

Effects of Changing the Exposure Time of CR-39 Detector to Alpha Particles on Etching Conditions

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Abstract: In our paper, we attempt to verify the best etching conditions of CR-39 track detector given by two duration exposure times at (5 and 15sec) by ²²⁶Ra alpha source using three techniques for heating etchant chemical solvent, which are the traditional heating technique using a water bath, ultrasound and microwave. Several cases for CR-39 detector for the development of tracks and its growth with using the three heating techniques of chemical etching solvent (NaOH) are described and compared. It's found that, the maximum track densities for water bath were found to be (5659 and 12976 tracks/mm²) which were appeared at (120 and 90min) for the detector exposed at (5 and 15sec) respectively, while the maximum track densities for ultrasound were found to be (5577 and 11660 tracks/mm²) which were appeared at (120 and 90min) for the detector exposed at (5 and 15sec) respectively. The microwave is the optimum etching technique, because of the highest track densities and the very short time required (10min) to developed the tracks, which are (7051 and 13333 tracks/mm²) at the detector exposed for (5 and 15sec) respectively. Also, the chemical etching parameters for the three techniques are determined, a significant increase were observed in bulk etching rate (V_B), track etching rate (V_T) when using microwave relative to the parameters when using water bath and ultrasound. And because the increased of the etch rate of track to the bulk etch rate in the microwave technique is less than that in the other two techniques, the efficiency of the detector (η) using the microwave technique is less than the both (water bath and ultrasound) techniques.

Keywords: CR-39detector, Exposure time, Etching technique, Microwave, Ultrasound, water bath, Chemical etching parameters.

1 Introduction

Solid state nuclear track detectors are vastly used in monitoring and investigating the environmental radiation [1]. CR-39 nuclear track detector (Polyallyl diglycol carbonate, composition: C₁₂H₁₈O₇) is one of the most commonly detector used [2,3], which was used in this work because of its stability against various environmental factors, good sensitivity and high degree of optical clarity [4].

Nearly almost the (SSNTDs) applications interest the detection of α -particles emitted by natural radioactive materials. Thus, the response of track detector materials versus particles of alpha is of essential attention [1].

The fact of the operation of nuclear track detector is based on extensive ionization of the detector material due to a heavy charged particle pass through the detector [5] then registration of charged particles by formed the damages trace of the radiation along the trajectory of charged particles, which can be magnified by a chemical etching process to a visible micro-substructure (track) [6], which that if a piece of detector material containing latent tracks is exposed to some chemically aggressive solution (such as KOH or NaOH) of concentrations between 4 and 8N at temperature around 60 to 70°C for few hours, chemical reactions would be more strong along the edges of latent tracks, which is called the etchant solution [7,8].

Then the latent track becomes visible as a particle "track" by used an optical microscope [9]. The chemically

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etching of damaged regions along the particle path as a faster rate than the undamaged regions of detector material, it is leading to the visible tracks. This chemical etching (CE) procedure is an unavoidable step to show the ion-induced latent tracks in SSNTDs [5,10].

In last ten years some attempts have been made to reduce the time of etching for faster tracks development [11,12]. There are many previous studies using new techniques for heating the etchant solution such as microwave: Tripathy *et al.* (2010) [11], Hassan (2015) [13] and Kadhim *et al.* (2016) [14], ultrasound: Saeed (2014) [15], Jebur (2016) [16] and Kadhim *et al.* (2016) [14], novel temperature: Chavan *et al.* (2011) [17], and also dry etching such as plasma: Brown and Liu (1996) [18] to appearing the large number of clear alpha tracks and enable etching during a short time. Each heating technique has its inveterate advantages, but can serve as alternative to other techniques to estimate the optimum etching of nuclear track detectors [19].

The aim of this work is to study the effects of changing the time of exposed CR-39 detector to alpha particles emitted from ^{226}Ra source on accelerating the development of alpha tracks, and other etching conditions; bulk etching rate (V_B), track etching rate (V_T), and efficiency of the detector (η): using three heating techniques; water bath, ultrasound, and microwave.

2 Experimental Section

2.1 Materials and Exposure

CR-39 nuclear track detector (Peirshore Mouldings Ltd., UK, 500 μm thickness) was used in this study. Three groups of two pieces from CR-39 detector (1x1cm²) was exposed in direct contact with alpha particles emitted from ^{226}Ra source of 5 μCi activity and 4.87MeV energy. First group of the detectors were exposed to 5 sec and second group were exposed to 15 sec.

