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Research Article

Radiation-Induced Graft Copolymerization Ethyl Acrylate onto Poly (Tetrafluoroethylene-co-ethylene) Film by Discontinuous Method

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Abstract

Graft copolymerization of vinyl monomer such as ethyl acrylate, EA, onto poly(tetraflouroethylene-co-ethylene), (Tefzel) film was attempted by double-irradiation method in aqueous medium. Optimum conditions for obtaining maximum percentage of grafting were evaluated for EA. Maximum percent grafting for EA (320.3%) was obtained at an optimum dose of 27kGy, using [EA]=0.027 moles/L. The effect of aliphatic alcohols of different chain length, such as methanol, ethanol, n-butanol, cyclohexanol and amyl alcohol on percent grafting of EA has also been examined. Characterization of Tefzel and grafted Tefzel film has been carried out by IR spectroscopy and thermogravimetric analysis. It has been found that grafted Tefzel film showed better thermal behavior.

Keywords: Pre-irradiation, discontinuous method, optimum dose, percent grafting, modification, functional group, swelling and separation process

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Introduction

Tefzel, although is a tough polymer with properties of polyethylene and poly(tetrafluoroethylene) and lacks in having any functional moieties in the backbone. As a result, its use in various fields, where functional groups are required, is restricted. Different group of workers, however, have successfully carried out modification of Tefzel by the graft copolymerization method, using monomers with different functional groups. A study has been made on the graft copolymers obtained by radiation induced grafting of acrylic acid onto ply(tetrafluoroethylene) films by Hegazy and workers [1, 2]. Grafting is a versatile means to modify polymers was studied by A Bhattacharya et. al [3]. Grafting of styrene into pre-irradiated fluoropolymer film has been studied by U. Lappanet. Al [4]. Omichi and Okamoto [5] studied the grafting of α,α,β trifluoroacrylate onto polyethylene (PE) and fluorine containing films by simultaneous grafting method using γ-rays. These membranes showed good oxidation resistance. Kaur et. al. [6, 7] have studied the effect of different grafting parameters and effect of aliphatic alcohols of varying chain lengths on grafting of acrylonitrile (AN), methacrylonitrile (MAN), methylacrylate (MA) and ethylacrylate (EA) onto Tefzel film by preirradiation method.

Modification of Tefzel film by graft copolymerization of acrylonitrile and methacrylonitrile for use as membrane was carried by I Kaur et al [8]. A study on graft copolymerization of electron donor and electron acceptor monomer mixture onto Tefzel film was studied by I Kaur et al [9]. Modification of poly(tetrafluoroethylene-*co*-ethylene), Tefzel (ETFE), film has been carried out by grafting methylmethacrylate (MMA) by radiation method including preirradiation and double-irradiation methods by Inderjeet Kaur et al [10] with improved thermal stability. The effect of styrene monomer in the graft copolymerization of acrylonitrile onto deproteinized natural rubber was studied by Tuti Indah Sari et al [11]. Graft copolymerization of a binary mixture of methacrylic acid and 4-vinyl pyridine, onto poly(vinyl fluoride) film has been carried out by preirradiation method by Inderjeet Kaur et al [12]. Ionic bifunctional membranes have been synthesized by grafting binary mixture of methacrylic acid (MAAc) and 4-vinyl pyridine (4-VP) onto Teflon-FEP film by pre-irradiation method by Inderjeet Kaur et al [13]. The styrene/methacrylonitrile (MAN) co-grafted membrane exhibits the best performance among the tested radiation grafted membranes [14]. Graft copolymerization of (acrylonitrile-co-*N*-vinylpyrrolidone) AN/NVP binary monomer onto (low density polyethylene) LDPE film was achieved using simultaneous irradiation method by N.M.El-Sawy et al [15]. In the present manuscript, the grafting of EA has been attempted by discontinuous irradiation with a break of 4 hours at room temperature between two successive irradiations to introduce functional moiety such as ester (-COOR) group to the backbone which would affect the properties of the backbone that would be useful in separation process. Percentage of grafting was studied as a function of all the relevant parameters to optimize the grafting conditions.

Materials and Method

Tefzel film(5mil,127µm) was obtained as a gift from E.I Dupont de Nemours, USA. Ethyl acrylate (EA) (Merck), was washed with 0.5% NaOH solution, dried over anhydrous Na_2SO_4 and distilled. The middle fraction was collected and used. All the alcohols were distilled before use. Distilled water was used as the reaction medium.

Double-Irradiation of Tefzel Film

Tefzel film was washed with methanol and dried under vacuum. The film (4cm x 2cm) was irradiated in air from Co^{60} source (Obtained from Bhabha Atomic research Center Bombay, India, Installed at HPU Chemistry Department) housed in a Gamma Chamber-900 at a constant dose rate of 0.52kGy/h for different periods. The irradiated film was left at room temperature for four hours and re-irradiated by the Co^{60} source for the same time as used during first irradiation.

