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Determining bulk etch rate of LR-115 type II SSNTD: a novel approach

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The LR-115 detector is a commonly used SSNTD for the detection and measurement of Rn^{222} and its progeny through track formation. Mostly the chemical etching technique was used for the revelation of these tracks. Etching converts a latent track into a visible structure, whose information can be read under an optical microscope. So bulk attack by the etching solution is a necessary condition and the bulk etch rate of the polymeric sample is an important factor that controls track formation in SSNTDs. Normally the bulk etch rate (V_b) for the LR-115 SSNTD was calculated by measuring the removal of the thickness of the polymeric sample for different time intervals at a constant temperature. However, in this paper a novel method of % mass change of the polymeric sample was explored to know the bulk etch rate (V_b) of the sample. The suitability of the method was checked by comparing its results with the bulk etch rate measured by another method. A good correlation was found between the two methods of measurement of bulk etch rate.

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1. Introduction

Solid State Nuclear Track Detectors (SSNTDs) are well known for the detection of ionizing radiation through track formation of heavy ionizing particles. The composition, processing, and applications of solid-state nuclear track detectors (SSNTDs) are sufficiently discussed by many researchers [1-4]. SSNTDs, particularly LR-115, are used to measure the Rn^{222} and its progeny. The red LR-115 type II detector (from DOSIRAD) is based on cellulose nitrate and is a commonly used solid-state nuclear track detector (SSNTD). The LR-115 type II detector is a commercially available red cellulose nitrate detector with an active layer of 12 μm on a 100 μm clear polyester base (as declared by the manufacturer). The chemical composition of cellulose nitrate is shown in Fig. 1 [5]. Alpha particles emitted by Rn^{222} and its progeny hit the detector and leave the latent tracks in it. Several different techniques of track revelation are known, e.g. grafting [4, 6] but, the chemical etching technique is the most frequently used in which tracks can be made visible under the optical microscope after chemical amplification via etching. Track development in an SSNTD is based on two parameters V_t (track etch rate) and V_b (bulk etch rate). As it is known that V_b depends on many factors like structure, chemical composition and preparation of the detector, etching conditions (like chemical nature of etchant, temperature, concentration as well as the stirring conditions) aging, pre-irradiation treatment and storage conditions, environmental conditions during irradiation (ionizing as well as the non ionizing radiation) etc. [7]. Various techniques have been used for the determination of the bulk etch rate [8-11]. However, the chemical etching technique was preferred to measure the bulk etch rates of LR-115, so its surface chemical etching properties should be studied in more details. Among other information, the bulk etch characteristics will be vital in the practical use of this type of SSNTD in radon measurement experiments. The present paper is devoted to determine the bulk etch rates of the LR-115 detectors (Type II, non-strippable, purchased from DOSIRAD, France) under different etching conditions by exploring a novel method of % mass change of the polymeric sample.

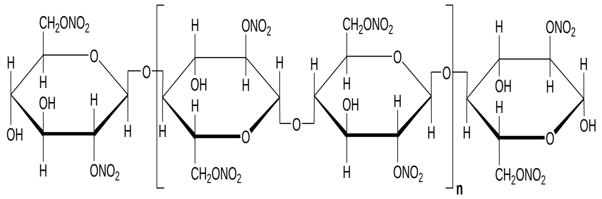

Fig. 1. Chemical formula of LR-115 (Cellulose Nitrate) [5].

2. Experimental details

Pristine LR-115 type-II (cellulose nitrate) was procured from DOSIRAD, France in the form of thin films of active layer thickness of 12 μm on a clear 100 μm polyester base as shown in Fig. 2. Several samples of size 1.5’ × 1.5’ were cut from the films and weighed initially using a digital weighing balance (Sartorious, Germany with a least count of .01mg) as shown in Fig. 3. Five pristine samples of LR-115 were installed for the chemical etching for different time intervals in the steps of 20 minutes each in a constant temperature water bath (Polltech Instruments Pvt Ltd, Mumbai) at 60°C ± 0.5°C in 1.5 N aqueous solution of NaOH without stirring as shown in the Fig. 4.

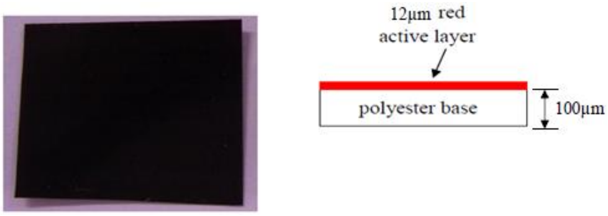

Fig. 2. (a) LR-115 sample (b) schematic of a red LR-115 detector.

