



New polymers for solid state nuclear track detection

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Abstract

Two novel polymers have been prepared from a monomer *N*-allyloxycarbonyl diethanolamine-bis(allyl carbonate) i.e., NADAC by cast polymerization and were successfully used as solid state nuclear track detectors for α particles and fission fragments. The homopolymer PNADAC was prepared by IPP catalyzed polymerization of NADAC. A copolymer of NADAC with allyl diglycol carbonate (ADC) called poly-[(NADAC)-co-(ADC)] was also prepared similarly. A preliminary study on the etching conditions and track detection characteristics of the newly developed materials was carried out. It was found that the copolymer is more sensitive to α particles as compared to commercially available PM-250™ track detector.

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1. Introduction

Solid state nuclear track detection (SSNTD) is a widely used technique among the various known methods for detection of nuclear radiation (Durrani and Bull, 1987). This method owes its popularity mainly to its simplicity, cost effectiveness and capacity to store permanent records. Although minerals and glasses have been tested as SSNTDs, the higher sensitivity of the plastic materials to many charged particles and ease of track development and evaluation has made plastic materials more popular as SSNTDs. Since the introduction of plastics as SSNTDs in 1965 (Collver et al., 1965), a number of plastic materials, such as cellulose, polycarbonates, acrylates and polyvinyls, have been extensively studied as track detectors. The introduction of poly

allyl diglycol carbonate (PADC) polycarbonate (Cartwright et al., 1978) replaced many of the plastics due to its very high sensitivity to many charged particles, and superior optical properties. It is observed that since 1965, efforts are mainly directed towards testing the commercially available plastics as SSNTDs rather than attempts to design newer plastics as SSNTDs. A few systematic reports on development of some sulphonate resins and some other materials as SSNTDs (Fujii and Yokota, 1988; Fujii et al., 1990; Ogura et al., 1995) are known. This included generalization of some of the structural features of the polymer matrix, which may help in designing materials of higher sensitivity. Other studies have also revealed that the copolymer with ADC has been more sensitive than the homopolymers. Fujii reported the possible relation between structure and track detection characteristics (Fujii et al., 1990) as follows: (1) All polymers containing aromatic rings tend to show lower sensitivity. (2) The highly cross-linked thermosets and the amorphous polymers show better sensitivity. (3) The chain

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bridging the carbonate moieties can be crucial for sensitivity. (4) Materials having one of the functional groups like $-\text{ONO}_2$, $-\text{CO}_3-$, $-\text{SO}_2-$ and $-\text{SO}_3-$ have been found to be more sensitive. (5) The density of the cross-links in the polymer matrix was also an important factor. (6) For PADC (CR-39)TM, it is known that, the copolymers are more sensitive than homopolymer. Thus, there is ample scope for designing new polymer materials in terms of its constitution and structural features for use as SSNTDs.

We have been working on the preparation of plastic materials as SSNTDs, since 1996 (Nadkarni and Samant, 1996). In continuation with our efforts to indigenously prepare and improve plastic materials for SSNTD applications, we present the preliminary results of casting homo and copolymer films from a novel monomer (Nadkarni et al., 2003,2004) *N*-allyloxycarbonyl diethanolamine-bis(allyl carbonate) (NADAC) monomer with allyl diglycol carbonate (ADC) in this paper. It was thought that a hexafunctional monomer would give a polymer matrix of high-density cross-links, which would lead to high sensitivity of the materials to charge particles. The idea was also to introduce different types of functionalities in a single system. A brief description of the study of some of the etching conditions, the effect of initiator concentration on the sensitivity of the materials and also track detection efficiency studies in comparison with commercially available PADC has been reported.

