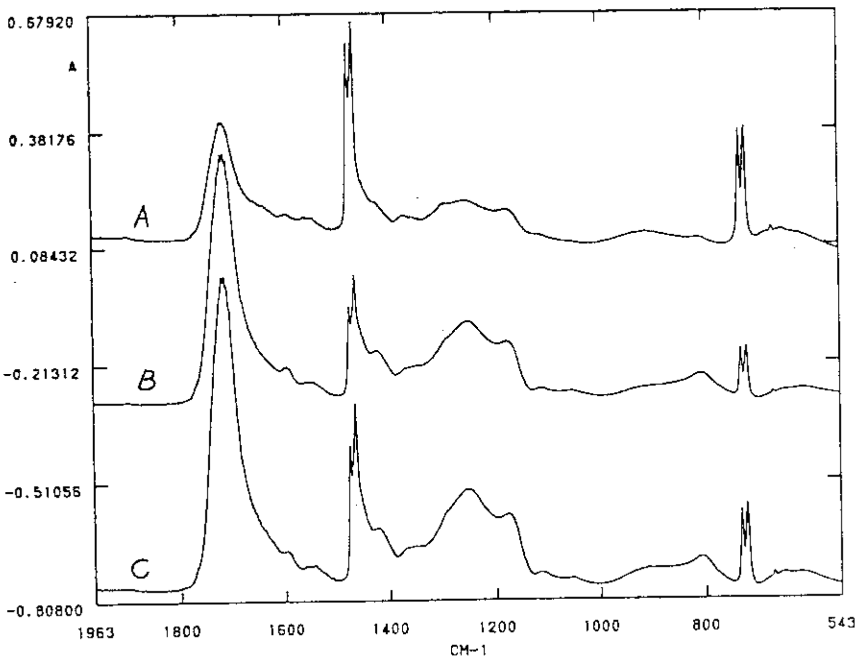

Fig. 3.36 Infrared spectra of PE film grafted with AA followed by the second grafting with VP; G1(AA)/G2(VP) (A) 9.91, PEAV176T, f=1.00, (B) 19.0, PEAV172T, f=2.109, (C) 26.73, f=1.383, PEAV178T.

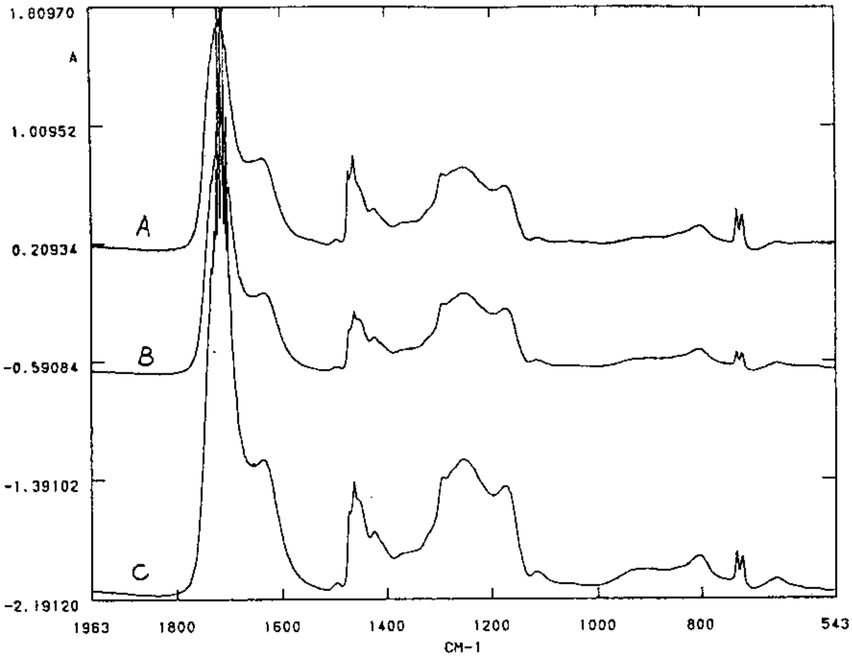

Fig. 3.37 Infrared spectra of PE film grafted with VP followed by the second grafting with AA; G2(AA)/G1(VP) (A) 1.65, PEVA93T, f=1.00, (B) 3.31, PEVA71T, f=1.638, (C) 3.76, f=1.025, PEVA90T.

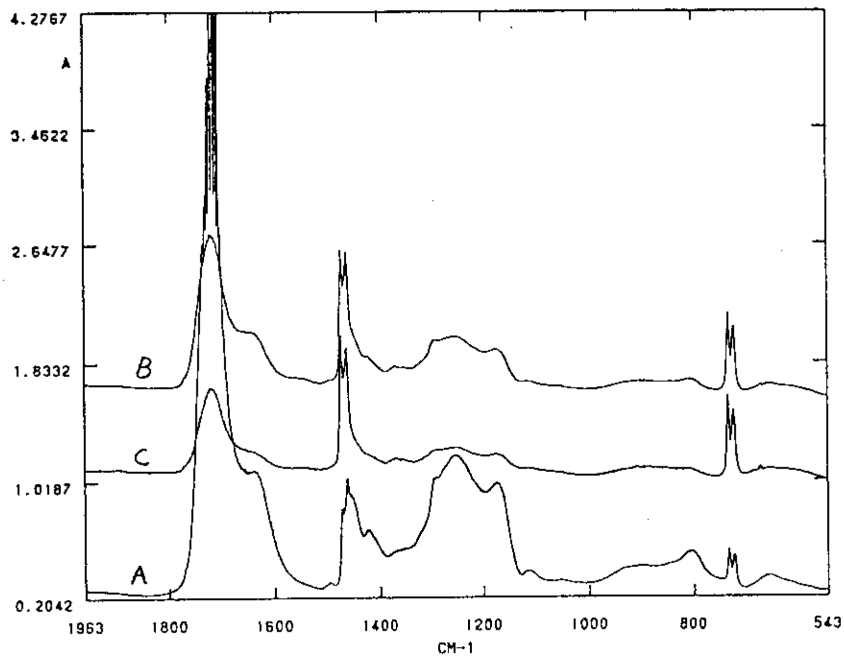

Fig. 3.38 Infrared spectra of PE film grafted with VP followed by the second grafting with AA; G2(AA)/G1(VP) (A) 3.99, PEVA89T, f=1.00, (B) 5.97, PEVA94T, f=0.569, (C) 9.37, f=0.569, PEVA87T.

