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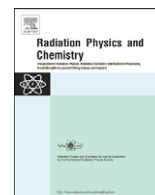
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Novel polysulphonates and poly(sulphonate-co-carbonate) polymers for nuclear track detection

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ABSTRACT

Preparation of homopolymer from diethylene glycol bis(allyl sulphonate) (DEAS) and its several copolymers with allyl diglycol carbonate (ADC) and their preliminary evaluation as nuclear track detector is described. The monomer is synthesized by reacting diethylene glycol with allyl sulphonyl chloride in presence of pyridine as a base. Spectral characteristics of the monomer are also given for the first time. The designed polymers were successfully tested for detection of ^{239}Pu alpha particles and ^{252}Cf fission fragment tracks after etching with 6 N NaOH at 70 °C. Some of the polymers prepared showed even better sensitivity than (SR-86)(20) track detectors for track detection properties. The polymers are also compared with commercial CR-39 track detectors for its track detection properties.

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1. Introduction

Fuji et al. (1990) prepared copolymers of solid diethylene glycol bis(allyl sulphonate) (DEAS) with allyl diglycol carbonate (ADC) in the ratio of 10:90 (SR-86 10) and 20:80 (SR-86 20), respectively. They also reported that due to limited solubility of DEAS (20%w/w) in ADC, they could not prepare other copolymers with higher proportions of DEAS. Both 10:90 (SR-86 10) and 20:80 (SR-86 20) were found to be more sensitive than PADC i.e. CR-39 track detector and other known polymeric track detectors. It may be expected that DEAS homopolymer may show better track detection properties to CR-39 due to the presence of more labile S = O links as compared to C = O links in CR-39.

Similarly, DEAS:ADC copolymers with higher amount of DEAS may also show better sensitivity towards charge particle track detection. Thus, in continuation with our efforts to indigenously develop and improve plastic materials for the said purpose (Mascarenhas et al., 2005, 2006), we report for the first time the preparation of homopolymer of DEAS and various copolymers of DEAS with ADC where in the concentration of DEAS is higher than 20% w/w in the polymer matrix.

Further in this paper, we report the preliminary evaluation of track detection properties of DEAS polymer in comparison with CR-39 polymer obtained from Pershold Moldings UK. A brief study of optimization of etching conditions, track detection efficiency, effect of initiator concentration and sensitivity of the polymers has been reported. Further studies pertaining to proton recoil tracks, neutron dosimetry, etc. will be reported subsequently.

2. Material and methods

The DEAS monomer was synthesized and was recrystallized from ethyl acetate before use. Plasticizer dioctyl phthalate (DOP) manufactured by M/s Loba chemie, India was used without further purification. Commercially available benzoyl peroxide (BP) initiator from M/s Loba chemie, India was recrystallized from chloroform/methanol (1:2 v/v) mixture and used after drying under vacuum. Isopropylperoxydicarbonate initiator was prepared in our laboratory and used without further purification. Poly allyl diglycol carbonate (PADC) films from Pershore Moldings (UK)(CR-39) were used for comparative studies. Optical glass plates from Schott, Germany and TeflonTM sheets were used for preparation of polymer molds. Polymerization was carried using a polymerization bath controlled using microprocessor-based electronic temperature controller F25 HP from M/s Julabo, Germany.

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2.1. Monomer synthesis: the monomer can be conveniently synthesized in a three-step process

2.1.1. Synthesis of allyl sodium sulphonate

Allyl chloride was added drop wise to a stirred solution of aqueous sodium sulphite in a three neck flask provided with a reflux condenser and a thermometer. After completion of addition, the reaction mixture was stirred at 60 °C for 6 h. Water was removed by distillation and the solid obtained was recrystallized from water ethanol mixture. The product obtained was dried and had a yield of 70% with respect to allyl chloride. The synthetic Scheme 1 is given below.

2.1.2. Synthesis of allyl chloro sulphonate

The allyl sodium sulphonate was reacted with phosphorous oxy chloride and the mixture was heated at 120 °C for 8 h (Dauban and Dodd, 2000). Reaction mixture was poured in ice water and the brown coloured product was extracted in ether. The ether was evaporated and the mixture was finally distilled under reduced pressure. The yield of the product was 65% based on allyl sodium sulphonate. The synthetic Scheme 2 is given below.

2.1.3. Synthesis of diethylene glycol bis(allyl sulphonate)

Diethylene glycol was condensed with allyl chloro sulphonate in the presence of pyridine as base to get DEAS. The product obtained was purified by column chromatography to get a white crystalline solid having 80% yield based on diethylene glycol. Scheme 3 depicts the reaction.