2. Materials and methods

Indigenously prepared NADAC and ADC monomers, isopropyl peroxydicarbonate (IPP) initiator were used for the preparation of poly-[*N*-allyloxycarbonyl diethanolamine-bis(allyl carbonate)] i.e., (PNADAC) homopolymer and NADAC-ADC copolymer. PADC films from Pershore Molding (UK) of thickness 250 μm was used for comparative studies. Dioctyl phthalate (DOP) plasticizer manufactured by S.D. Fine Chemicals, India was used as such. Glass plates (Schott, Germany), thin TeflonTM sheets,

aluminium plates were used for the polymerization molds. The various steps involved in the synthesis and polymerization of monomers are briefly described below. IPP was prepared in our laboratory and polymerization carried out using a polymerization bath controlled using microprocessor-based electronic temperature controller F25 HP, from Julabo, Germany.

2.1. Synthesis of NADAC

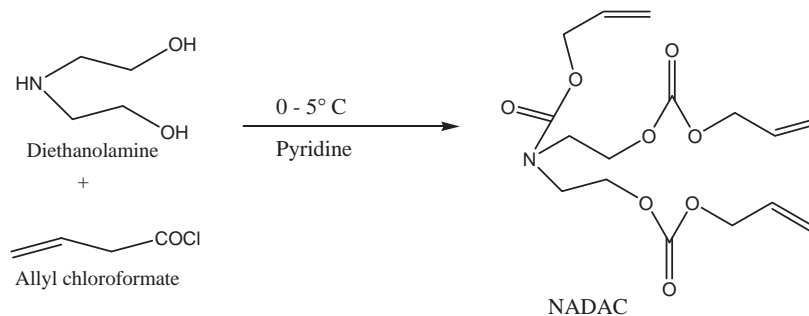
The NADAC monomer was synthesized by reacting diethanol amine and allyl chloroformate (Nadkarni et al., 2004) as indicated in Scheme 1:

2.2. Preparation of the mould

A square-shaped gasket of Teflon with an outer length of 100 mm, inner window of length 80 mm, and a thickness of $500 \pm 10 \mu\text{m}$ was specially prepared for this purpose. It was sandwiched between two clean optical glass plates of size 100 mm (*l*) \times 100 mm (*b*) \times 4 mm (*t*). A typical mould assembly is shown in Fig. 1.

2.3. Preparation of SSNTD films by cast polymerization

NADAC and ADC monomers were filtered carefully to remove any solid impurities. The monomers were stirred under nitrogen atmosphere to remove any dissolved air and oxygen which hinder the process of polymerization. The mixture containing the monomer, initiator (3% by wt) and plasticizer (DOP, 1% by wt) was carefully injected into the mould using a syringe, through a previously prepared tiny hole in the Teflon gasket. Care was taken to avoid any air bubbles in the liquid monomer film during the injection in the mould. The mould was sandwiched between two flat aluminium plates of uniform thickness and pressurized using wing-nut bolts fitted at the four corners of aluminium plates. The entire assembly was then kept in the polymerization bath and heated as per predetermined temperature–time intervals obtained after a careful polymerization kinetics study of NADAC



Scheme 1.

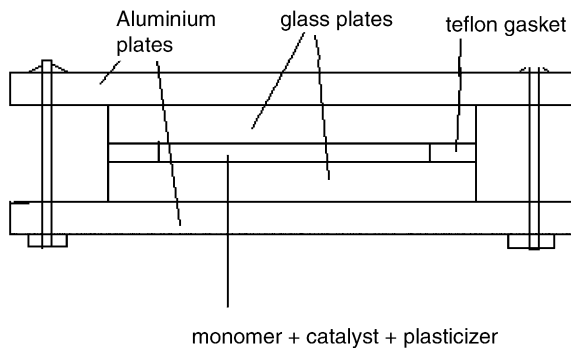


Fig. 1. Mould arrangement for polymerization.

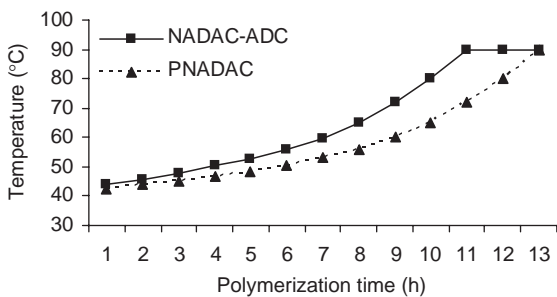


Fig. 2. Calculated 12 h constant rate polymerization cycle for homo and copolymerization.

monomer/mixture of NADAC–ADC monomers. (Nadkarni et al., 2003,2004). The polymerization temperature–time profile used for the polymerization is shown in Fig. 2.