Graft Copolymerization

The doubly irradiated and weighed Tefzel film (4cm x2cm) was suspended in a known amount of binary solvent mixture of water and methanol (1;1, v/v). To the reaction flask a definite amount of EA was added in definite proportions. The reaction mixture was placed in an oil bath maintained at a constant temperature of 100^0C and refluxed for a specific time period. After the stipulated time, the film was removed from the reaction mixture, washed thoroughly with appropriate solvent i.e. acetone for Poly (EA) for the complete removal of the homopolymer formed during the reaction. The grafted Tefzel film free from homopolymer was dried and weighed. Percentage of grafting was determined from the percent increase in the initial weight of the film in the following manner:

$$\% \text{ Grafting} = (W_1 - W_0)/W_0 \times 100$$

Where W_0 and W_1 are respectively the weights of original and grafted films after the complete removal of the homopolymer formed during the reaction.

Results and Discussion

Irradiation of Tefzel film was carried out in air discontinuously at a constant dose rate of (0.52 kGy /h) for a particular period then removed from the gamma chamber, left at room temperature for a specific time period (4 hrs) and then again re-irradiated with gamma rays for the same time as given in the first irradiation. The doubly irradiated film was used as the backbone polymer. Irradiation in air, introduces hydroperoxide groups in the Tefzel film following the cleavage of the C-H bonds. The formation of these groups along with some alcoholic groups on the surface of Tefzel film upon irradiation with gamma rays were observed by Singh and Prasad [16]. The hydroperoxide groups thus formed, offer sites for grafting when polymerization of the monomer is carried out in its presence. The purpose of double irradiation is to generate more hydroperoxide groups since, during double irradiation the radicals trapped in the bulk of the film are also converted in to hydroperoxide groups. The time interval given between the two irradiations may help the embedded radicals to interact with oxygen or diffuse towards the surface of the film. Thus, double irradiation method seems to increase the number of hydroperoxide groups on the surface as well in the bulk of the film. This is expected to improve the overall percentage of grafting. Following plausible mechanism for grafting of EA onto doubly irradiated Tefzel film is proposed **Figure 1**.

Thus, the formation of graft copolymer can take place either via (eq. 5) or (eq. 6) involving grafting from and grafting onto process respectively. However, under preirradiation conditions when grafting is carried out thermally onto preirradiated backbone, process (5) is less favourable. The hydroxyl radical formed upon decomposition of the hydroperoxide is very reactive to initiate the polymerization of the added monomer to give the growing radicals which attach themselves to the macrooxy radical to produce graft. Thus, In the former case, monomer gets polymerized directly on the macrooxy radical giving the graft copolymer while in the latter case the initiation of monomer occurs by hydroxyl radical (eq. 4) and growing the polymeric chain thus formed react with the macrooxy radical to produce the graft. Thus, grafting is presumed to take place via process (6). The formation of graft copolymer is influenced by different reaction parameters and in order to optimize the conditions, grafting of EA was studied as a function of these parameters.

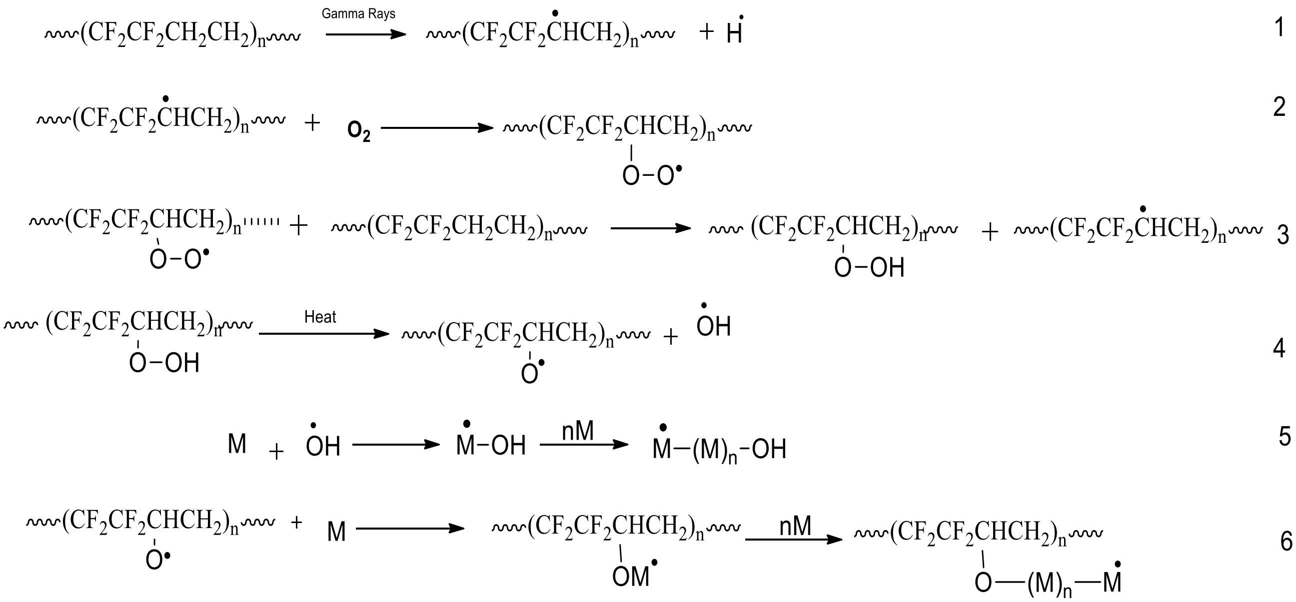

Figure 1 Mechanism of grafting of EA onto Tefzel film

Evidence of Grafting

IR Spectral Analysis

IR spectra of Tefzel-g-poly (EA) has been taken on Beckman Spectrophotometer (**Figure 2**). It was observed that the grafted films shows broad peaks in the range between 3000-2800 cm^{-1}due to associated hydroxyl and hydroperoxide groups. The peaks at 1720 cm^{-1} is due to >CO group of poly (EA). The absence of these peaks in the ungrafted Tefzel film confirms the formation of the graft.