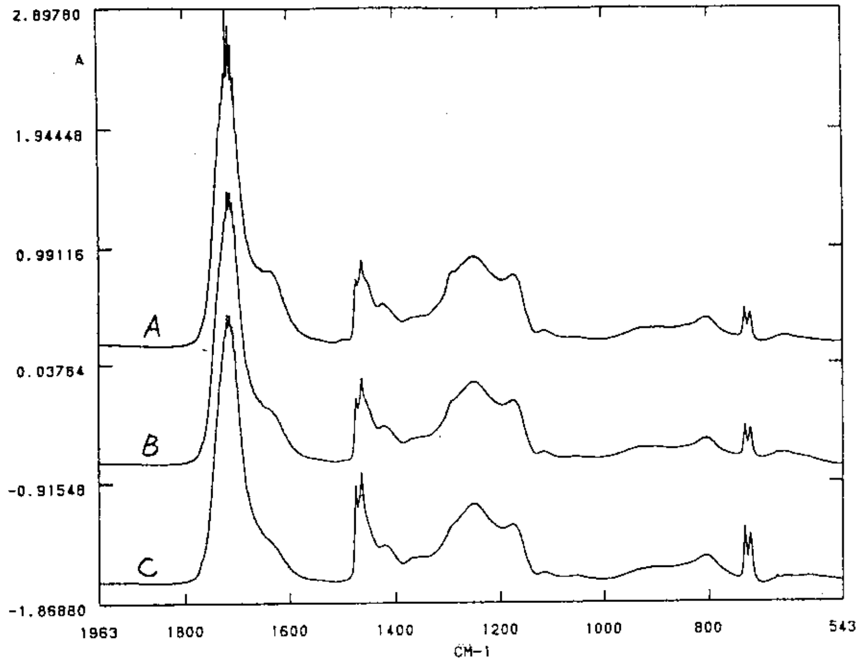

Fig. 3.39 Infrared spectra of PE film grafted with VP followed by the second grafting with AA; G2(AA)/G1(VP) (A) 6.27, PEVA92T, f=1.00, (B) 11.36, PEVA91T, f=0.983, (C) 27.78, f=0.820, PEVA86T.

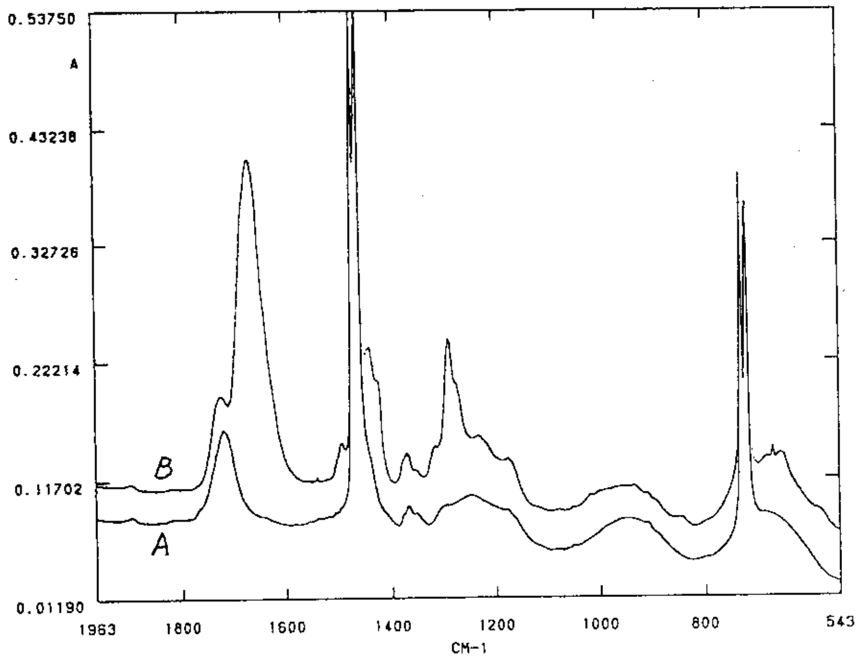

Fig. 3.40 Infrared spectrum of PE film grafted with AA (A) and that of film grafted with VP as the second grafting onto the film previously grafted with AA (B); G1(AA)/G2(VP)=0.251: (A) PEA143, f=1.00, (B) PEAV161T, f=0.994.

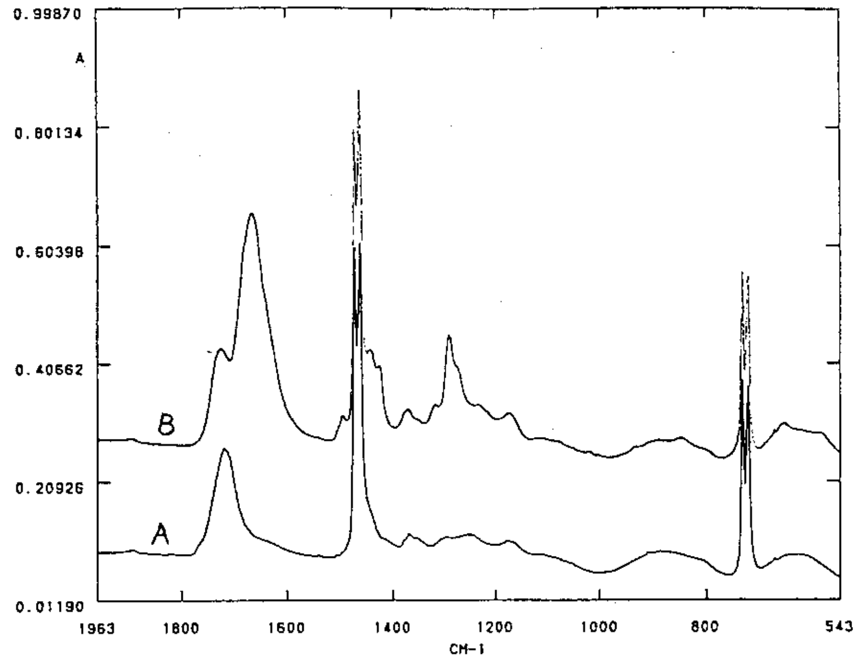

Fig. 3.41 Infrared spectrum of PE film grafted with AA (A) and that of film grafted with VP as the second grafting onto the film previously grafted with AA (B); G1(AA)/G2(VP)=0.327: (A) PEA144, f=1.00, (B) PEAV162T, f=1.006.