The mould was then allowed to cool slowly for another 12 h and then opened. A clear transparent film of approximately $500 \pm 10 \mu\text{m}$ thickness was obtained which was carefully detached from the glass plates. It was cut into pieces of size $10 \times 10 \text{ mm}^2$ and used for further studies.

2.4. Studies of etching conditions

Small pieces of size $10 \times 10 \text{ mm}^2$ of homo and copolymer films prepared using 3.0% IPP initiator were used for the etching study. They were suspended vertically in an etching bath containing aqueous NaOH and the temperature was maintained using a constant temperature circulating water bath. The etching studies were carried out by varying concentration of NaOH at three different temperatures.

2.5. Determination of sensitivity of the films

The sample pieces of the homo and copolymer films were exposed to normally incident α particles and fission fragments from a ^{252}Cf in vacuum (~ 0.1 Torr) at a distance of 50 mm from the source and etched in 6N NaOH at 80 and

75 °C. The etch pit diameters were determined using an optical microscope (Axiostar, Carl Zeiss, Germany) at a magnification of $40\times$. For the thickness measurement, a digital thickness gauge (Alpha meter, Para Electronics, India) with a least count of $1 \mu\text{m}$ was used. All the weight measurements were carried out using a digital single pan balance (Sartorius) having a least count of 10^{-4} g.

3. Results and discussion

3.1. Preparation of the polymer

IPP initiator cannot be purified before its use, due to its unstable nature. But it needs to be estimated for its percentage purity by titrimetric analysis just before its use. The initiator can be added to the NADAC and mixture of NADAC–ADC (1:1 wt. proportions) monomer at room temperature ($28\text{--}30^\circ\text{C}$) safely without any appreciable decomposition of the catalyst. However, the monomer needs to be stirred under nitrogen for several hours to remove any dissolved air or oxygen before the addition of the IPP initiator under nitrogen atmosphere.

As allylic polymerization is an exothermic process accompanied by shrinkage in monomer volume, during the process of solid polymer formation (Dial and Gould, 1945; Dial et al., 1955) the polymerization was carried out using the experimentally calculated heating time cycle and periodic tightening of the moulds.

3.2. Testing of homo and copolymer films for charged particle detection

Since the newly developed materials also have a 3-D cross-linked network (Stejny, 1987) similar to PADC plastic material, it was decided to employ the etching conditions of PADC i.e. 6N NaOH or KOH at 70°C to see whether the material would reveal α and fission fragment tracks as expected, as well as to get an idea about track development time in comparison to PADC plastic track detector PM 250 from Pershore Mouldings, UK.

On etching the films under the above-mentioned conditions, the homo and copolymer films were found to develop α and fission fragment tracks. Figs. 3 and 4 show the photomicrographs of α particle and fission fragment tracks in the homo and copolymer materials. However the time required for the development of the charge particle tracks by chemical etching, were relatively higher compared to PM PADC track detector. The fission fragments were visible in the homo and copolymer after 1.5 and 1.0 h of etching time in 6N NaOH at 70°C , respectively. The α particles could be revealed in the homo and copolymer films only after 15 and 10 h of etching time in 6N NaOH at 70°C , respectively. Under these conditions, bulks etch rate (V_b) values for the homo and copolymer was found to be $0.51 \mu\text{m/h}$ and $1.68 \mu\text{m/h}$ whereas for PM PADC films it was $1.80 \mu\text{m/h}$.