Thermo gravimetric Analysis

Thermo gravimetric analysis (TGA) of Tefzel and Tefzel-g–poly(EA) film was carried out on Schimatzu Simultaneous Thermal Analyzer in air at a heating rate of 10^0C/min. The primary thermogram are presented in **Figure 3**, and **Figure 4** respectively.

The initial decomposition temperature (IDT), final decomposition temperature (FDT) and decomposition temperature (DT) at every 10% weight loss is presented in **Table 1**.

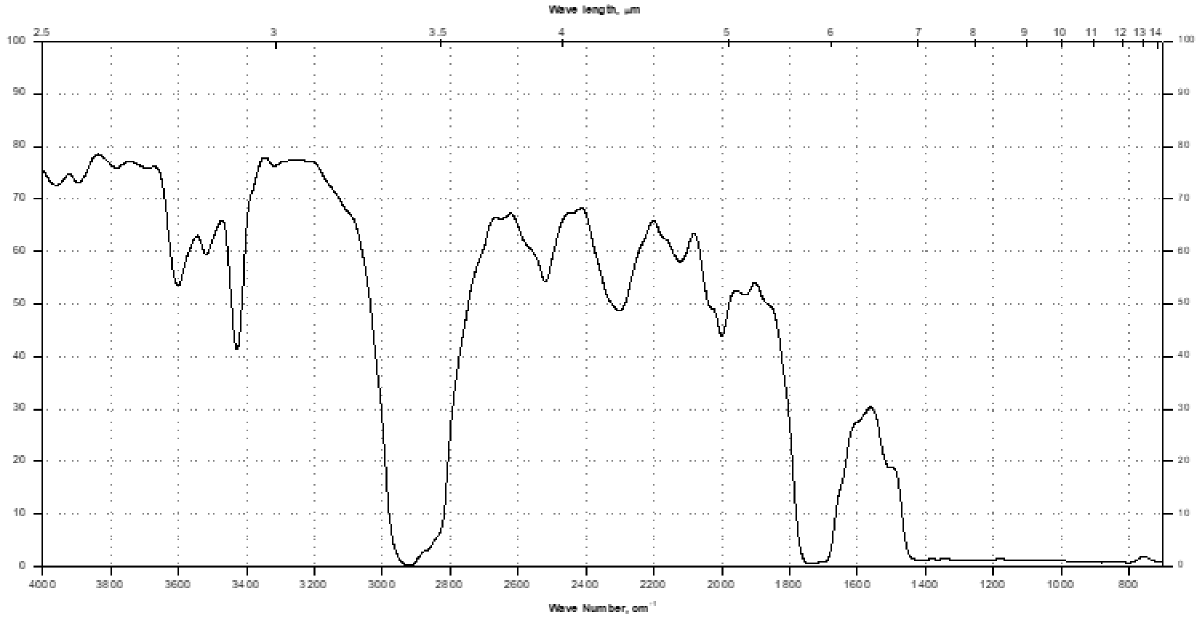

Figure 2 IR Spectra of TEFZEL-g-POLY(EA)