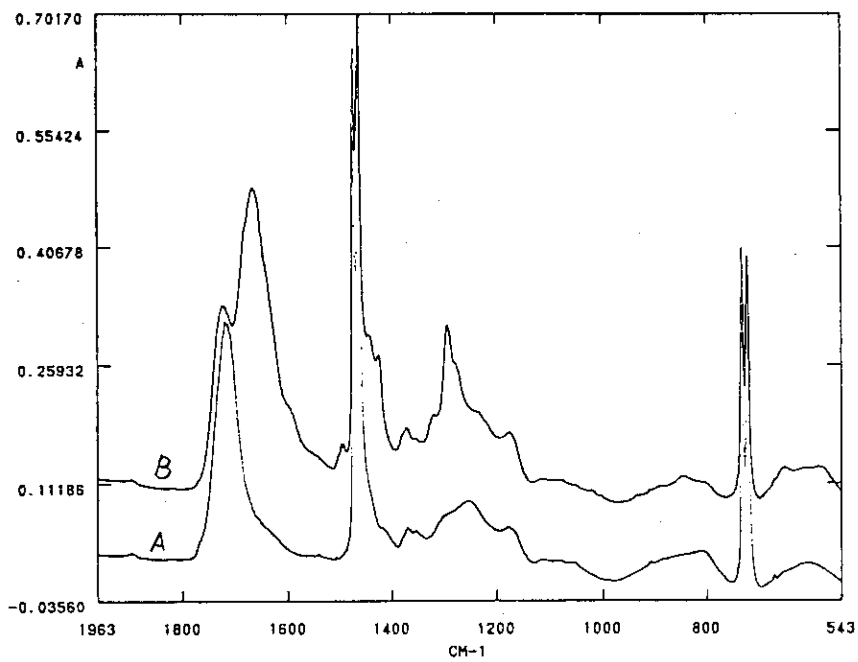

Fig. 3.42 Infrared spectrum of PE film grafted with AA (A) and that of film grafted with VP as the second grafting onto the film previously grafted with AA (B); G1(AA)/G2(VP)=0.591: (A) PEA145, f=1.00, (B) PEAV163T, f=0.993.

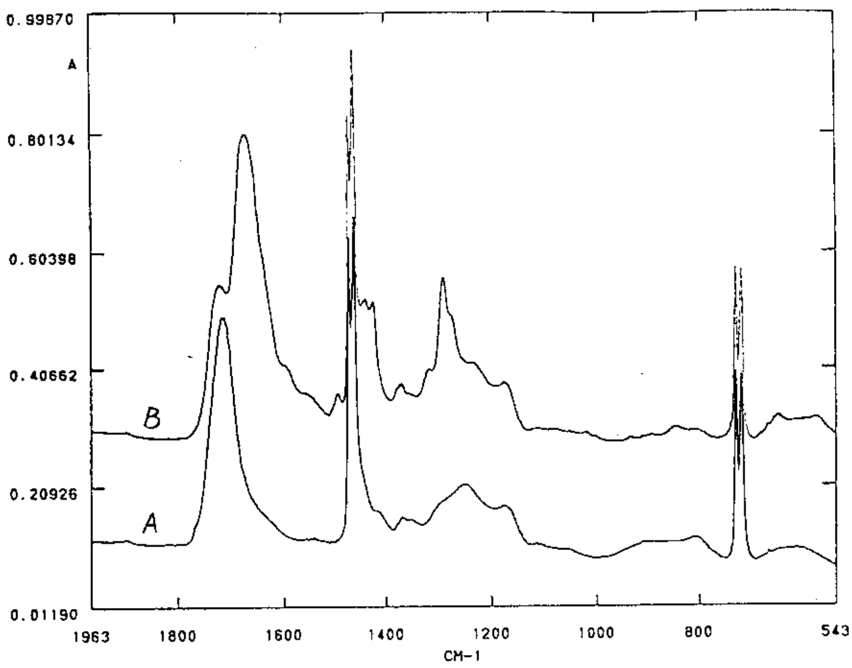

Fig. 3.43 Infrared spectrum of PE film grafted with AA (A) and that of film grafted with VP as the second grafting onto the film previously grafted with AA (B); G1(AA)/G2(VP)=0.643: (A) PEA146, f=1.00, (B) PEAV164T, f=1.006.

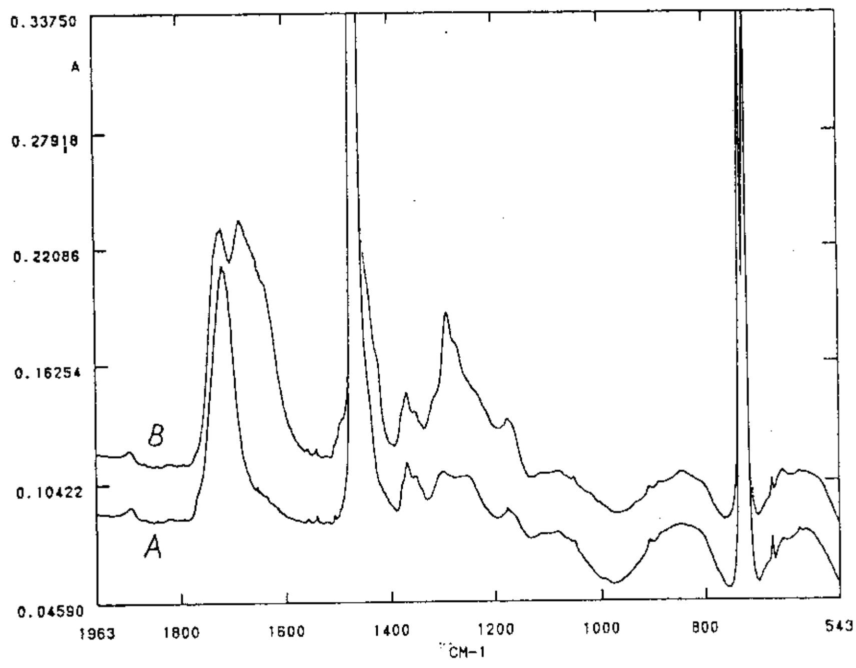

Fig. 3.44 Infrared spectrum of PE film grafted with AA (A) and that of film grafted with VP as the second grafting onto the film previously grafted with AA (B); G1(AA)/G2(VP)=0.984: (A) PEA149, f=1.00, (B) PEAV167T, f=0.989.