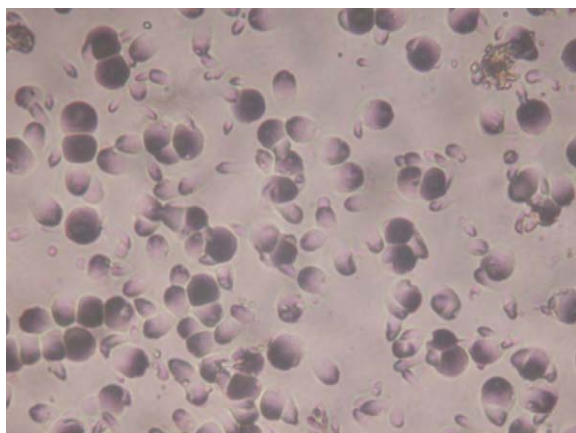


Fig. 3. Photomicrograph of ^{239}Pu α source autoradiograph in PNADAC film. Magnification: $160\times$.

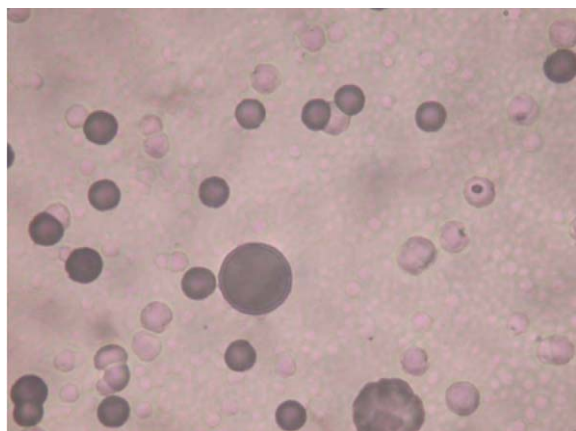


Fig. 4. Photomicrograph of ^{252}Cf fission fragments and α particles in copolymer film. Magnification: $160\times$.

The bulk etch rate for homopolymer was found to be three times lower than that of PM PADC. Thus, in order to increase V_b slightly and in turn optimize the track revelation time, it was necessary to carry out a detailed study on the conditions of etching for the newly developed charge particle detectors.

3.3. Study of the etching conditions

It is known that the conditions under which plastic detectors are etched profoundly affect the response of the plastic to the charged particles. Though, there are several etchants reported in the case of plastic SSNTDs: NaOH and KOH are the most commonly used. Since NaOH and KOH do not show any significant difference in their etching behaviour, (Charvat and Spurny, 1988) NaOH was decided to be used for all the etching studies. Generally, the changes in thick-

Table 1

Comparison of the variation in thickness of PNADAC, NADAC-ADC and PM PADC films

Polymer material	Average thickness (μm)	Standard deviation	% Variation
PNADAC (Batch I)	552.25	± 6.42	1.16
PNADAC (Batch II)	545.00	± 7.00	1.27
NADAC-ADC (Batch I)	549.00	± 7.00	1.27
NADAC-ADC (Batch II)	553.25	± 6.38	1.15
PM PADC	251.25	± 2.29	0.90

ness and weight methods are used for the determination of V_b . The change in thickness method involves the measurement of thickness at regular intervals of etching, whereas the change in weight method involves decrease in weight of the films upon etching. In order to use the thickness method the films should be of uniform thickness throughout. The films were studied for their initial thickness. It was observed that the homo and copolymer films had somewhat higher variation in the film thickness as shown in Table 1.

Taking into account the fact that the change in thickness of the films upon etching was less than the standard deviation observed in the initial thickness of the films, it was decided to use the change in weight method instead of the change in thickness method for the evaluation of the bulk etch rate (Sharma et al., 1991). Eq. (1) given below was used for the V_b measurements.

$$V_b = (M_1 - M_2) \times T_i / 2M_1 \times t, \quad (1)$$

where M_1 and M_2 are the initial and final weights of the films, t is the etching time and T_i is the initial thickness.

3.4. Optimization of etching conditions for PNADAC and NADAC-ADC plastic track detectors

Three pieces each, of size $10 \times 10 \text{ mm}^2$ from each of the two batches of the homo and copolymer films were cut and exposed to ^{239}Pu α particle source at a distance of 20 mm from the source and etched along with PM PADC. The films were etched at three different sets of temperatures 70, 75, 80 $^\circ\text{C}$ for varying concentrations of 5–8 N NaOH solutions and V_b was calculated. The results are shown in Figs. 5–7. The track revelation time for the α particles were also studied in comparison to PM PADC and are given in Table 2.