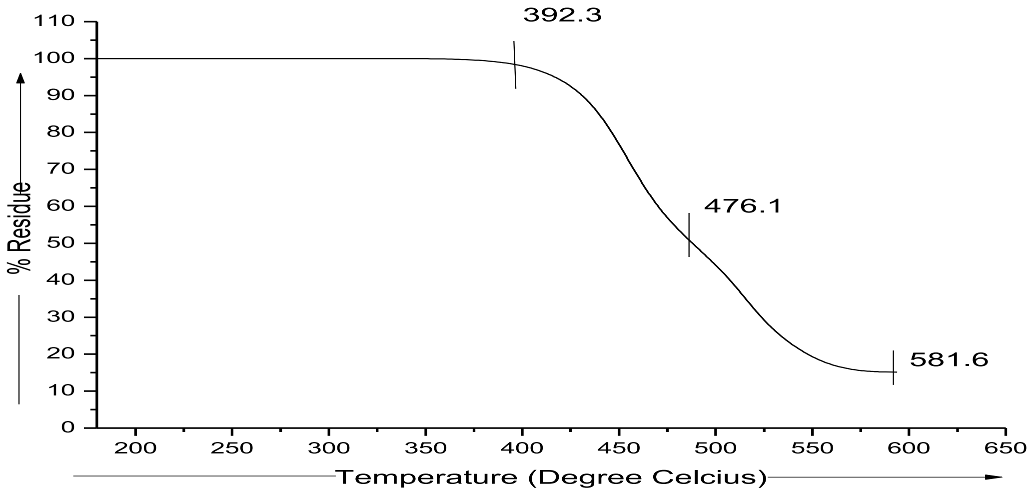

Figure 3 Primary Thermogram of TEFZEL Film

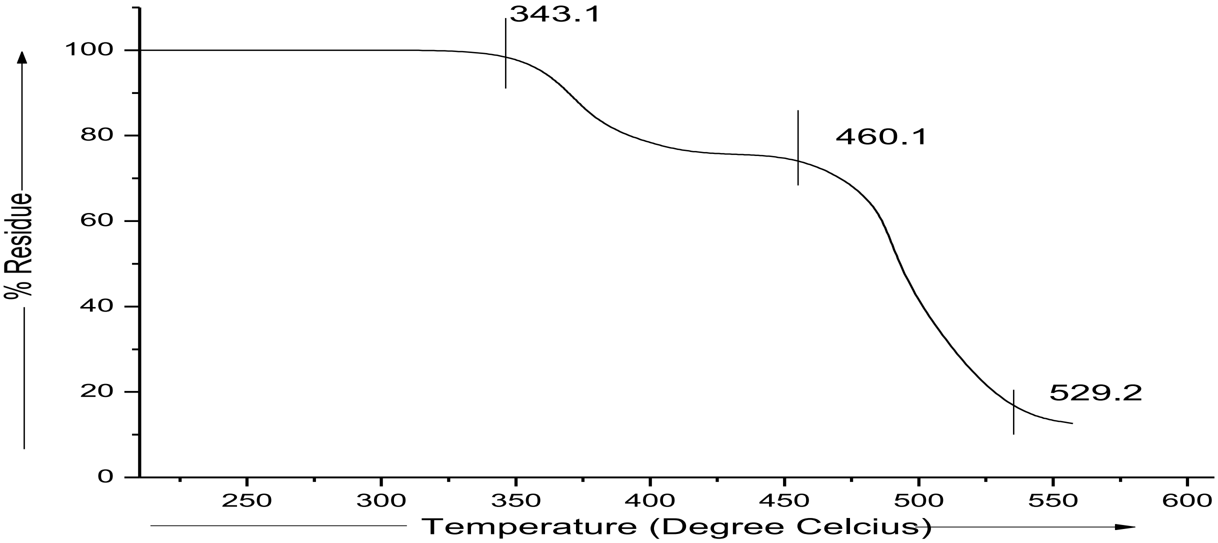

Figure 4 Primary Thermogram of TEFZEL-g-POLY (EA)

Table 1 Thermo gravimetric Analysis of Tefzel Film and Tefzel- g-poly (EA)

Sample	IDT (^{0}C)	FDT (^{0}C)	DT (^{0}C) at every 10% weight loss (%)									
			10	20	30	40	50	60	70	80	90	Residue left (%)
Tefzel Film	392.3	535	425.6	440.2	458.5	469.5	476.8	484.1	493.3	510	-	12.7
Tefzel-g-poly (EA)	343.1	529.2	379.3	410	439	489.9	501.5	516.6	516.6	529.2	-	15.2

The initial decomposition temperature (IDT) ($392,3^0$C) of the original film is much higher than that of Tefzel-g-poly(EA) (343.1^0C).The FDT values (529.2^0C) of EA grafted film is not much less than that of the original film (535^0 C). It is observed from Table 1 that the decomposition temperature of Tefzel-g-poly(EA) shows lower DT values up to 30% weight loss and above that much higher values are observed as compared to the original film. The left-over residue is also higher for EA (15.2%) as compared to the original ungrafted film (12.7%). It is observed from the primary thermograms that while Tefzel film shows single stage of decomposition and the difference in the temperature between every 10 % weight loss is within the range $7\text{-}18^o$C while Tefzel-g-poly (EA) film the temperature difference between 10-20%, 20-30% and 30-40% lies between 30.7^0 C, 28^0C and 50^0C respectively. Beyond these values the temperature difference between DT values for the region of all the grafted films between 60-80% weight losses, lies almost within the same range as that of the original film, although the DT values for the grafted film is much higher than those of the ungrafted film. From these observations, it is observed that the grafting of vinyl monomer decreases the initial decomposition temperature (IDT) of the film due to the reason that the grafted chains are affected in preference to the backbone polymer. The temperature difference of > 30^0C between 10-20% weight loss in the EA grafted films as compared to <20^0C of ungrafted film indicates thermal stability is gained due to grafting. Higher DT values beyond 40% weight loss of the grafted films are because of anhydrides formed during the degradation of polyethyl acrylate grafted chain.

When the thermal data of EA grafted on to doubly irradiated Tefzel film is compared to that EA grafted materials prepared by preirradiation method [7], it is observed that the IDT values of doubly irradiated grafted film has increased from 251.7^{0}C to 343.1^{0}C with not much difference in the FDT values (529.2^0 as against 523.2^{0}C for EA graft). The DT values for every 10% weight loss for doubly irradiated EA grafted film is also higher than those for the grafted film prepared by preirradiation method[7]. However, when the temperature difference between each 10% weight loss is noticed, in the initial decomposition up to 40% weight loss for EA grafted film (double irradiation) the temperature is much higher than that observed in single irradiated EA grafted film. This may be due to the fact that the grafted chain in the doubly grafted EA film is not only confined to grafting on the surface, but it occurs in the bulk of the film also. This will result higher temperature difference. However, beyond 40% weight loss the temperature difference between every next 10% weight losses for doubly irradiated grafted film becomes almost equal to that of the ungrafted film. However, much higher temperature difference above 40% weight loss is observed for single stage irradiated EA film. This difference in temperature behavior at higher weight losses may be due to the fact that upon double irradiation, as discussed previously, the hydroperoxide groups are also formed in the bulk of the film where grafting occurred, and this would disturb the crystallinity of the backbone polymer. The second stage of decomposition corresponds to the backbone polymer which decomposes at a higher temperature with a lower temperature difference because of loss of crystallinity.