2.2 Chemical Etching

Each group of the exposed detectors was etched in (NaOH) solution with 6.25N for several selected etching time. Using three heating techniques; water bath (the traditional technique), microwave oven (LG made, Model: MS1142GW) with frequency of 2.45GHz and 6 variable power levels, and ultrasound cleaner made in china from Guangzhou Lingchian company Ltd. model: PS-D40 with ultrasonic power of 240W and frequency of 40 kHz.

For both techniques (water bath and ultrasound), the detectors were etched within volumetric flask at a temperature of 70°C, and a frequency of 40kHz for ultrasound device. For microwave a handmade quartz container shown in Fig.1, is specially manufactured to achieve etching instead of volumetric flask which damaged by microwave.

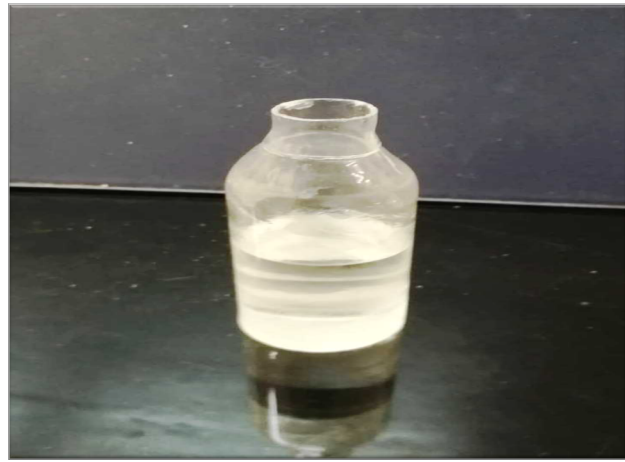


Figure 1. The quartz container used for etching in microwave.

2.3 Calculations

An optical microscope equipped with a digital camera supplied with a software program (Toup View) was used to view and count alpha tracks at CR-39 and measure their diameters. The track density (ρ) then calculated using Eq.(1) by count the mean number of the tracks in 12 views within 0.052mm² area for each view [16,20].

$$\rho = \frac{\text{The average number of total tracks}}{\text{area of field view}} \quad (1)$$

The parameters of etching: the bulk etch rate, the track etch rate, the etching rate ratio and the etching efficiency, were calculated to show the variation in their values between the three heating techniques used in this research. The etching parameters were calculated as follows [14,16]: The bulk etch rate (V_B) is one of crucial etching parameters, the rate at which the undamaged surface of the detector is removed during chemical etching is calculated using Eq.(2)

$$V_B = \frac{D}{2t} \quad (2)$$

Where D: the mean diameter of the tracks at each etching time and t is the etching time. Track etch rate (V_T), the etch rate along the latent track was calculated from the Eq.(3) [14]:

$$V_T = V_B \frac{1 + \left(\frac{V_D}{V_B}\right)^2}{1 - \left(\frac{V_D}{V_B}\right)^2} \quad (3)$$

Where V_D : the diameter rate of the tracks computed from the slope of the relation ($V_D = \Delta D / \Delta t$) between the variation of the track diameters and the variation of the etching times. Etching rate ratio (V), the sensitivity of the detector to the etching solution, calculated by Eq. (4) [14]:

$$V = \frac{V_T}{V_B} \quad (4)$$

Etching efficiency (η), the rate between the numbers of the

tracks etched in the detector to the number of α -particles incident on it [21] and computed from Eq. (5):

$$\eta = 1 - \frac{V_B}{V_T} \tag{5}$$

3 Results and Discussion

3.1 Track Density to Each Heating Device

Alpha track density was calculated for different etching times using water bath and ultrasound at two exposure times (5 and 15 sec) as shown in Table 1. The maximum track densities for water bath were found to be (5659 and 12976 tracks/mm²) which were developed at (120 and 90 min) for the detector exposed at (5 and 15 sec) respectively, while the maximum track densities for ultrasound were found to be (5577 and 11660 tracks/mm²) which were developed at (120 and 90 min) for the detector exposed at (5 and 15 sec) respectively. Thus, the optimum etching time that required developing the alpha tracks recorded at CR-39 detector when etching with water bath and ultrasound are 120 min for exposure time 5 sec and 90 min for exposure time 15 sec, where the tracks after these times were overlapped and decreased. It's clear that the alpha track densities of water bath were higher than the alpha track densities of ultrasound as shown in Figs.2-3.