As can be seen in case of etching of the homo and copolymer films, the bulk etch rates of the newly developed homopolymer differ considerably, whereas that of copolymer show a similar trend as compared to PM PADC. The homopolymer films showed a maximum V_b of 1.4 $\mu\text{m}/\text{h}$ at 80 $^\circ\text{C}$, even at the highest temperature used. As pointed out by Amin and Henshaw (1981), it was observed that NaOH concentration above a certain value (20%) had no effect on V_b of PNADAC, copolymer and ADC.

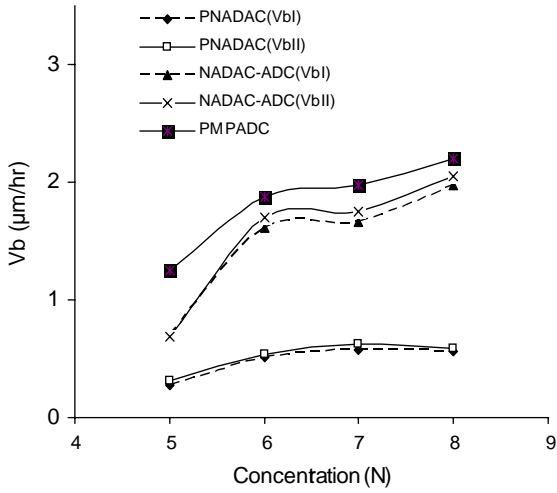


Fig. 5. Variation of the bulk etch rate of PNADAC, NADAC-ADC and PM PADC films with 5–8N NaOH at 70 °C.

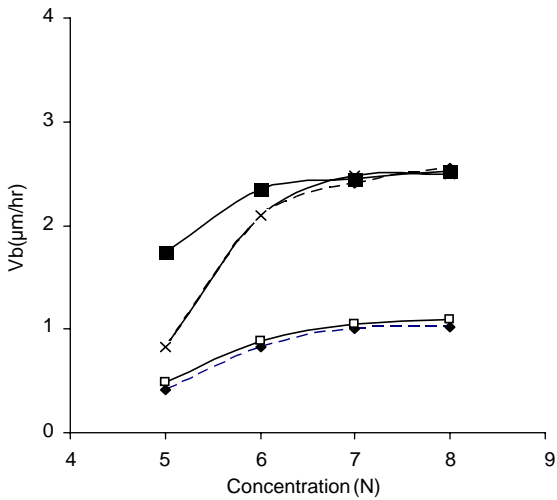


Fig. 6. Variation of the bulk etch rate of PNADAC, NADAC-ADC and PM PADC films with 5–8N NaOH at 75 °C.

Thus, the optimum etching conditions for the PNADAC and NADAC-ADC materials were chosen as 6 N NaOH, at 80 and 75 °C, respectively.

3.5. Determination of sensitivity of the homo and copolymer films

In order to find the sensitivity of α particles in the homo and copolymer films, and its dependence on the initiator concentration, the films were prepared using varying initiator concentrations of 1–5%, and were etched under similar etching conditions determined above. Figs. 8 and 9 show the variation in average etch pit diameter of the fission frag-

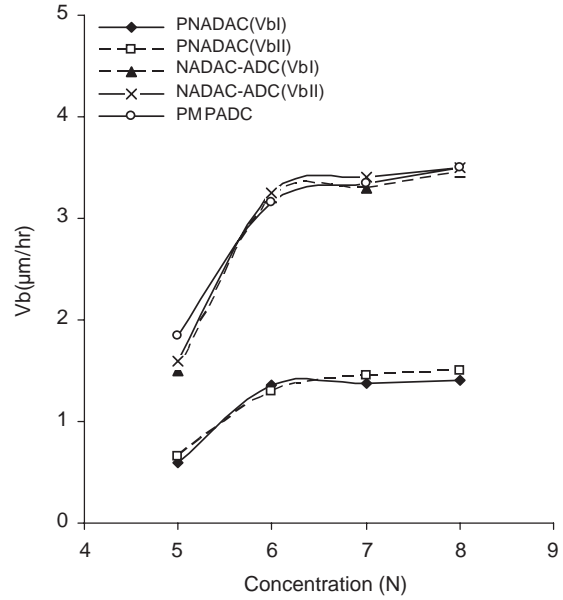


Fig. 7. Variation of the bulk etch rate of PNADAC, NADAC-ADC and PM PADC films with 5–8N NaOH at 80 °C.