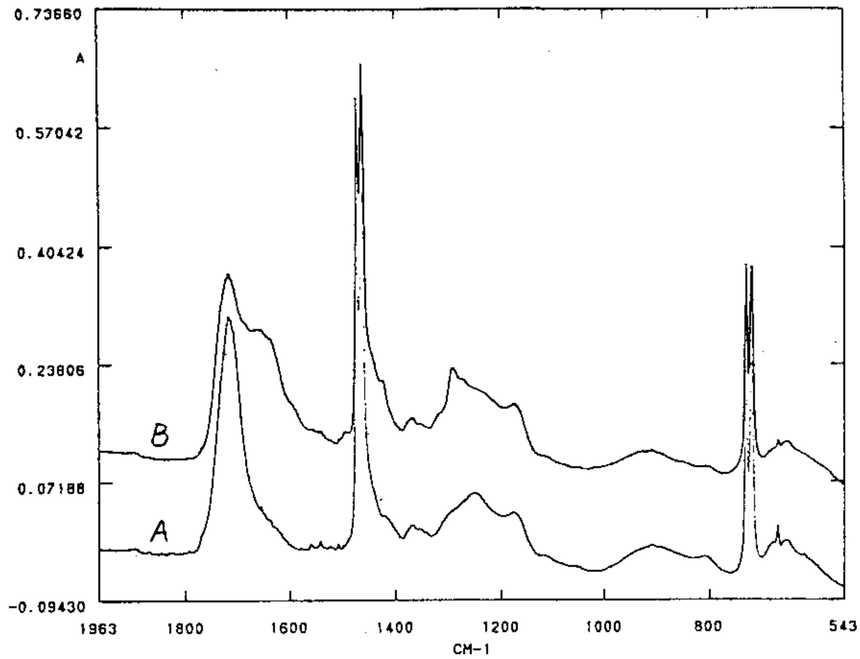

Fig. 3.45 Infrared spectrum of PE film grafted with AA (A) and that of film grafted with VP as the second grafting onto the film previously grafted with AA (B); G1(AA)/G2(VP)=1.66: (A) PEA151, f=1.00, (B) PEAV169T, f=0.995.

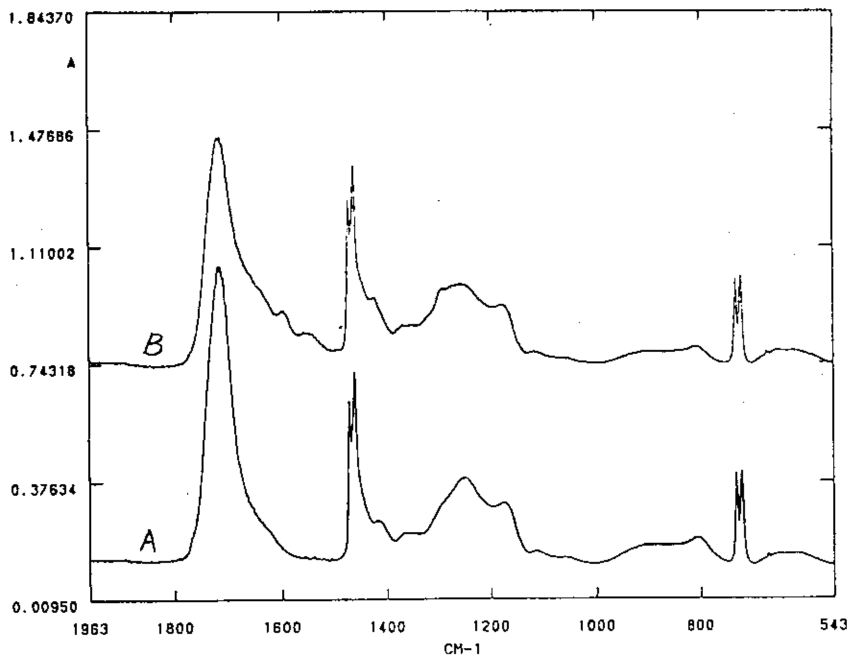

Fig. 3.46 Infrared spectrum of PE film grafted with AA (A) and that of film grafted with VP as the second grafting onto the film previously grafted with AA (B); G1(AA)/G2(VP)=4.89: (A) PEA153, f=1.00, (B) PEAV171T, f=0.989.

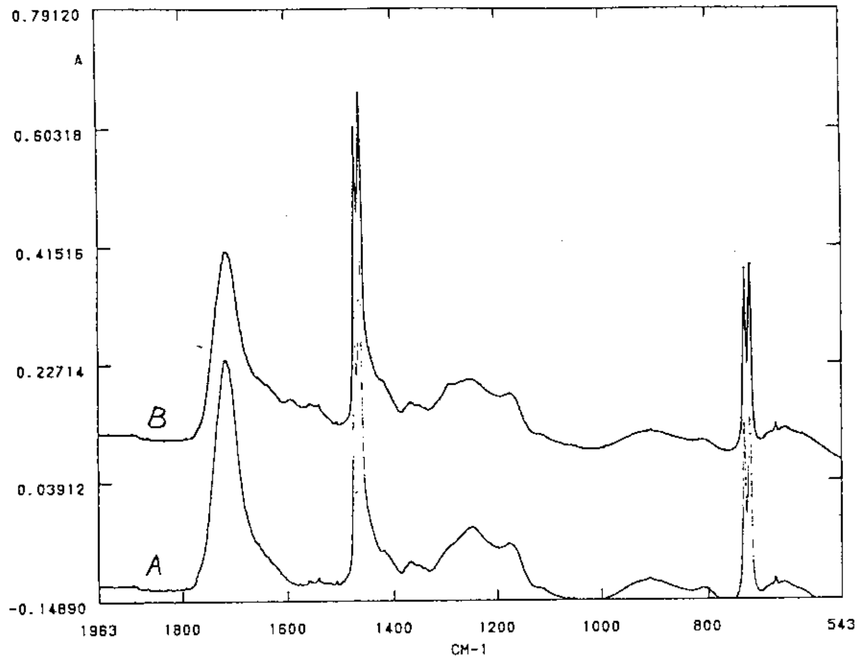

Fig. 3.47 Infrared spectrum of PE film grafted with AA (A) and that of film grafted with VP as the second grafting onto the film previously grafted with AA (B); G1(AA)/G2(VP)=9.91: (A) PEA158, f=1.00, (B) PEAV176T, f=0.985.