Area Change in Grafted Film

It was observed during grafting of (EA) under different reaction conditions that the film expanded in size following increase in grafting percentage. The area change caused by grafting was calculated by the following equation:

$$\text{Percentage area change} = (A_1 - A_0) / A_0 \times 100$$

A_1 and A_0 represent the area of the grafted and ungrafted film respectively. Change in the size of the film has been observed as a function of percentage grafting and the results are presented in **Table 2** and **Figure 5** for grafted EA.

Table 2 Area Change During Grafting of EA on to Tefzel Film

Sr. No.	Sample	Percentage Grafting	Area (cm^2)	Percentage of Area Change
1	Tefzel	0.0	8.0	0.0
2.	Tefzel-g-poly(EA)	139.30	17.10	113.75
3	Tefzel-g-poly(EA)	210.60	19.53	144.12
4	Tefzel-g-poly(EA)	245.50	21.78	172.25

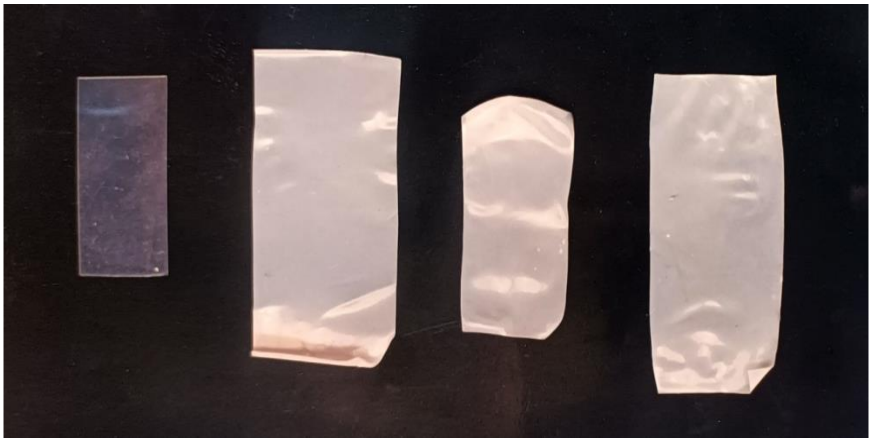

Figure 5 photographs of Tefzel-g-Poly EA film

It is observed from the Figure 5 that the size of the grafted Tefzel film increases gradually with the increase in percentage of grafting but the geometric form remains the same. It indicates that the grafting occurs on the surface of the film and then proceeds with the diffusion of the monomer in to the bulk of the film, thus increasing the area of the film. Figure 5 reveals that the percent area of EA continuously increases with increasing percentage grafting. When these plots are compared with the percent area change of EA (preirradiation method) [7] a significant difference in the trend is observed. The percent area change as against the percent grafting of EA almost remains the same. This difference in the trend for the monomer may be due to difference in the grafting conditions. In the preirradiation method, the reactivity of the monomer is discussed on the basis of its physical properties. The same is true here but with the difference that the backbone is doubly irradiated with the time gap of four hours between two irradiations. Thus, double irradiation process might have formed more of hydroperoxide groups also in the bulk of the polymer. The formation of these groups can induce diffusion of the monomer and the polymeric chains into the bulk of the film. This would produce higher percentage of grafting, and this has been observed. As for the difference in the trend of the plots, since EA remaining more in the liquid state would polymerize and diffuse or penetrate at a faster rate because of the availability of more of the hydroperoxide groups as compared to that in the preirradiation method involving single irradiation.

Swelling Behavior

In order to study the difference of behavior of original Tefzel film, Tefzel-g-poly (EA) towards different polar and non-polar solvents, grafted films of known weight were immersed in different solvents at room temperature for 24 hours. The films were removed, blotted quickly with absorbent paper to remove the liquid adhering to its surface, and weighed immediately. The degree of swelling was calculated as:

$$\text{Percent swelling} = (W_s - W_g)/W_g \times 100$$

W_S and W_g represents the weights of wet and dry grafted film respectively. Percent swelling has been studied as a function of percentage of grafting and the result is presented in **Figure 6** for grafted EA.

Tefzel film did not show swelling behavior due to its compactness whereas Tefzel-g-poly (EA) shows swelling behavior in all the solvents except water which may be due to the bonding interactions of the solvents with the pendant groups (ester) attached to the backbone due to grafting or there might be some interactions of the solvents with the ethylene and the fluorinated segments of the backbone film. Maximum swelling of the grafted film is observed in DMF a polar solvent may be due to more interactions of this solvent with the polar groups (ester) of the grafted film. Benzene since, a non-polar solvent shows higher swelling than acetone a polar solvent may be due to more interactions of benzene with the ethylene segment of the backbone film. Water, though maximum polar solvent but shows least swelling behavior could be due to non-interactions of water with the pendant groups of the grafted film and non-penetration into the grafted film due to strong intermolecular H-bonding in water molecules itself which might have unable them to interact as well penetrate into the grafted film.