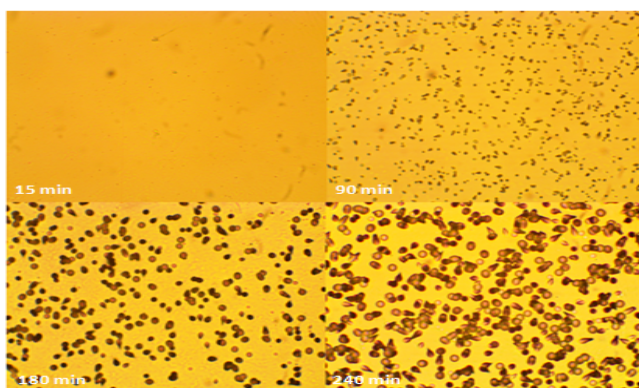


Figure 2. Views of CR-39 detector exposed for 15 sec etched at (15,90,180 and 240 min) using water bath technique.

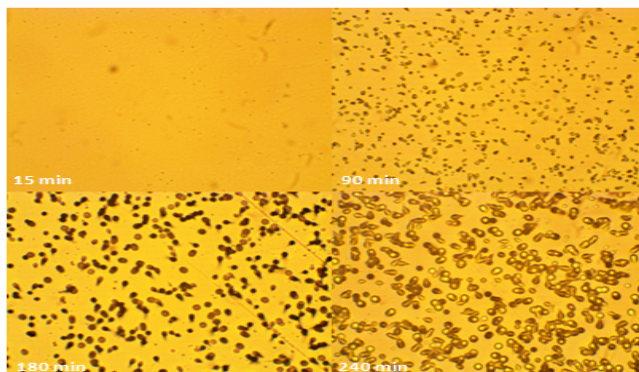


Figure 3. Views of CR-39 detector exposed for 15 sec etched at (15, 90,180 and 240 min) using ultrasound technique.

Table 1. Track densities for water bath and ultrasound versus etching times for 5 and 15 sec of exposure.

Etching time (min)	Track Density ρ (tracks/mm ²)			
	Exposure Time to Alpha Source			
	(5sec)		(15sec)	
	Water Bath	Ultrasound	Water Bath	Ultrasound
15	904	981	1712	2125
30	3687	2144	5807	6186
60	4109	3103	7942	7019
90	5284	5034	12976	11660
120	5659	5577	10385	10718
150	3567	3995	8952	7853
180	2798	3788	6870	6365
210	2548	3224	4784	5168
240	2447	2699	3956	3962
Average	3444	3394	7043	6784
Max	5659	5577	12976	11660

Table 2. Track densities for microwave versus etching times for 5 and 15 sec of exposure.

Etching time (min)	Track Density ρ (tracks/mm ²)	
	Exposure Time to Alpha Source	
	(5sec)	(15sec)
1	1737	2801
2	3795	5417
4	4288	8718
6	5282	8891
8	5968	8487
10	7051	13333
12	5782	8571
14	4827	7385
16	3949	6115
Average	4742	7746
Max	7051	13333

For microwave technique, the track density for the two times of exposure (5 and 15 sec) against different etching times is shown in Table 2. The maximum values of track

densities were developed at (10 min) were (7051 and 13333 tracks/mm²) for two times of exposure (5 sec and 15 sec) respectively.

Etching with microwave is faster rate than etching with other devices, because the quick vibration and localized heating of the polarized molecules along the ion-exposed latent tracks is leading to rapid tracks development in solid state nuclear track detectors [22]. Fig.4 showed four screens shoot captured from the digital camera of four etching times (1, 6, 10 and 16min) showing the gradually increased in density of track with the etching time until the third picture, then the track density began to decrease because the tracks were overlapped.

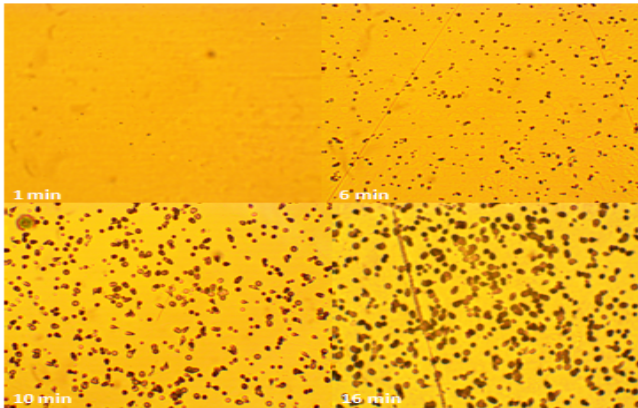


Figure 4. Views of CR-39 detector exposed for 15 sec etched at (1, 6, 10 and 16 min) using microwave technique.