Another set of five pristine samples of LR-115 were installed for the chemical etching for different time intervals in the steps of 20 minutes each in a constant temperature water bath at 60°C ± 0.5°C in 2.5 N aqueous solution of NaOH without stirring. In order to ascertain the bulk etch rate (V_b) for these samples, the samples were removed one by one after completion of the etching cycles. After each etching interval the samples were washed thoroughly in distilled water and then dried in open air for 30 minutes. The etchant was changed periodically so that concentration of etchant remained the same during the experiment. The dried and etched samples were then weighed in digital weighing balance and the % mass change was utilised to find the bulk etch rate (V_b) of the samples. However to check the accuracy of this method the pristine LR-115 samples thickness measurements were done before & after etching on a specified area as discussed by Mehta et al. and Shikha et al. [12-13]. Etch rate was determined by measuring the foil thickness using a digital micrometer (Mitutoyo, No. 293-821) having least count of 1 μm.

Fig. 3. Digital weighing balance.

Fig. 4. (a) Constant temperature water bath, (b) sample etching arrangement in the water bath.

3. Results and discussion

The bulk etching behavior of LR 115 type-II polymer was studied by measuring the % mass change in the samples after chemical etching. The sample was subjected to etching by submerging it in the chemical. The etching process starts uniformly on the whole surface layer of polymeric samples (but not on the polyester base as it is not etched by NaOH solution). The layer starts coming out of the sample and leads to the reduction in the mass of the sample. The % mass change of the sample was related to the sample thickness removed using the relation

$$\rho = \frac{mass}{volume} = \frac{mass}{l \times b \times t} \tag{1}$$

Where ρ stands for density and l, b, t stands for length, breadth & thickness respectively.

The % mass change in the samples after etching was shown in the Table 1 for etching in 1.5 N and 2.5 N aqueous solution of NaOH. Table 1 also shows the estimated thickness removed using equation (1). Fig. 5 show the graph of the estimated thickness removed of pristine LR-115 samples in 1.5 N and 2.5 N NaOH with time at 60°C ± 0.5°C.

Table 1. % mass change of pristine LR-115 samples in 1.5 N and 2.5 N NaOH with time at 60°C ± 0.5°C and estimated thickness removed.

Time (min.)	% mass change of pristine LR-115 samples		Estimated thickness removed (µm) from equation (1)	
	1.5 N NaOH	2.5 N NaOH	1.5 N NaOH	2.5 N NaOH
20	0.89	1.05	0.98	1.15
40	1.78	2.22	1.96	2.44
60	2.50	3.18	2.75	3.50
80	3.74	4.94	4.11	5.44
100	4.56	5.95	5.02	6.55

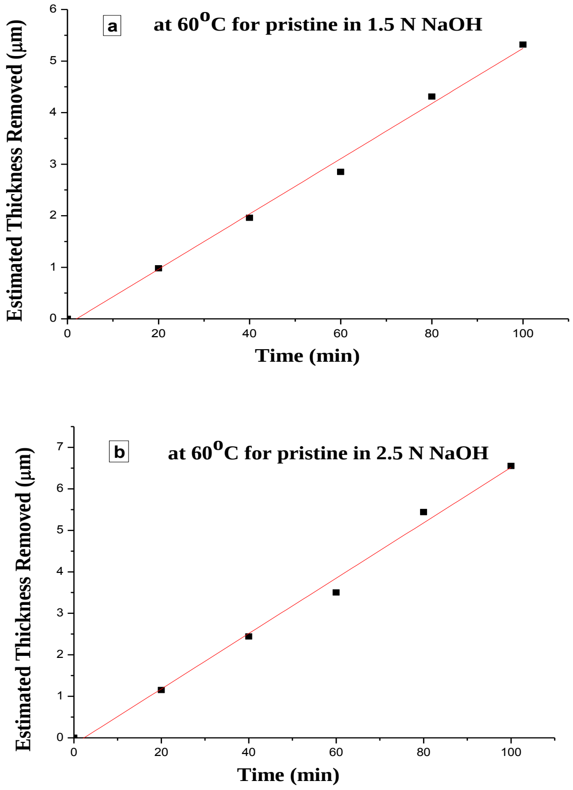

Fig. 5. Estimated thickness removed of pristine LR-115 samples in (a) 1.5 N and (b) 2.5 N NaOH with time at 60°C ± 0.5°C.

Table 2 shows the cumulative thickness removed measured by a digital micrometer. Fig. 6 show the graph of the cumulative thickness removed of pristine LR-115 samples in 1.5 N and 2.5 N NaOH with time at 60°C ± 0.5°C. Table 3 shows the coefficients (A and B) for the linear regression equation (y = A + Bx) for the relationship between estimated thickness removed from the % mass change (y) [cumulative thickness removed(y) in second method] and the etching time (x) for 1.5 N and 2.5 N NaOH at 60°C ± 0.5°C and the corresponding determined bulk etch rates (V_b in μm h^{-1}).