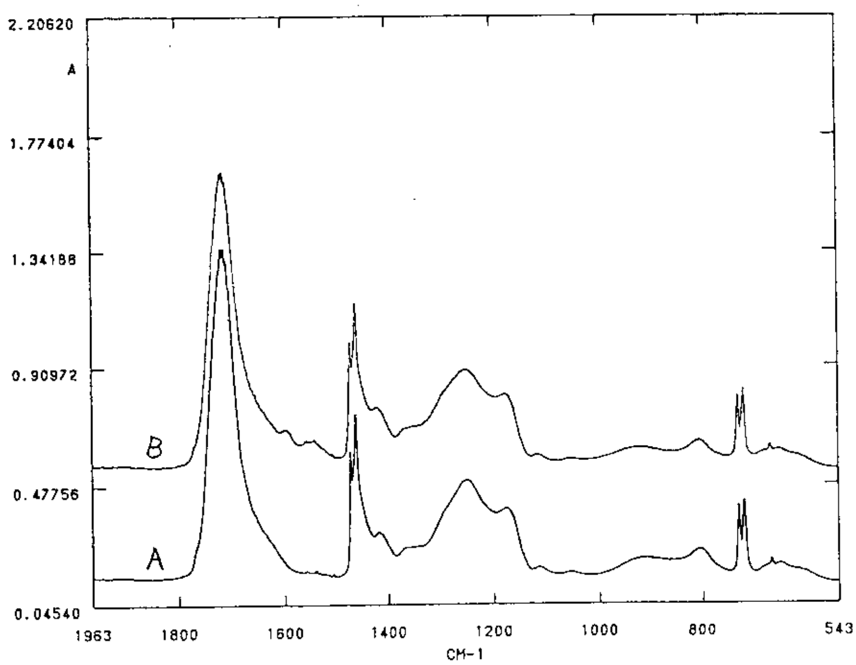

Fig. 3.48 Infrared spectrum of PE film grafted with AA (A) and that of film grafted with VP as the second grafting onto the film previously grafted with AA (B); G1(AA)/G2(VP)=25.01: (A) PEA159, f=1.00, (B) PEAV177T, f=0.999.

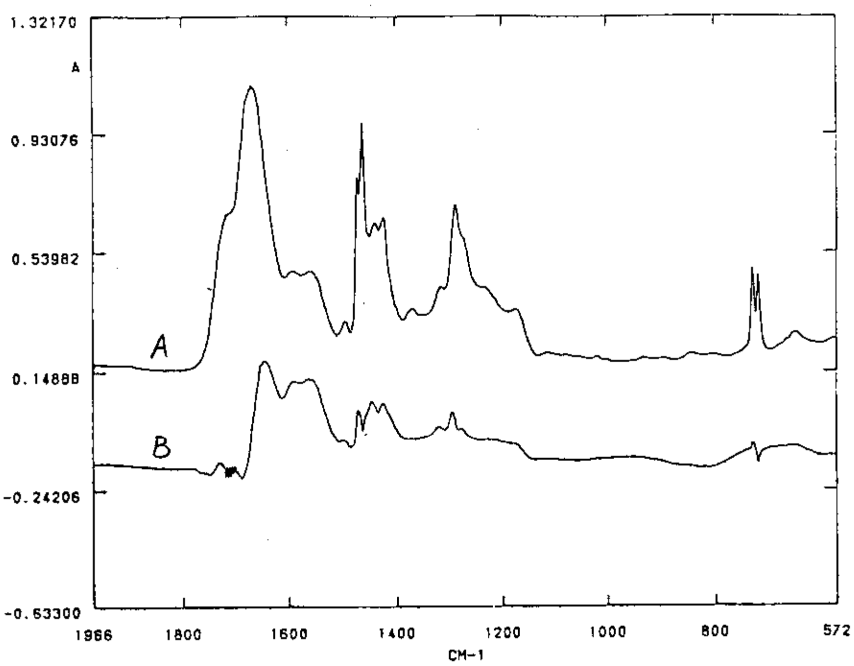

Fig. 3.49 Infrared spectrum of (A) PE film grafted with AA followed by the second grafting with VP; G1(AA)=23.2%, G2(VP)=20.0, PEAV72T, and (B) differential spectrum, 72SUBAV, obtained by subtraction of spectra of AA and VP from spectrum (A); The spectra of AA and VP for subtraction were synthesized in advance by subtraction of spectrum of PE from that of AA-grafted film and from VP-grafted film, respectively. f for (B)=0.998, and (C) original PE (f=0.992).

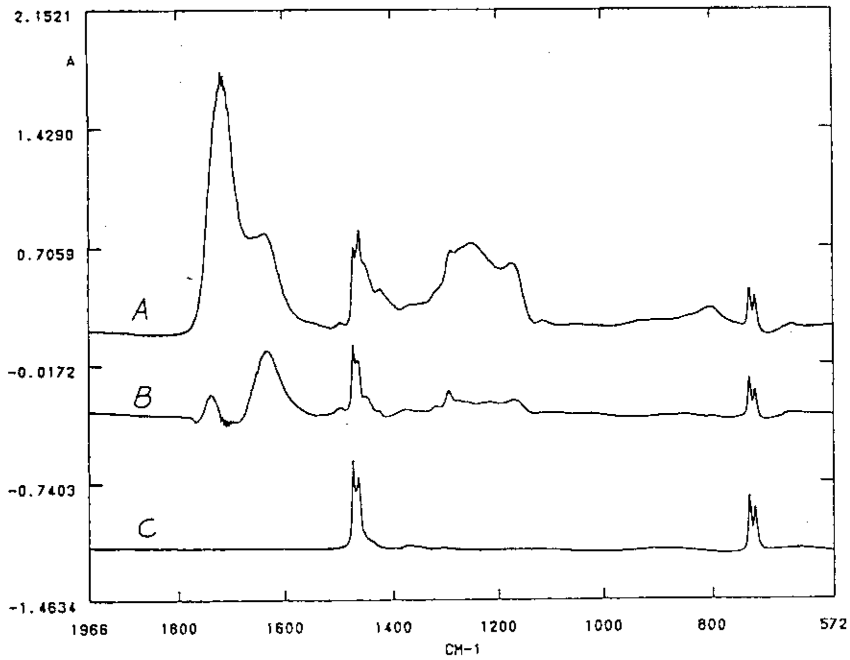

Fig. 3.50 Infrared spectrum of (A) PE film grafted with VP followed by the second grafting with AA; G1(VP)=21.2%, G2(AA)=34.8, PEAV93T, (B) differential spectrum, 93SUBVA, obtained by subtraction of spectra of AA and VP from spectrum (A), and (C) that of polyethylene, PEO; The spectra of AA and VP for subtraction were synthesized in advance by subtraction of spectrum of PE from that of AA-grafted film and from VP-grafted film, respectively. f for (B)=1.004; f for (C)=1.013.