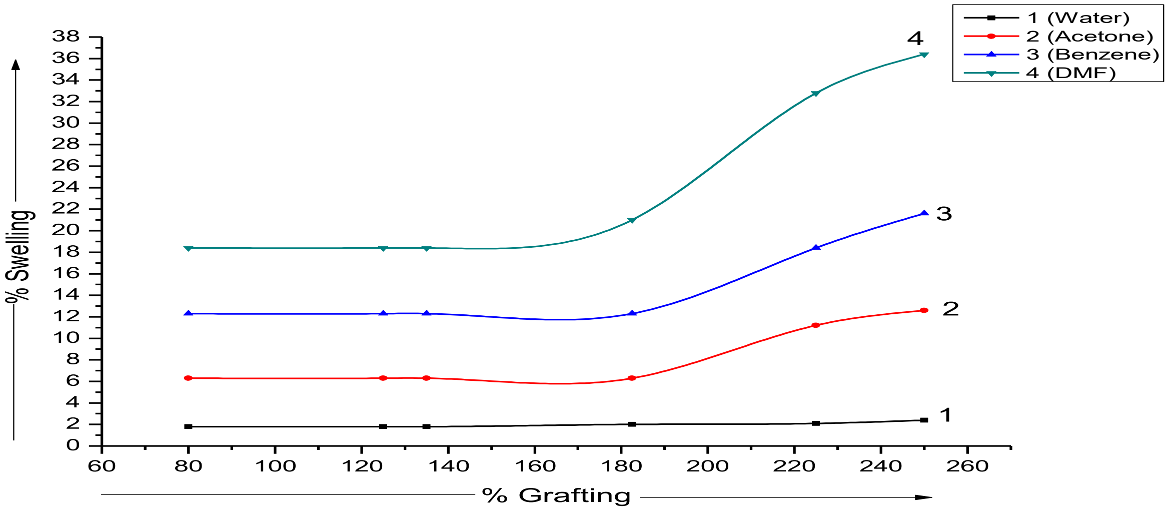

Figure 6 The effect of grafting on swelling of TEFZEL-g-POLY (EA) in different Solvents

Water, though maximum polar solvent but shows least swelling behavior could be due to non-interactions of water with the pendant groups of the grafted film and non-penetration into the grafted film due to strong intermolecular H-bonding in water molecules itself which might have unabled them to interact as well penetrate into the grafted film.

Percent swelling of Tefzel film grafted with EA monomer in different solvents except water, increases with increase in the percentage of grafting. In lower ranges of percentage of grafting up to 182.5%, the percent swelling is not much affected beyond these values, a sharp increase in swelling is observed for EA grafted films in DMF, acetone, benzene. In all the grafted films, the swelling behavior is same. Different solvents showed the following order towards swelling.

Tefzel-g-Poly(EA) : DMF > Benzene > Acetone > Water

The pattern of swelling is same as observed for these monomers grafted by single irradiation method as was studied by KAUR ET AL[7].

Effect of Total Dose

Percentage of grafting of EA on to Tefzel film has been carried using different total doses and the effect of the variation of total dose on percentage of grafting has been evaluated. The results are presented in **Figure 7** The Tefzel film was irradiated twice with a break of four hours between two irradiations. The total dose given is taken as the sum of total of the total doses of both the irradiation periods. It is observed from the Figure 7 that the percent of grafting for EA shows initial smooth rise in grafting but the trend changes thereafter. There is initial steady increase in P_g giving 245.5% at a total dose of 27.0 kGy and falls sharply to 83.8% at a total dose of 29.1 kGy. Further increase in total dose again raises the P_g sharply followed by falls and rise again. The difference in behavior towards grafting may again be attributed to their difference in the physical property. Since, EA has lower vapour pressure, means EA tends to remain more in the liquid state under the given reaction conditions consequently, EA is more accessible to the reaction sites on the backbone resulting in higher P_g. The diffusion of the monomer into the bulk of the film or the crystalline region is also affected by the physical state of the monomer EA.