Table 2. Cumulative thickness removed of LR-115 samples in 1.5 N and 2.5 N NaOH with time at 60°C ± 0.5°C.

Time (min.)	Cumulative thickness removed of pristine LR-115 samples (μm)	
	1.5 N NaOH	2.5 N NaOH
20	1	1
40	2	2
60	3	4
80	4	5
100	5	7

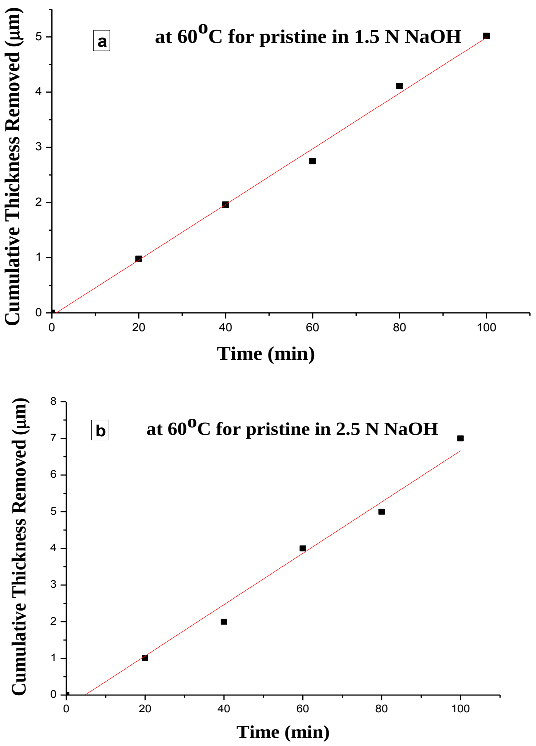

Fig. 6. Cumulative thickness removed of pristine LR-115 samples in (a) 1.5 N and (b) 2.5 N NaOH with time at 60°C ± 0.5°C.

The bulk etch rates for the pristine LR-115 was found to be 3.02 μm h^{-1} and 4.01 μm h^{-1} with the estimated thickness removed from the % mass change method in 1.5 N NaOH and 2.5 N NaOH at 60°C ± 0.5°C respectively. The bulk etch rates for the pristine LR-115 was found to be 3.00 μm h^{-1} and 4.20 μm h^{-1} with the cumulative thickness removed from the measurements done by a digital micrometer in 1.5 N NaOH and 2.5 N NaOH at 60°C ± 0.5°C respectively. A good correlation was found between the bulk etch rates determined by using two different methods.

Table 3. The coefficients (A and B) for the linear regression equation (y = A + Bx) for the relationship between the estimated thickness removed from % mass change (y) [cumulative thickness removed(y) in second method] and the etching time (x) for 1.5 N and 2.5 N NaOH at 60 °C ± 0.5 °C and the corresponding determined bulk etch rates (V_b in μm h^{-1}).

Normality	With estimated thickness change from % mass change	With cumulative thickness removed
1.5 N NaOH	A = -0.05 ±.002 B = 0.0504 ± 0.0016 Bulk etch rate(V_b) = 3.02 ± 0.09 μm h^{-1}	A = 0 B = 0.05 ± 0.001 Bulk etch rate(V_b) = 3.0 ± 0.06 μm h^{-1}
2.5 N NaOH	A = -0.15429 ± B = 0.06669 ± 0.0028 Bulk etch rate(V_b) = 4.01 ± 0.17 μm h^{-1}	A = -0.3333 ± 0.003 B = 0.07 ± 0.0044 Bulk etch rate(V_b) = 4.2 ± 0.26 μm h^{-1}

4. Conclusion

Investigation of the suitability of a new method of % mass change for determining the bulk etch rate of a polymeric sample have been reported. For checking the validity of the method another well known method of cumulative thickness removed was used to find the bulk etch rates of the same samples. Table 3 shows the results of the study by the two methods. The bulk etch rates were 3.02 μm h^{-1} and 3.00 μm h^{-1} with the two methods in case of etching in 1.5 N NaOH at $60^oC \pm 0.5^oC$. The bulk etch rates were 4.01 μm h^{-1} and 4.20 μm h^{-1} with the two methods in case of etching in 2.5 N NaOH at $60^oC \pm 0.5^oC$. These results showed a good correlation between the two methods. The % mass change method has the advantage over some other methods as it is a non-destructive method.

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