2.1.4. Spectral characterization

The structure of DEAS was characterized by recording its IR, ¹H NMR and ¹³C NMR data:

IR KBr: 1643 cm⁻¹ (s), 1346 cm⁻¹ (m), 1165 cm⁻¹ (s), 935 cm⁻¹ (m),

¹H NMR (CDCl₃): 3.7 ppm (–SO₂–CH₂–, 4H, t), 4.1 ppm (–O–CH₂–, 4H, t), 4.3 ppm (–SO₂–O–CH₂–, 4H, t), 5.4 ppm (CH₂=, 4H, dd), 5.90 ppm (–CH=, 2H, m),

¹³C NMR (CDCl₃): 54.90 ppm (–SO₂–CH₂–, 2C, t), 69.51 ppm (–O–CH₂–, 2C, t), 69.53 ppm (–SO₂–O–CH₂–, 2C, t), 124.36 ppm (CH₂=, 2C, d), 124.72 ppm (–CH=, 2C, t).

The structure of the monomer was also confirmed by using single-crystal X-ray crystallography. (Srinivasan et al., 2007).

Testing protocol for new polymers: Based on our experience since 1992 in designing polymeric track detectors, we have prepared a protocol that may be useful for a track detector worker to design and synthesize new monomers/polymers for track detection (Nadkarni, 2008).

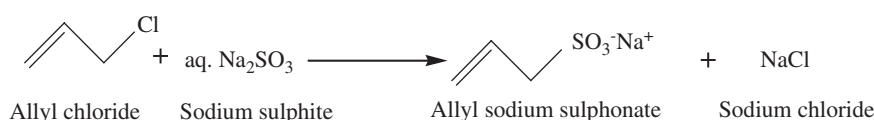
Step 1: Initial studies of a monomer/polymer. The (allylic) monomer is designed keeping in mind certain known facts and is then synthesized. After testing the thermal stability by TG–DSC techniques, a sample polymer film is prepared using cast polymerization using suitable initiator. This sample detector film is exposed to a radiation source (like ²⁵²Cf) and etching in 6 N NaOH at 70 °C. Observe revelation of tracks (fission tracks or alphas). A copolymer with ADC (normally 1:1 w/w) (or with other monomers) may be prepared and tested as above.

Step 2: Kinetics of polymerization. Apply Dial's kinetic model (Dial et al., 1955) to pure allylic monomer/mixture of allylic monomers and generate a constant rate polymerization cycle.

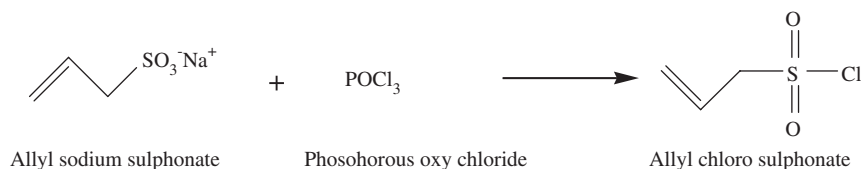
Step 3: Optimization of initiator concentration. Prepare films using different concentration of initiator/different initiators. Find the sensitivity by noting diameters of alpha/fission tracks and determine the optimum initiator concentration where sensitivity is maximum. Find the initiator which gives films with higher sensitivity.

Step 4: Optimization of etching conditions. Prepare the detector films using the optimized initiator concentration and etch the films, keeping temperature of etchant constant but varying normality and then by keeping normality constant but varying the temperature of etchant. Note bulk etch rate (V_b) at every etching interval and also track appearance time; select those conditions (normality and temperature) at which V_b is moderate so that the track development occurs within a reasonable time, post-etch appearance of the film is good.

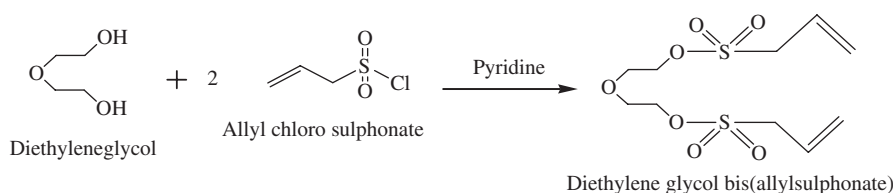
Step 5: Find the sensitivity of these films at the optimized etching conditions.