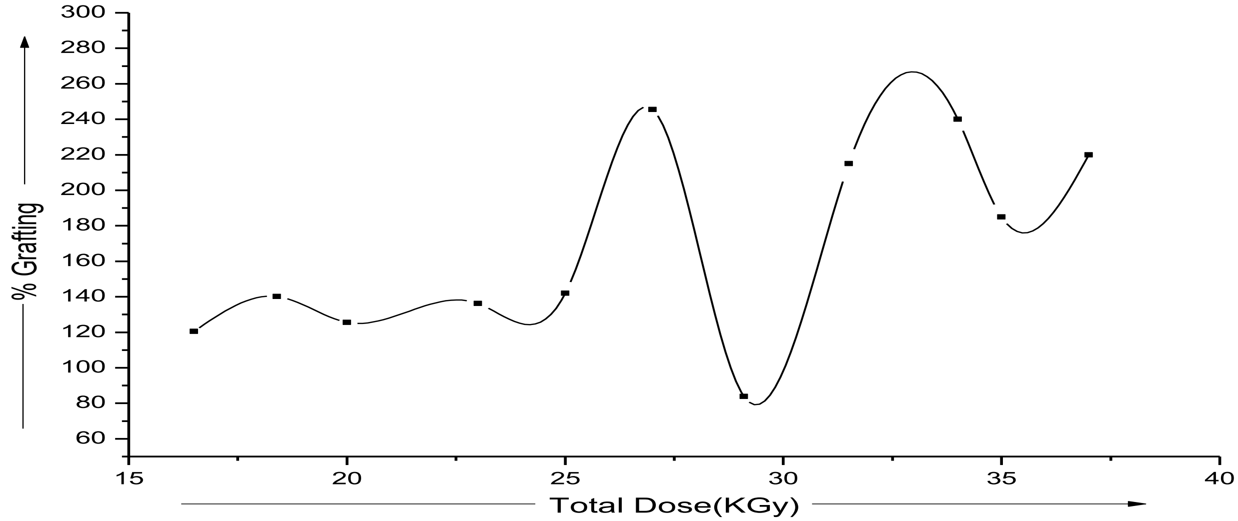

Figure 7 The effect of total dose on Percentage grafting of EA onto Tefzel film

The initial rise in P_g with the monomer corresponds to grafting in the amorphous region. A rise and fall thereafter with further, increase in the total dose is due to diffusion of the monomers in to the bulk of the film where grafting occurs in the crystalline region. For a particular total dose between 16.5 kGy to 24.2 kGy the trend of grafting is almost same with not much rise and fall, which indicates the initial generation of active sites at the amorphous region and then at the crystalline region after which grafting becomes dependent on the diffusion of the monomer into these different regions.

Effect of Monomer Concentration

The percentage of grafting of EA has been studied as a function of concentration of the monomers and the result has been presented by **Figure 8**.

P_g of EA, rises steadily with increasing monomer concentration producing 320.3% grafting at [EA] =0.027 moles/L. Since, grafting of EA is carried out in the presence of methanol-water solvent system, as the amount of monomer increases, wastage of monomer in the side reactions involving chain transfer occurs. The monomer chain transfer for EA [C_M =0.351 X10^{-4}] is also high, thus at higher concentration of EA wastage of the monomer in the side reactions and formation of the homopolymer in preference to the graft copolymer is the reason for lower P_g. Hence, EA shows same trend in both single and double irradiation methods.

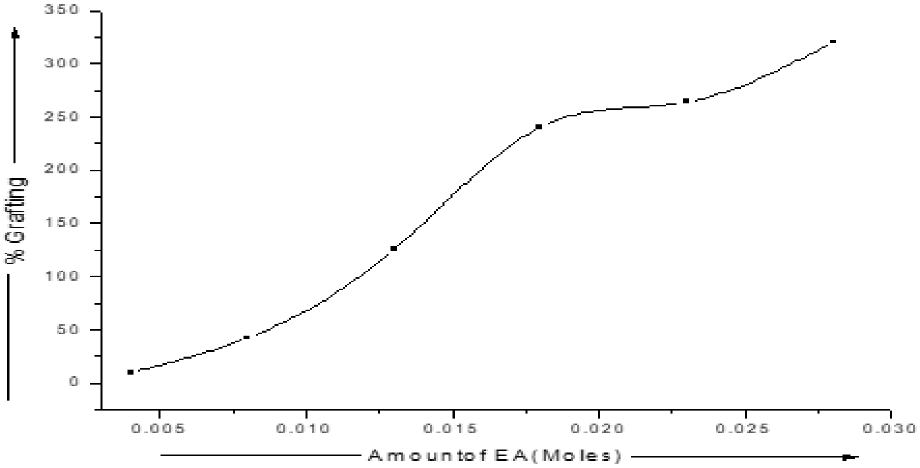

Figure 8 Effect of Amount of (EA) on Percentage Grafting

Effect of Liquor Ratio

The Pg of EA was studied as a function of amount of reaction medium. Grafting of EA was carried in water/methanol binary solvent system. The effect of this reaction medium on P_g of EA on the Tefzel film has been represented in **Figure 9**.

While studying the variation of amount of water for EA in the binary solvent system, the amount of methanol was kept constant at 5 ml and the amount of water was varied from 5 ml onwards to 25 ml and it is found from Figure 9 that the increase in the amount of water from 5 ml to 10 ml causes a sharp increase in P_g. Further increase in the amount of water to 15 ml increases P_g to maximum of 426.1%. Beyond the maximum a slow decrease in P_g is observed with increasing amount of water. Since EA is only slightly soluble in water and hence, the swelling of the backbone is also not much affected with water. But with the addition of methanol, the hydrogen bonded structure of water breaks and formation of new hydrogen bonds between methanol and water occurs. EA being soluble in methanol, becomes more accessible to the reaction sites and thus produces higher P_g.

Effect of Alcohols

The effect of composition of water –alcohol mixed solvents (10 ml) on P_g of EA onto Tefzel film has been studied and the results are presented in **Figure 10**.