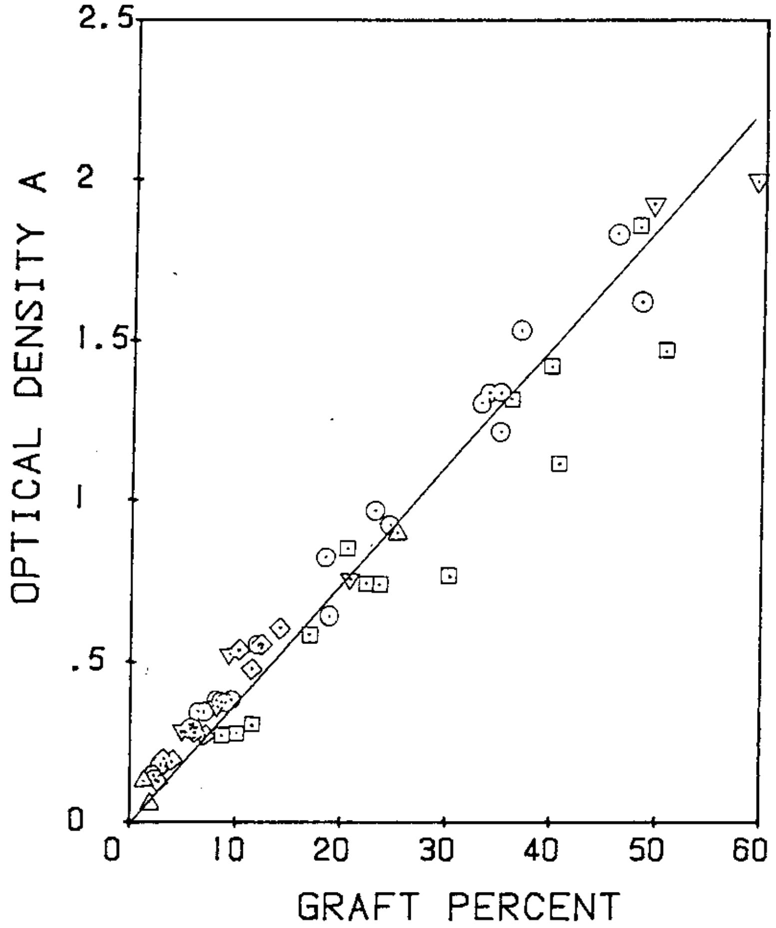

Fig. 3.51 Optical density as a function of graft percent: optical densities are not corrected for expansion of the film due to grafting. ○ AA grafted film, △ VP-grafted film, □ AA-VP grafted, ▽ VP-AA grafted, ◇ simultaneously co-grafted with AA and VP. #57,58, 62,64,69,70,72,75,81,91,94,109,114,119-126, 129,130,132-134,144-148,150-166,168-172, 174-178.

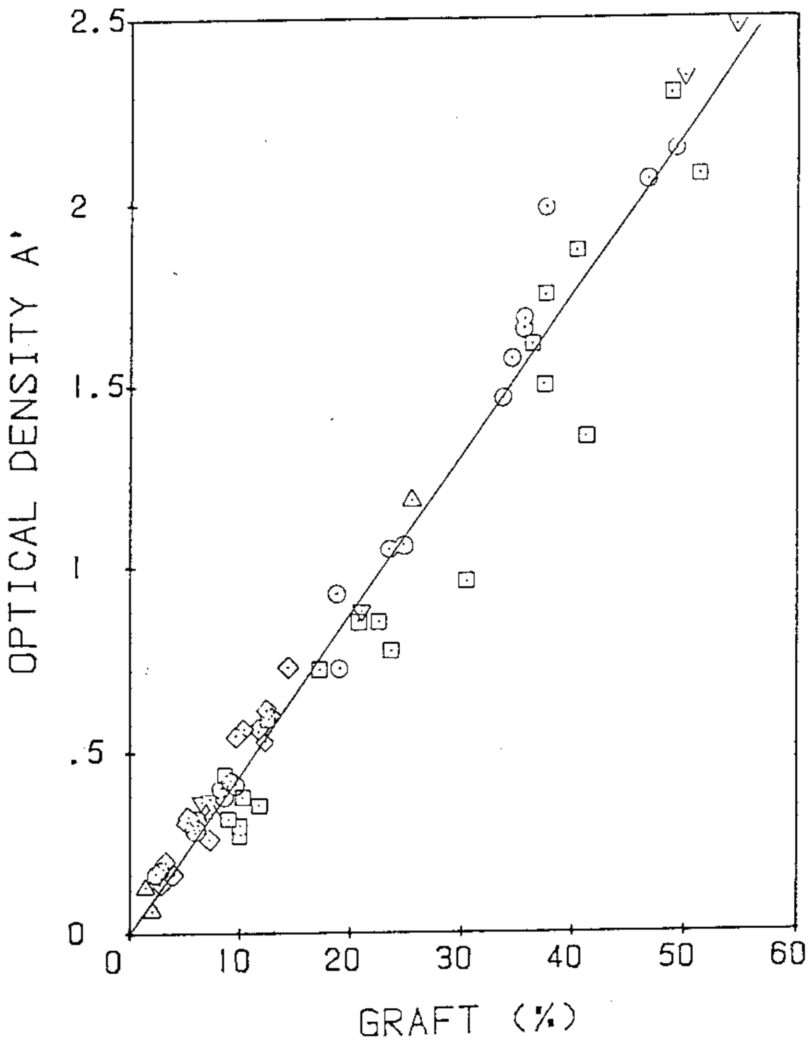

Fig. 3.52 Optical density as a function of graft percent: optical densities are corrected for expansion of the film due to grafting. ○ AA grafted film, △ VP-grafted film, □ AA-VP grafted, ▽ VP-AA grafted, ◇ simultaneously co-grafted with AA and VP. Corrected optical density is given by A'= A(B /B), where A is optical density uncorrected, B is optical density at 723 cm before grafting and B, that after grafting. Samples, the same to those listed in Fig. 8.