Scheme 1. Synthesis of allyl sodium sulphonate.



Scheme 2. Synthesis of allyl chlorosulphonate.



Scheme 3. Synthesis of DEAS.

Other studies: Any other studies of such new detector films for neutron/proton/gamma detection, electrochemical etching studies, etc. may be carried out further. In our ongoing research in this field, we follow this protocol so as to get better performing polymeric track detectors.

2.2. Preparation of polymer films

2.2.1. Mold assembly

The polymerization was carried out by using a mold designed for casting thin (about 500–600 μm) films. A square-shaped gasket of Teflon with thickness of 500 μm with an opening of 2 mm was specially prepared for this purpose. A thin layer of a commercially available adhesive was applied to it from both sides and sandwiched between two clean optical glass plates of size 100 mm × 100 mm × 4 mm.

2.2.2. Cast polymerization

The monomer DEAS was melted at the temperature of around 60 °C and dry nitrogen was then flushed for 30 min. BP (4% w/w) initiator and dioctyl phthalate (1%w/w) was added as a plasticizer. The mixture was injected in the mold using a syringe with at most care to exclude the formation of air bubbles. The mold was sealed and kept in the pressurizing assembly and was polymerized using 12 h constant rate polymerization cycle devised for ADC as per kinetic model proposed by Dial et al. (1955).

The mold was heated using a programmable circulating water bath and was periodically tightened in order to prevent cracking of the film due to the polymer shrinkage and allowed to cool for further 12 h. Similarly all the copolymers of the DEAS with ADC were prepared by mixing molten DEAS with ADC in required w/w proportions. It was found that the colour of the polymers varied from yellow to dark brown as proportion of DEAS was increased.

Although, we have successfully extended the above kinetic model to many other allylic monomers (Mascarenhas et al., 2005, 2006), the same could not be applied to study kinetics of DEAS mainly because S=O in all the sulphur containing monomers like DEAS, allyl diglycol sulphite or diallyl sulphite reacts with titrimetric reagents like ICl thereby hindering the kinetic measurements. Thus, looking at the structural similarity of polymerisable parts (two allyl groups) of DEAS and ADC monomers, we decided to polymerize the monomer mixture of ADC+DEAS as per constant rate polymerization cycle earlier obtained by us for ADC polymerization. (Mascarenhas, 2007).

3. Results and discussion

The DEAS monomer melts at relatively low temperature of 53.3 °C. Thus, the polymerization of molten DEAS would be possible with BP initiator which decomposes at a higher temperature of 70 °C. DEAS monomer was first heated on water bath to melt and to the molten DEAS BP initiator was added. The initiator was dissolved in molten DEAS by swirling and was followed by the addition of dioctyl phthalate plasticizer. The polymerization mixture was injected in mold and polymerized using the previously mentioned ADC polymerization cycle. All the copolymers discussed in the paper are prepared in a similar manner.

The DEAS:ADC (3:7 w/w) copolymer was prepared by using isopropyl peroxydicarbonate (IPP) initiator. A hard light brown polymer obtained was studied for its track detection properties, sensitivity and optimization of initiator concentration. The polymer obtained had less brown colour as compared to polymer obtained by using BP initiator.

3.1. Polymerization using benzoyl peroxide (BP) initiator

The homopolymer of the DEAS (PDEAS) and DEAS:ADC copolymers in the ratio 8:2, 7:3, 6:4, 1:1, 4:6, 3:7, 2:8 and 1:9 (all w/w), respectively, were prepared by using BP as initiator. The homopolymer was hard and brown coloured, whereas the colour of other copolymers increased in brown colour as the concentration of DEAS increased.

3.1.1. Testing of films for charged particle track detection

The polymer obtained were cut into sizes of 1 × 1 cm² and exposed to alpha particles from ²³⁹Pu in close contact (~1 mm from source) and to fission fragments from ²⁵²Cf source for track detection characteristics. The polymers could be conveniently etched in 6 N NaOH solution at 70 °C. The time required for alpha and fission fragments appearance is given in Table 1.

Thus, it is clear that the copolymers show shorter track development time than the PADC track detector. The tracks in the copolymer films could be easily viewed under an optical microscope. Although the impression of the autoradiograph of the ²³⁹Pu alpha source was noticed after etching the homopolymer PDEAS for 2 h, the alpha particles could not be observed clearly due to dark brown colour of the film along with slight opacity developed in the film after etching. From the above results one can notice that the time required for track detection for alphas and fission fragments is nearly half than that of PADC. It was further observed that the copolymers tend to become soft on standing for about 15 days, while the homopolymer turned soft within 3–4 days.