Table 2

Track revelation by chemical etching for varying concentration of etchant and temperature for PNADAC, NADAC-ADC and PM PADC

Conc. of etchant (N)	Temperature (°C)	Track revelation time (h)		
		PNADAC	NADAC-ADC	PM PADC
5	70	—	14	6
6		15	10	3
7		15	10	3
8		15	10	3
5	75	17	10	5
6		10	6	2.5
7		10	6	2.5
8		10	6	2.5
5	80	12	9	5.0
6		8	5.5	2.0
7		8	5.5	2.0
8		8	5.5	2.0

ments with respect to the etching time for the homo and copolymer films. It can be seen that the response of the detector is linear with respect to the etching time for a given amount of the initiator. It is well known in the case of PADC track detector, sensitivity is dependant on the etching time. Studies pertaining to variation of the sensitivity values with respect to the initiator concentration and the optimum

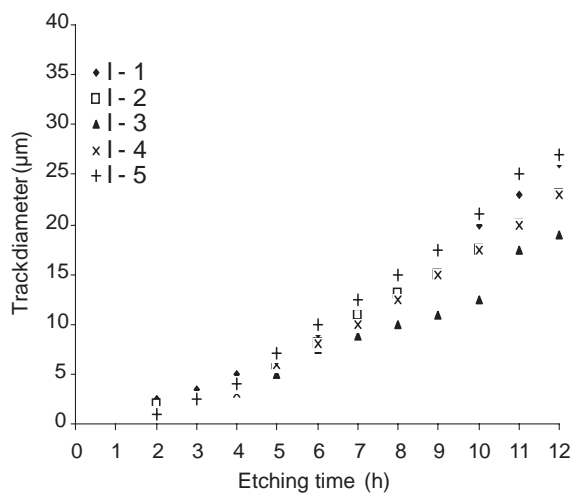


Fig. 8. Variation of the fission fragment track diameters in PNADAC films as a function of etching time using 6N NaOH at 80 °C. I-1 to I-5 indicate PNADAC films prepared using 1–5% IPP initiator, respectively.

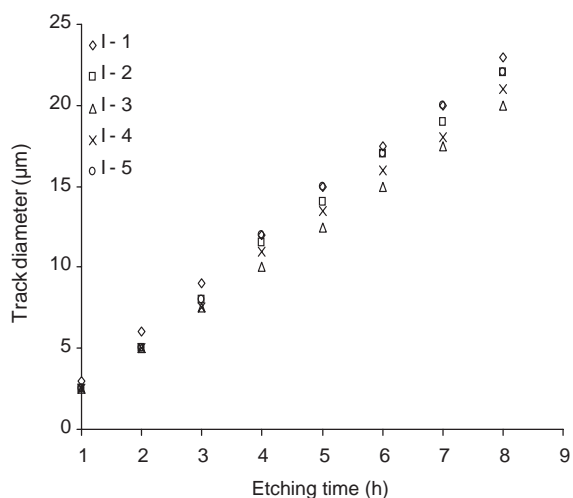


Fig. 9. Variation of the fission fragment track diameters in NADAC–ADC (copolymer) films as a function of etching time using 6N NaOH at 80 °C. I-1 to I-5 indicate NADAC–ADC films prepared using 1–5% IPP initiator, respectively.

etching time were also performed for additional two batches of homo and copolymer films; the results are shown in Figs. 10 and 11.