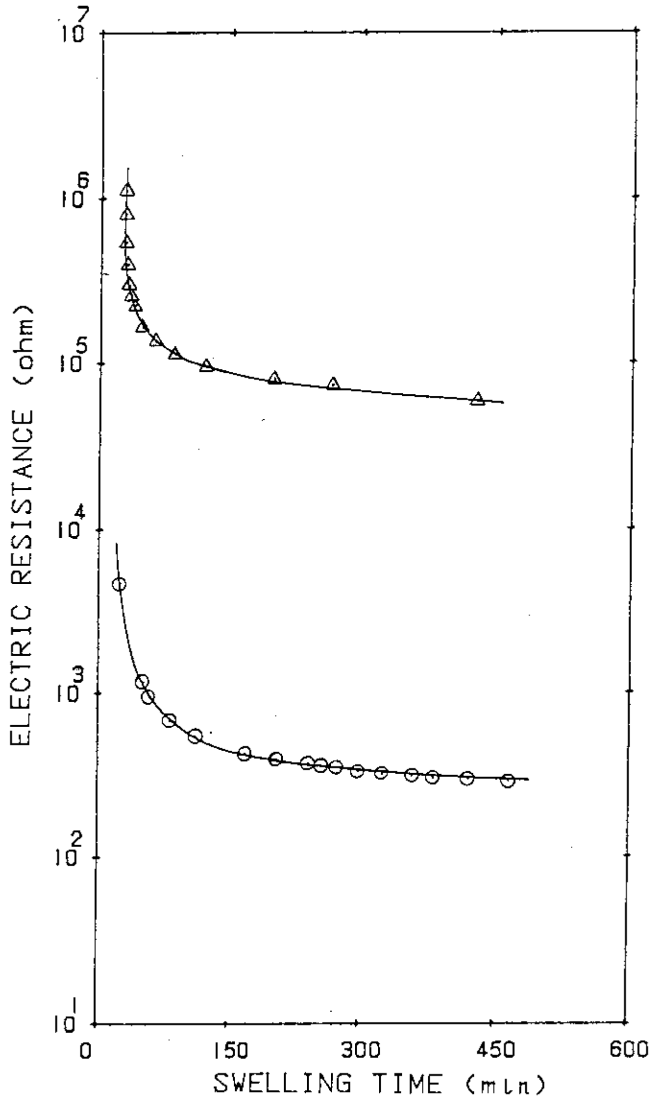

Fig. 3.53 Electric resistance across grafted film as a function of swelling time in 10% KOH aqueous solution; (Δ) #328 100 μm HDPE; dose rate, 1.18×10^5 rad/h, irradiated at 50°C, (O) #336 100 μm HDPE; dose rate, 1.88×10^5 rad/h, irradiated at 50°C; cell resistance was 26Ω.

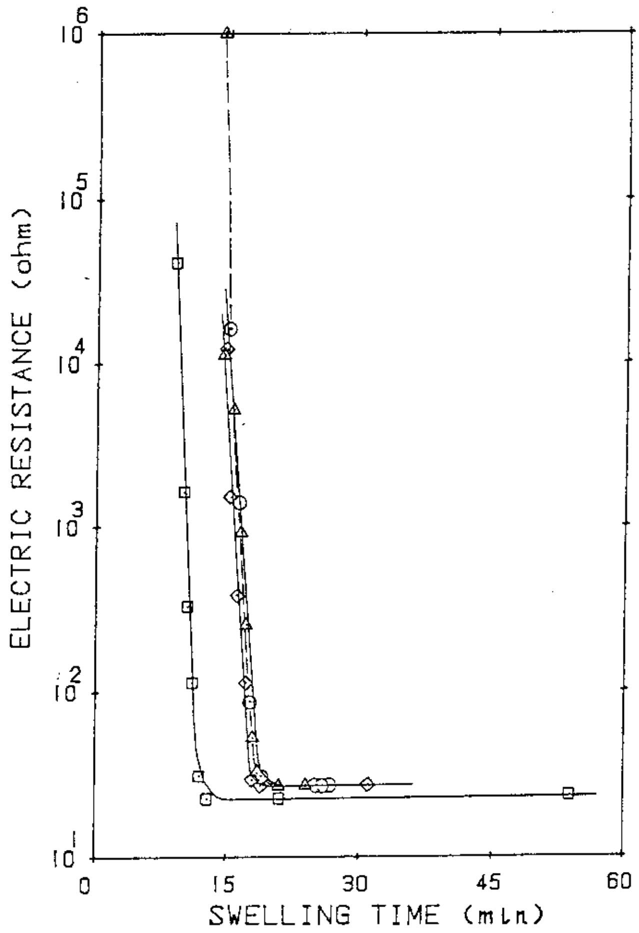

Fig. 3.54 Electric resistance across grafted film as a function of swelling time in 10% KOH aqueous solution; 100 μm HDPE; dose rate, 3.1×10^3 rad/h, irradiated at 50°C, cell resistance was 26Ω; (△) #339 G(AA)=32.1%; (○) #338 G(AA)=26.2%; □ #340 G(AA)=29.2%; × #329 G(AA)=45.5%. Dose rate = 1.18×10^5rad/h; cell resistance was 20Ω.

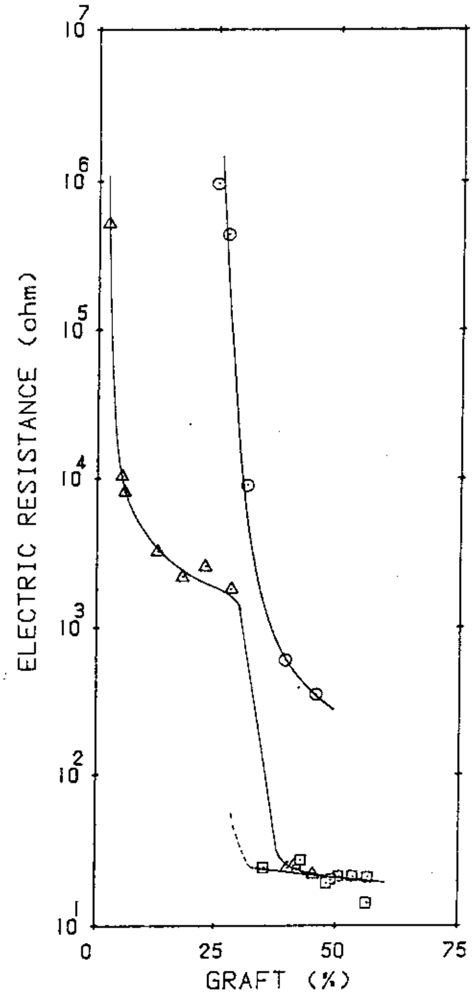

Fig. 3.55 Electric resistance across grafted film as a function of graft percent in 10% KOH aqueous solution; 100 μm HDPE; irradiated at 50°C, cell resistance was 26Ω; (○) grafted with VP, dose rate, 2.62×10^4rad/h, #372,276,282, 285-287; cell resistance, 3.5Ω; (△) grafted with AA, dose rate, 1.12 - 1.18×10^4rad/h; #314,315,326,327,329,341,342,344,345,435,436; cell resistance, 20 - 26Ω; □ grafted with AA, dose rate, 2.66 - 2.77×10^4rad/h, #263-268, 302-304; cell resistance, 20 - 26Ω.