3.1.2. Sensitivity studies

The alpha sensitivity of these polymers was determined by noting the diameters of alpha and fission fragments tracks (Fujii et al., 1990) at 1 h interval by etching the films up to 4 h. The DEAS:ADC (3:7w/w) copolymer was found to have the maximum alpha sensitivity among the copolymers prepared and was even more sensitive than PADC and SR-86. The alpha sensitivity for the DEAS homopolymer could not be determined as the tracks could not be identified clearly after etching under the microscope, due to slight opaqueness of the film after etching.

The sensitivity of the polymer was determined by using the formula

$$\text{sensitivity} = 1 + (D_\alpha/D_f)/1 - (D_\alpha/D_f)^2$$

where D_α is the diameter of alpha particle track and D_f is the diameter of fission fragment track.

The bulk etch rate of the polymer was found by using the formula

$$V_b = D_f/2t$$

Table 1
Track revelation time for different copolymers

Composition of polymer DEAS:ADC	Time required (min) to observe fission fragment tracks (exposed at a distance of 1 mm from source)	Time required (min) to observe autoradiograph of ²³⁹ Pu alpha source
PDEAS	60	180
1:9	30–45	60
2:8	30	60
3:7	30	60
4:6	30	60
1:1	30	60
6:4	30	60
7:3	30	60
8:2	60	90
PADC	60	120
CR-39	60	120

where D_f is the diameter of fission fragment track and t is the time (Durrani and Bull, 1987).

The bulk etch rate of the polymer was found by using weight loss method. (Mascarenhas et al., 2005),

$$V_b = (M_1 - M_2)T_i / 2M_2t$$

Table 2 gives the comparative account of the sensitivity studies and bulk etch rates of the polymers.

It may be seen that V_b increases as the proportion of DEAS monomer increases in the copolymer film. This is due to the presence of more labile $-O-SO_2-$ linkage in the polymer. If, sensitivity is considered as ratio of V_t/V_b then it is obvious that such films will have lesser sensitivity.

Thus, from the above results it is clear that the DEAS:ADC (3:7 w/w) copolymer has the maximum sensitivity in the series. The (3:7 w/w) copolymer is a hard film with light brown colour. Further studies about the optimization of etching condition, effect of initiator concentration and track detection efficiency along with alpha to fission branching ratio were performed using the same copolymer.

3.1.3. Optimization of initiator concentration for BP initiator

The initiator concentration is an important factor on which sensitivity of particular material depends. Copolymers of DEAS:ADC 3:7 with BP initiator concentration of 2%, 3% and 4% (w/w) were prepared. It was found that BP is not soluble in the monomer mixture above this concentration. The higher initiator concentration polymers were not possible as above 4% concentration, initiator was insoluble in the monomer mixture. The polymer with 4% (w/w) initiator concentration gave the hardest film. Whereas the other films with lesser concentration were slightly soft. The polymer with 4% initiator concentration was most sensitive and the values obtained are given in Table 3.

3.2. Polymerization using isopropyl peroxydicarbonate (IPP) initiator

The copolymer DEAS:ADC in the ratio 3:7 (w/w) was prepared with 5% w/w IPP initiator. The polymer obtained was hard and had a light brown colour which was to a lesser extent than what was observed with DEAS:ADC polymerized using BP initiator. The sensitivity obtained for the polymer was 2.30.

3.2.1. Optimization of IPP initiator concentration

Different films of DEAS:ADC (3:7 w/w) were prepared using initiator concentration ranging from 2% to 6% w/w of IPP in order to determine optimum concentration of IPP where sensitivity of the polymer was maximum. The 4% IPP initiator concentration film gave maximum sensitivity of 2.54 as shown in Fig. 5. The films obtained with 4–6% w/w initiator concentration were hard

Table 2

Alpha sensitivities and bulk etch rates of various polymers

Polymer DEAS:ADC	Maximum sensitivity	Bulk etch rate (V_b) ($\mu\text{m}/\text{h}$)
CR-39	1.22	0.015
PADC (indigenous)	1.20	0.011
1:9 (SR-86(10))	1.41	0.0200
2:8 (SR-86(20))	1.74	0.0220
3:7	2.38	0.0220
4:6	1.60	0.0350
1:1	1.36	0.0300
6:4	1.23	0.0355
7:3	1.21	0.0505
8:2	1.20	0.0515
PDEAS	–	2.083 ^a

Exposure: ^{252}Cf source at 5 cm distance and 0.2 mbar pressure.