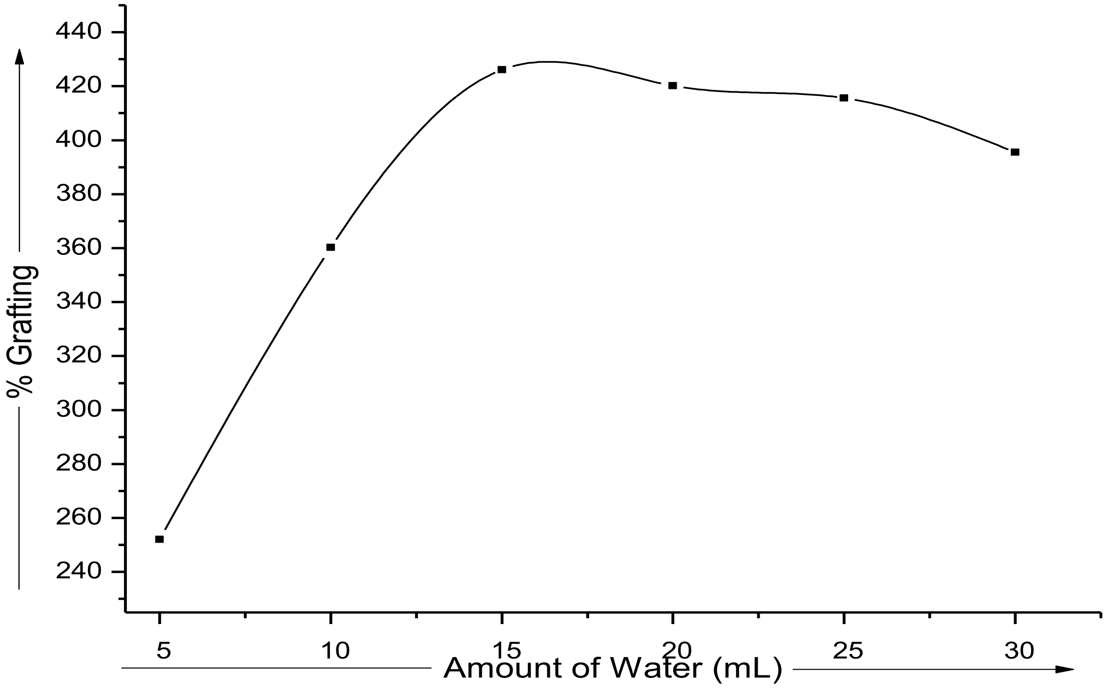

Figure 9 Effect of amount of water on percentage of grafting of EA onto Tefzel Film

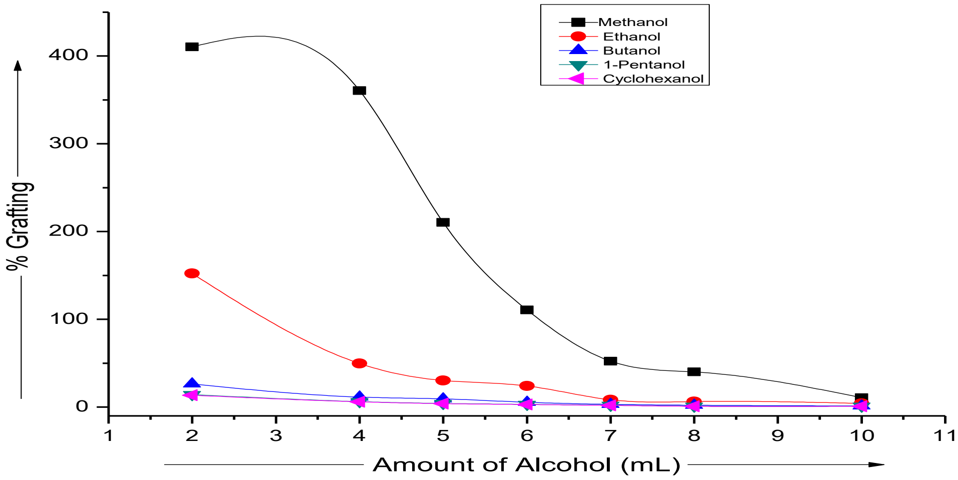

Figure 10 Effect of amount of alcohol in alcohol –water binary mixture on grafting of EA onto TEFZEL film

From the results it is observed that Pg obtained in water-alcohol medium is lower than that in aqueous medium alone for the monomer. The pattern for decrease in grafting for EA follows almost same trend in every water-alcohol system. The following order of reactivity of different alcohols influencing graft copolymerization reactions of EA has been observed as:

Methanol > Ethanol >Amyl alcohol = Cyclohexanol = n- Butanol

The order shows that the ability of the alcohol to influence graft copolymerization reaction decreases with increase in the chain length of the alcohol. The higher alcohols almost show same tendency to influence grafting in case of EA. The higher alcohols have a higher tendency to break the H-bonded water structure and also the bond between the backbone and water molecules and instead an associated structure between alcohol and water is formed. The whole process and the disturbance in the reaction medium decrease the moment and also the accessibility of the growing polymeric chains to the active sites leading to decrease in P_g.

Conclusions

P_g for EA = (245.5%) in double irradiation is higher than EA= (nearly 120%) in single irradiation[7] at a total dose of 27.0KGy. This may be due to more active sites produced on the backbone polymer resulting higher percentage of grafting in double irradiation method. Also, the vapour pressure of EA is less (39.3m bar) at 20^0C, which shows that under the given reaction conditions EA tends to remain more in the liquid state hence, EA may become more accessible to the active sites of the backbone resulting higher P_g. The rise and fall in the P_g for EA show that initial grafting occurs at the amorphous region, then the diffusion of monomer occurs into the bulk of the film where grafting occurs in the crystalline region.

Tefzel, since lacks in functional groups and on grafting with various monomers could introduce functional groups in this polymer which would increase its chemical reactivity. Modification of this polymer by grafting also increase its thermal stability which could show strong resistance to external attacks and to chemicals which would give outstanding life expectancy and insensitivity to even UV radiations and pollution. Swelling behaviour of the grafted film could increase its applicability in separation processes as membranes. The grafted film could raise its industrial applicability thereof.

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