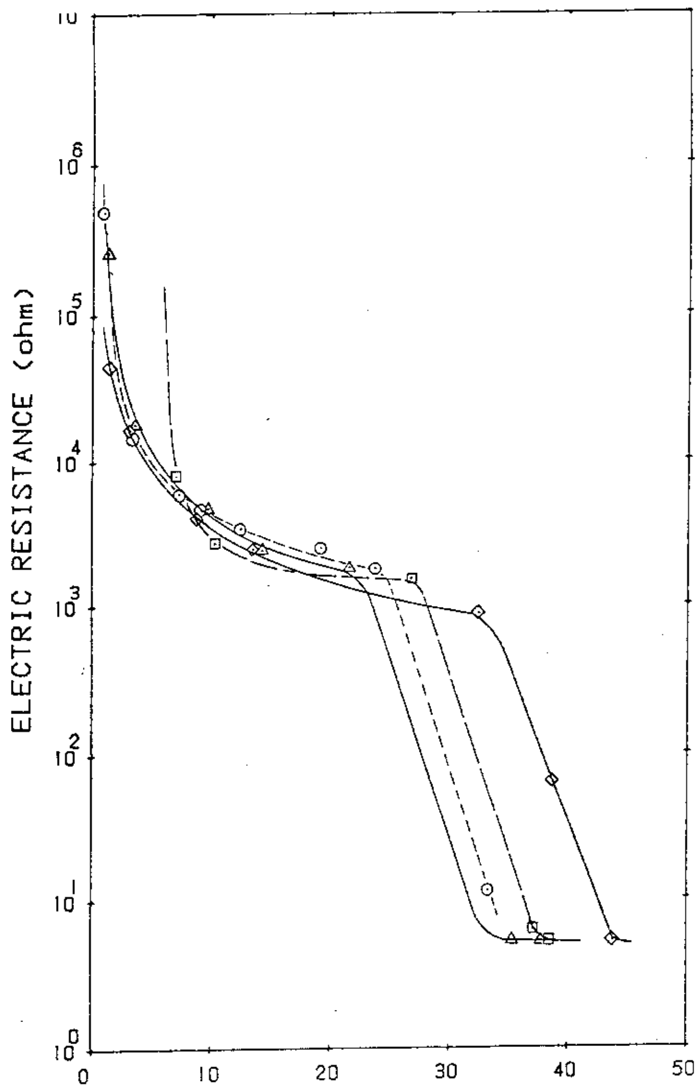

Fig. 3.56 Electric resistance across grafted film as a function of graft percent in 10% KOH aqueous solution--Grafted films were prepared by simultaneous co-grafting method; 100 μm HDPE; irradiated at 50°C, cell resistance was 3.5Ω; AA:VP (○) 20:30, #388-391,418-421; (△) 30:20, #392-395,415-417; (□) 40:10, #396-399,413-414; (◇) 45:5, #400-403,410-412.

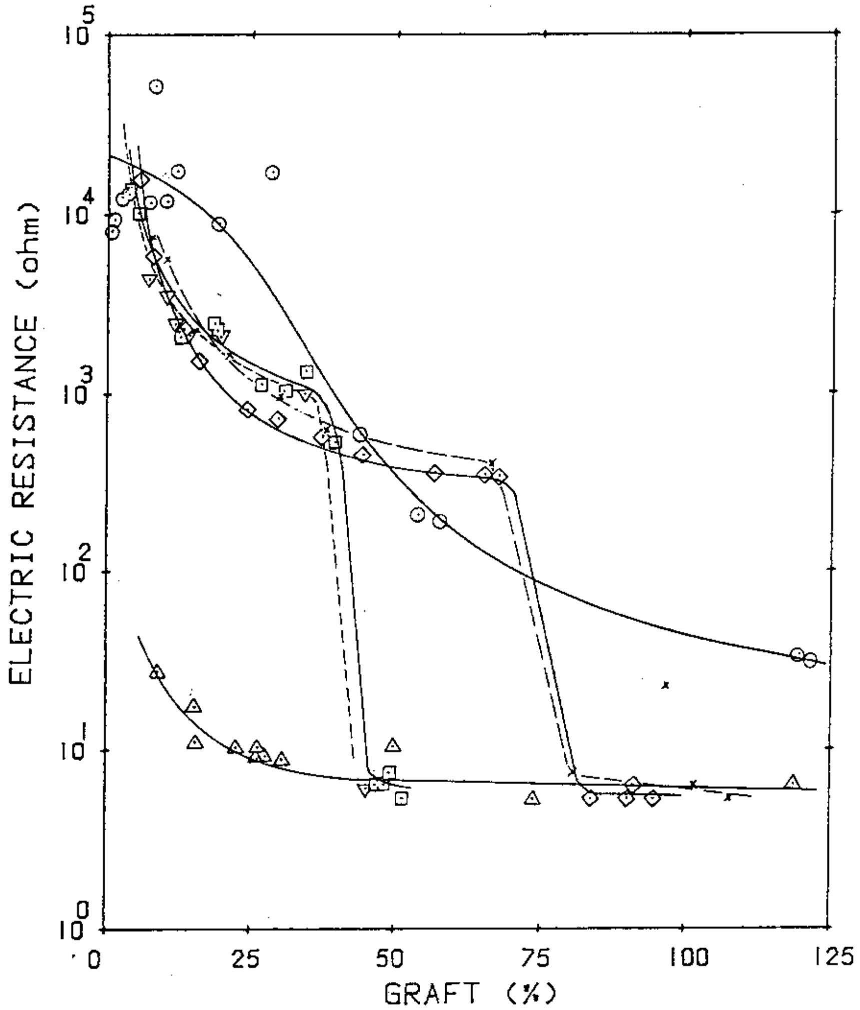

Fig. 3.57 Electric resistance across grafted film as a function of graft percent in 10% KOH aqueous solution--Grafted films were prepared by multi-stage grafting method one after the other monomer; 100 μm HDPE; irradiated at 50°C, cell resistance was 3.5Ω; VP after AA graft (○) Gl(AA)=ca.5%; #430(2-1),432(2-1)-435(2-1),431(3-1),456(4-1),457(2-1),458(4-1), 430(2-2)-434(2-2),456(3-2),457(4-2),458(4-2); AA after VP graft (△) Gl(VP)=0.29-1.07%; #379(2)-382(2),404(2-1)-406(2-1),409(2-1); (□) Gl(VP)=ca.2%; 445(2-1)-448(2-1), 451(2-1)-452(2-1),445(2-2)-448(2-2),451(2-2), 452(2-2); (◇) Gl(VP)=ca.5%; #422(2-1)-429(2-1),422(2-2)-428(2-2); ▽ Gl(VP)=ca.25%, #368(2)-378(2); (×) Gl(VP)=ca.12%; #437(2-1),437(2-2),439(2-1),439(2-2),440(2-1), 440(2-2),441(2-1),441(2-2),443(2-1),443(2-2), 444(2).