^a Weight loss method was used for bulk etch rate measurements.

Table 3

Optimization of benzoyl peroxide initiator concentration for DEAS:ADC (3:7 w/w)

% Concentration (w/w) of BP initiator	Particles detected	Physical appearance	Sensitivity
4	Alpha and fission fragments	Hard and light brown colour	2.38
3	Alpha and fission fragments	light brown colour	1.87
2	Fission fragments only	Soft film	Not determined

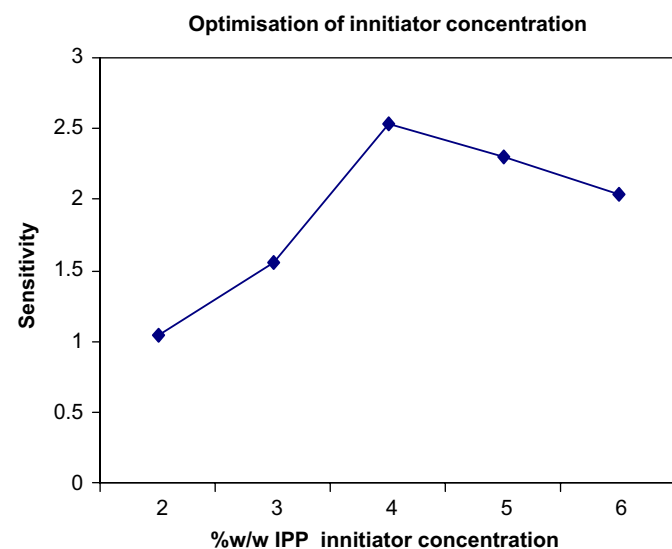


Fig. 1. Optimization of initiator concentration studies.

films whereas the other two films were comparatively soft. The change in sensitivity with respect to initiator concentration is depicted in Fig. 1.

3.3. Optimization of etching conditions

The polymer was cut into a size of $1 \times 1 \text{ cm}^2$ and was exposed to fission fragments and alpha particles from ^{252}Cf and ^{239}Pu source, respectively, in close contact (1 mm). The polymers were then etched in 4–7 N NaOH solution and at temperatures between 60 and 80 °C. The bulk etch rate of the polymer, track development time and the physical appearance of the film were determined of the film at different etching conditions. Physical appearance of the film was not damaged at the higher temperature. The film became soft at higher etching temperature when it was etched for longer time. The time required for track formation at different etching condition is given in Table 4.

It can be seen that the time required at 60 °C was almost double than that is required at 70 °C. The bulk etch rate of the polymer at different etchant concentration at different temperature is graphically represented in Figs. 2 and 3.

Like PADC, therefore, etching of DEAS:ADC (3:7) copolymer may be preferably done using 6 N NaOH at 70 °C. Alternatively etching using 6 N NaOH at 60 °C may be used. The sensitivity studies indicated that, maximum sensitivity values are observed only after etching for 8 h or more.

3.4. Sensitivity studies at optimized etching conditions

The DEAS:ADC copolymers were prepared from different initiators were etched at optimized etching conditions and the

Table 4
Time required at different temperatures for track development

Etchant	Temperature (°C)	Time required for track visualization in minutes	
		Alpha	Fission fragments
4 N NaOH	60	180	60–90
	70	60–90	30
	80	60	30
5 N NaOH	60	180	60–90
	70	60	30
	80	60	30
6 N NaOH	60	120	60
	70	60	30
	80	60	30
7 N NaOH	60	120	60
	70	60	30
	80	60	30

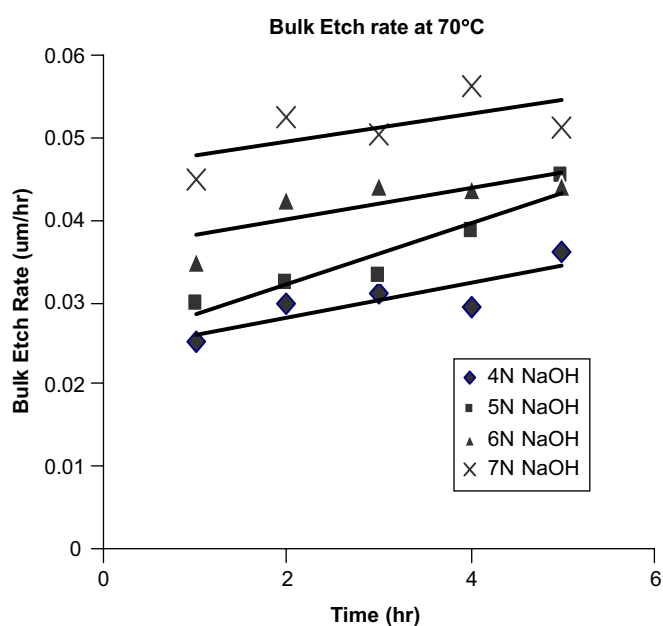


Fig. 2. Bulk etch rate at 70 °C.