The sensitivity (S) and V_b were calculated using Eqs. (2) and (3). (Durrani and Bull, 1987; Manzoor et al., 1988).

$$S = V_t / V_b = (1 + (D_a / D_f)^2) / (1 - (D_a / D_f)^2), \quad (2)$$

$$V_b = D_f / 2t, \quad (3)$$

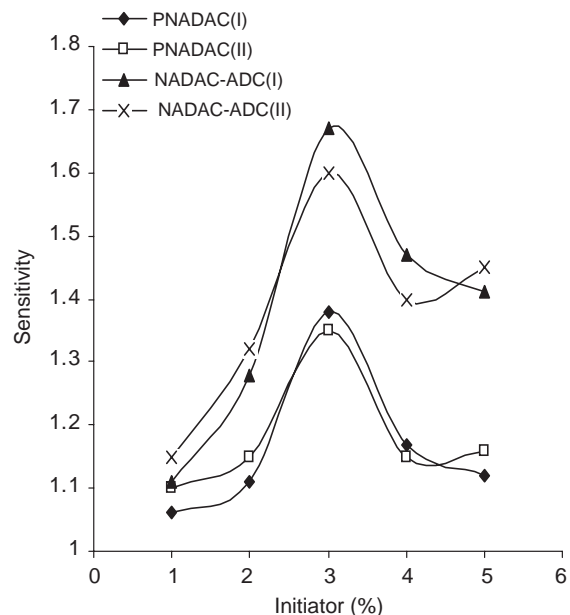


Fig. 10. Variation of the sensitivity of PNADAC and NADAC–ADC films with the amount of initiator (I and II indicate batch no.).

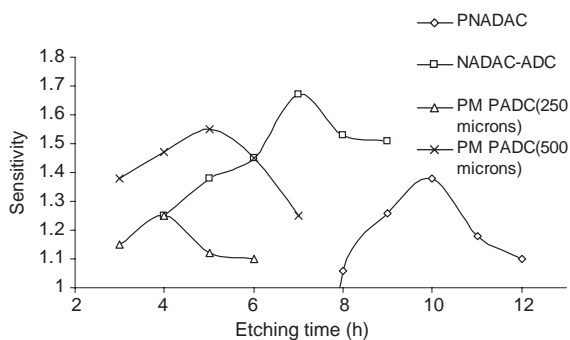


Fig. 11. Variation of sensitivity as a function of etching time at their respective optimum etching conditions.

where D_a and D_f are the track diameters of α particles and fission fragments, respectively and D_f/t is the slope of the curve plotted in Figs. 8 and 9.

From Fig. 10, it appeared that the sensitivity was maximum for films prepared using 3.0% initiator concentration. These observations are also in agreement with those of Portwood and Stejny (1984), Somogyi et al. (1986) and Nadkarni and Samant (1996). The sensitivity of homo and copolymer films prepared with 3.0% initiator concentration was found to be 1.35 and 1.60, respectively, as compared to 1.25 for PM PADC film (250 μm). A PM PADC film of 500 μm thickness had a sensitivity of 1.55. It can also be seen from Fig. 11 that the etching time for the homo and copolymer, corresponding to maximum sensitivity value, was 10 and 7 h respectively.

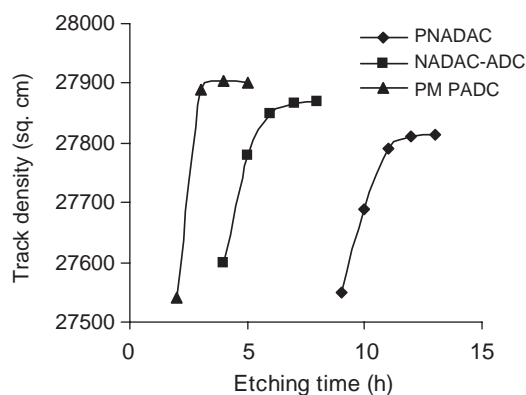


Fig. 12. Variation of α track density with etching time.