results obtained are given. The sensitivity for detector with IPP was more than that prepared with BP. This is in accordance with observations made with PADC detectors prepared with IPP and BP which was 1.38 and 1.19, respectively (Mascarenhas 2007). Fig. 4 gives results of comparative sensitivity study of 3:7 DEAS:ADC copolymers obtained from BP and IPP initiated polymerization. A photomicrograph of the tracks is shown in Fig. 5.

The track diameters were also larger in case of the polymer prepared by using IPP initiator. Fig. 6 gives the track growth rate of the polymers with different initiator.

Thus, it can be concluded that DEAS:ADC detector prepared with IPP initiator shows better performance and hence further studies are carried out using this detector.

3.5. Alpha track detection efficiency

The alpha track detection study for the polymer was performed by exposing the films to ²³⁹Pu alpha particle source. For comparison CR-39 film are used. The films were cut into the sizes of 20 × 20 mm² and exposed to ²³⁹Pu alpha particles at a distance of 30 mm for 5 min. The polymer pieces were then etched under the previously optimized etching condition i.e. 6 N NaOH at 70 °C. The tracks were then counted under an optical microscope and tracks per square centimeter were found out. The blank

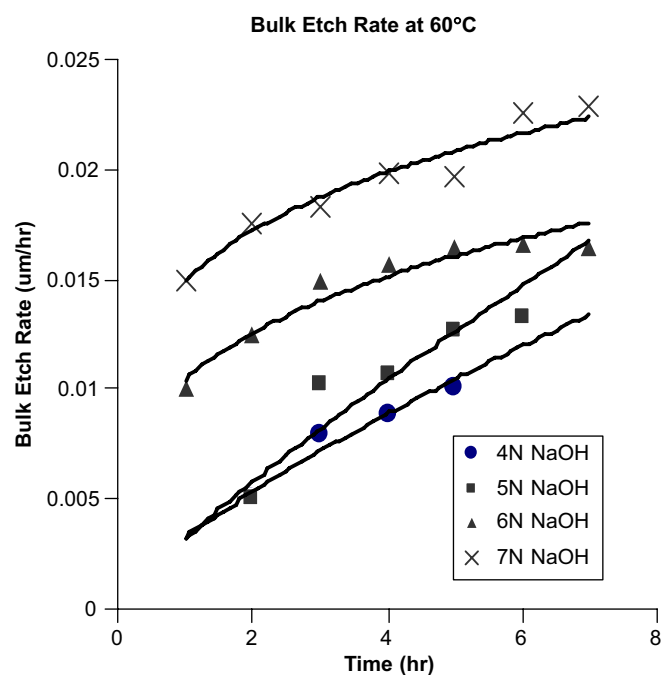


Fig. 3. Bulk etch rate at 60 °C.

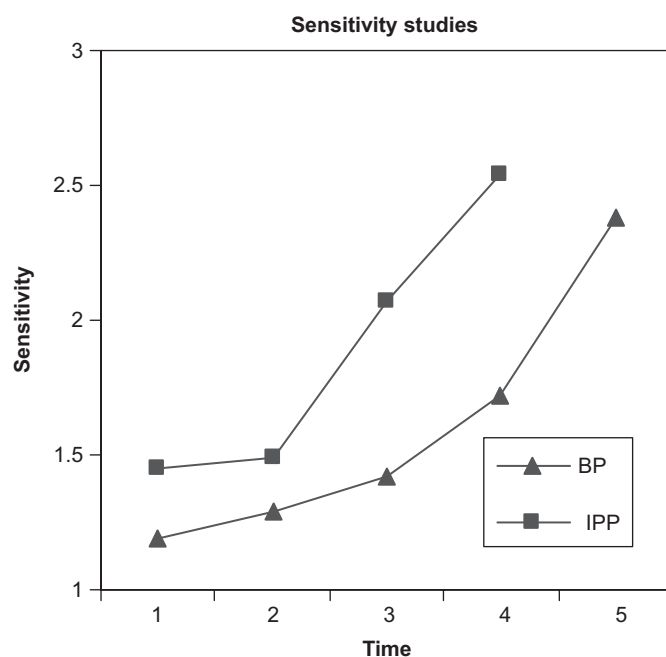


Fig. 4. Sensitivity studies of polymers with different initiators. BP: copolymer DEAS:ADC 3:7 with BP initiator. IPP: copolymer DEAS: ADC 3:7 copolymer with IPP initiator.

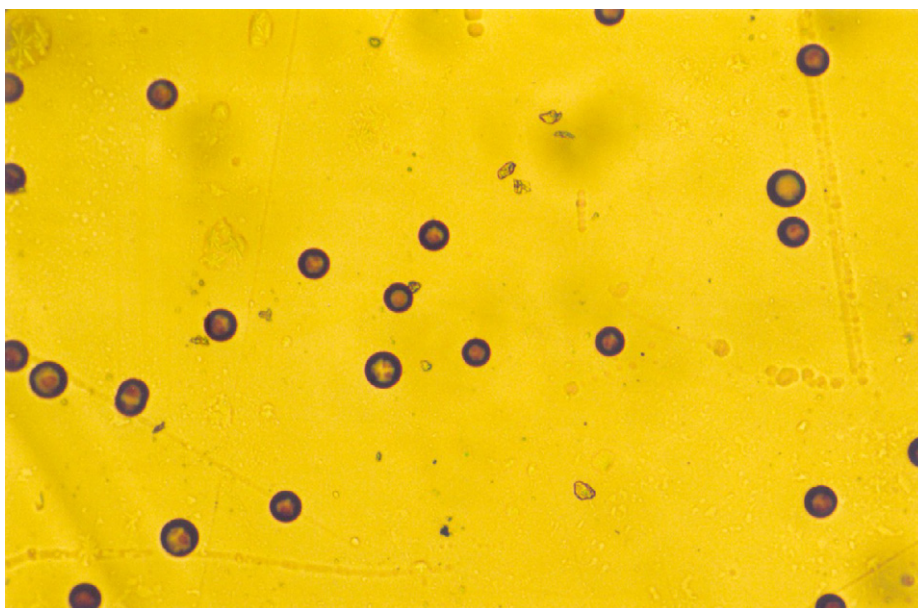


Fig. 5. A photomicrograph showing fission fragments and alpha particle tracks in DEAS:ADC (3:7 w/w) detector film from ^{252}Cf source exposed at 5 cm distance and 0.1 mbar pressure.

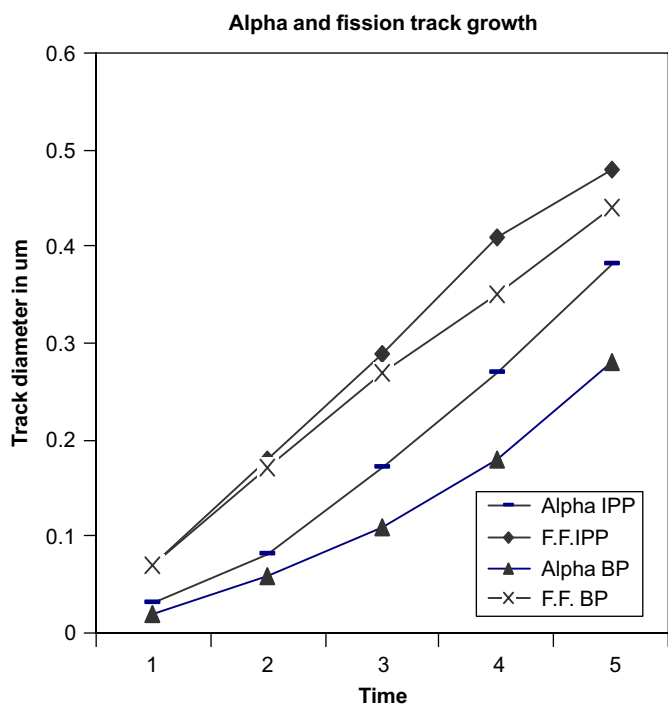


Fig. 6. Track growth of alpha and fission fragment particles with different initiators. Exposure: to ^{252}Cf at 5 cm distance at 0.2 mbar pressure.

determinations were also performed to determine the number of background alpha track-like features in the films and were subtracted from observed alpha tracks. The results obtained are given in Fig. 7.