3.6. α track detection efficiency

The α track detection efficiency under 4π exposure conditions was studied. It was thus necessary to study the variation of α track density with respect to the etching time. Small pieces of films, of size $20 \times 20 \text{ mm}^2$ were exposed to 707 706 dpm source strength ^{239}Pu α particles at a distance of 20 mm from the source for 2 min. The pieces were then etched under the previously determined optimum etching conditions of homo and copolymer films. The tracks were counted till a constant value of tracks/cm² were obtained. The results were also compared with PM PADC under its standard etching conditions. From the data obtained it can be seen that the optimum etching time for the homo and copolymer is 11 and 6 h, respectively. The results are shown in Fig. 12.

The background tracks were also found and were subtracted from the actual count of tracks. It was observed that the homo and copolymer films had a high background track density compared with that of PM PADC, This was attributed to trapping of dust particles during monomer preparation and filling of the moulds. Also the process of polymerization involves the decomposition of initiators to give carbon dioxide, tiny bubbles of which may be trapped in the polymer matrix resulting in increased background track-like features.

The track detection efficiency of the PADC films towards α particle tracks was determined by exposing the films at different distances from a source, but for a fixed optimum etching time as found in the previous experiment. The tracks were then counted using an optical microscope. A total of 100 views were counted and then averaged to find the track density. The results were compared to commercially available PM PADC under similar exposure conditions. The results are shown in Table 3. It can be seen that the track registration efficiency of homo and copolymer compare well with that of PM PADC.

3.7. Measurement of α to fission branching ratios by sequential chemical etching of α and fission tracks in homo and copolymer films

Study of the sequential etching method for revelation of α and fission tracks in PADC materials have been carried out by Pandey et al. (1993). This method can be used for studies of half lives, α to fission branching ratios and identification of radionuclides, using a single detector. The α particles and fission fragment tracks can be differentiated unambiguously if there are significant differences in their size and appearance time in a given detector. The newly developed homo and copolymer materials fully meet the requirements for the sequential etching conditions due to their considerable differences in the track diameters as well as appearance time. In order to find the α to fission ratio for ^{252}Cf source the films were kept at 5 mm from the source in 2π geometry for 5 min. The results are shown in Table 4.

It can be seen that the values obtained for homo and copolymer compare well with PM PADC. This shows that the homo and copolymer films can be used unambiguously for determination of $\alpha/F \cdot F$ branching ratio.

4. Conclusion

We have designed new polymer materials to be used as SSNTDs. The new materials are found to be favourable in all the requirements of SSNTD materials, such as transparency, track development upon chemical etching with alkali metal hydroxides, viewing of the tracks under optical

Table 3
Track registration efficiency of homo and copolymers compared with PM PADC

Distance of exposure (cm)	Time (min)	Tracks expected (cm ⁻²)	Tracks observed (cm ⁻²)			Efficiency (%)		
			I	II	III	I	II	III
1	0.5	28 173	27 789	27 895	27 952	98.63	99.01	99.21
2	2.0	28 173	27 815	27 860	27 890	98.72	98.88	98.99
2.5	2.0	18 030	17 580	17 620	17 630	97.50	97.72	97.78

I, II and III indicate the homopolymer, copolymer and PM PADC, respectively.

Table 4
 $\alpha/F \cdot F$ branching ratio of ^{252}Cf in homopolymer, copolymer and PM PADC

Detector	Fission tracks per mm^2	α tracks per mm^2	$\alpha/F \cdot F$ in 2π geometry	$\alpha/F \cdot F$ in 4π geometry
PNADAC	1.48×10^4	22.37×10^4	15.11	30.22
NADAC-ADC	1.45×10^4	22.35×10^4	15.41	30.82
PM PADC	1.46×10^4	22.35×10^4	15.30	30.60

Expected value for ^{252}Cf 15.75 in 2π geometry and 31.56 in 4π geometry.

microscope and stability of the materials. The study shows that monomer(s) which can give polymers with a denser/cross-linked polymer network can act as more sensitive SS-NTD materials. Further insight on the relation between sensitivity and polymer structure may be obtained by studying polymer structure using X-ray techniques and efforts are on in that direction. The newly developed materials can also be used solely as fission fragment detectors in presence of α particles.

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