The tracks were counted after every hour to optimize the time required for maximum number of alpha track formation. For DEAS:ADC 3:7 copolymer only 2 h were required for maximum number of alpha track revelation as compared to 3 h for CR-39TM. Thus, from the data obtained it is clear that the films are comparable to imported CR-39 track detector in terms of alpha track detection efficiency.

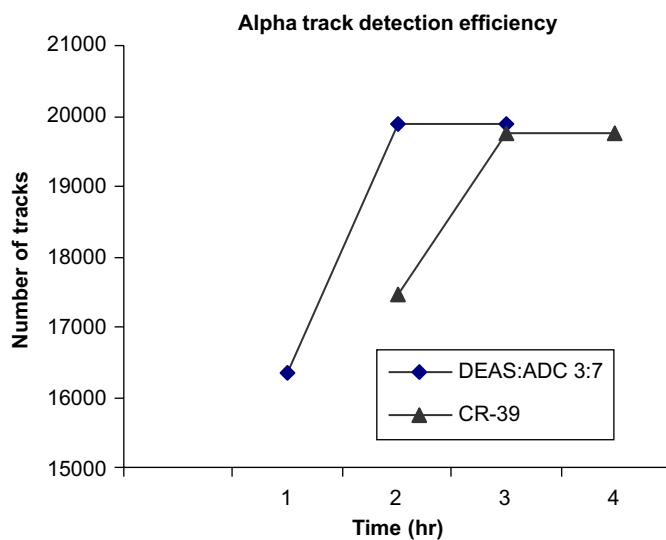


Fig. 7. Track detection efficiency. Exposure: to ^{239}Pu source at 3 cm distance for 5 min.

3.6. Alpha to fission branching ratios by sequential chemical etching of alpha and fission tracks

The sequential chemical etching method for revelation of alpha and fission fragment tracks can be used for studies of half lives, alpha to fission fragment branching ratios and identification of radio nuclides using single detector (Pandey et al., 1993). The tracks of alphas and fission fragments can be differentiated if there is a significant difference in the size and appearance time in a given detector. The DEAS:ADC 3:7 track detector meets all the requirements as nuclear track detector and is time convenient as there is considerable difference in etching time and sizes of the tracks formed. The number of tracks found is comparable to CR-39 track detector as can be seen in Table 5.

Films exposed to ^{252}Cf source for 2 h in 4π geometry at a distance of 10 mm and for 1 h in 2π geometry at a distance of 6 mm.

Table 5
Alpha to fission fragments branching ratios

Detector	Fission fragment tracks (/cm ²)	Alpha tracks (/cm ²)	$\alpha/F:F$ ratio in 2π geometry	$\alpha/F:F$ ratio in 4π geometry
CR-39	4.09×10^2	64.41×10^2	15.75	30.82
DEAS:ADC 3:7	4.27×10^2	65.73×10^2	15.37	30.57

3.7. Conclusion

We have successfully prepared DEAS homopolymer and some of its copolymers with ADC for the first time. The homopolymer can be prepared by melting the monomer. The DEAS monomer was also copolymerized with ADC in the ratio of DEAS:ADC 1:9, 2:8, 3:7, 4:6, 5:5, 4:6, 3:7, 2:8 (w/w), respectively. All the copolymers were good track detectors for alpha and fission fragments track detection.

The 3:7 DEAS:ADC copolymer was most sensitive with sensitivity higher than SR-86 and commercially available CR-39 from Pershore Moldings UK. The polymer has sensitivity almost double than that of PADC and CR-39TM.

The results also point out that the use of IPP initiator not only increases the sensitivity but also decreases the time required to attain the maximum sensitivity of the polymer. But, the IPP initiator cannot be used for polymerization of DEAS homopolymer or copolymers where DEAS is in higher proportions as it leaves some imperfection in the polymer matrix there by making the polymer opaque. This might be due to low temperature of polymerization which possibly allows solidification of the monomer during polymerization.

The polymers were time efficient as the time required for track detection was reduced. The alpha autoradiograph of ²³⁹Pu source could be seen in just 1 h, whereas the fission fragment tracks from ²⁵²Cf source can be seen in 30 min. The polymers were suitable as nuclear track detector in all respects like colour, appearance and observation under the microscope. Studies pertaining to detection of recoil proton/neutrons are in progress and will be discussed separately. Studies with respect to kinetics of polymerization and possible depolymerization or instability of this material are also in progress.

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