3.2 Etching Parameters

The obtained results of development latent tracks showed that the diameters of the tracks were increased with the increasing of the etching time, because the effects of the etching solution become deeper on the latent tracks. Bulk etch rate calculated from Eq.(2), are listed at Tables 3-5 for water bath, ultrasound and microwave techniques for two times of exposure as follow. It is observed from the Tables 3-5 that the track diameters values were varies according to the heating technique used for etching which leads to the differences in the bulk etch rate (V_B) the average bulk etch rate of water bath, ultrasound, and microwave techniques were (1.434, 1.561 and 18.995 $\mu\text{m}/\text{h}$) at 5sec, and (1.787, 1.804 and 20.996 $\mu\text{m}/\text{h}$) at 15sec respectively.

Parameters of etching (V_B , V_T , V and η) in addition to maximum value of densities for all heating techniques were calculated and listed in Table 6 for the detectors exposed at 5sec and Table7 for the detectors exposed at 15sec. The following slopes of Figs.(5-7) were plotted between the track density and etching times for all techniques at both time of exposure to calculate the diameter rate of the tracks (V_D) to complete the calculation of (V_T) from Eq.(3).

Table 3. The etching time, average track diameter and bulk etch rate for CR-39 detector etched with water bath.

Etching time (min)	Average Track Diameter (D μm)		Bulk Etch Rate V_B ($\mu\text{m}/\text{h}$)	
	Exposure Time to Alpha Source			
	(5sec)	(5sec)	(5sec)	(5sec)
15	1.443	2.004	2.886	4.008
30	1.932	2.438	1.932	2.438
60	2.652	3.269	1.326	1.635
90	3.264	4.417	1.088	1.472
120	4.687	5.797	1.171	1.449
150	6.104	6.846	1.221	1.369
180	6.894	7.756	1.149	1.293
210	7.576	8.646	1.082	1.235
240	8.373	9.463	1.047	1.183
Average			1.434	1.787

Table 4. The etching time, average track diameter and bulk etch rate for CR-39 detector etched with ultrasound.

Etching time (min)	Average Track Diameter (D μm)		Bulk Etch Rate V_B ($\mu\text{m}/\text{h}$)	
	Exposure Time to Alpha Source			
	(5sec)	(5sec)	(5sec)	(5sec)
15	1.613	1.996	3.226	3.992
30	2.079	2.261	2.079	2.261
60	3.019	3.690	1.509	1.845
90	3.688	4.274	1.229	1.425
120	4.921	5.751	1.230	1.437
150	6.224	7.258	1.244	1.452
180	7.555	8.286	1.259	1.381
210	8.156	8.688	1.165	1.241
240	8.827	9.589	1.103	1.199
Average			1.561	1.804

Table 5. The etching time, average track diameter and bulk etch rate for CR-39 detector etched with microwave.

Etching time (min)	Average Track Diameter (D μm)		Bulk Etch Rate V_B ($\mu\text{m}/\text{h}$)	
	Exposure Time to Alpha Source			
	(5sec)	(5sec)	(5sec)	(5sec)
1	2.039	2.095	63.718	65.468
2	2.2	2.281	33.333	34.561
4	2.216	2.328	16.787	17.636
6	2.342	2.481	11.710	12.405
8	2.423	3.959	9.109	14.883
10	3.369	4.155	10.147	12.515
12	3.634	4.202	9.085	10.505
14	4.023	4.858	8.633	10.424
16	4.487	5.621	8.434	10.565
Average			18.995	20.996

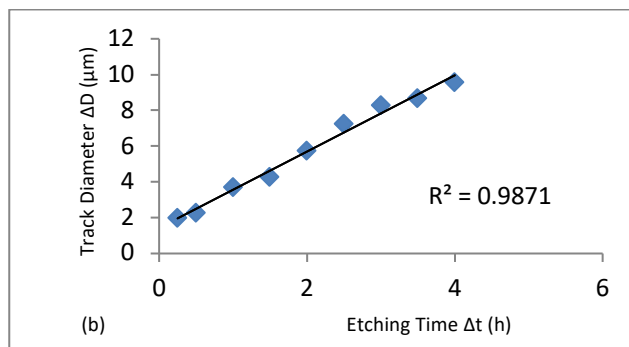
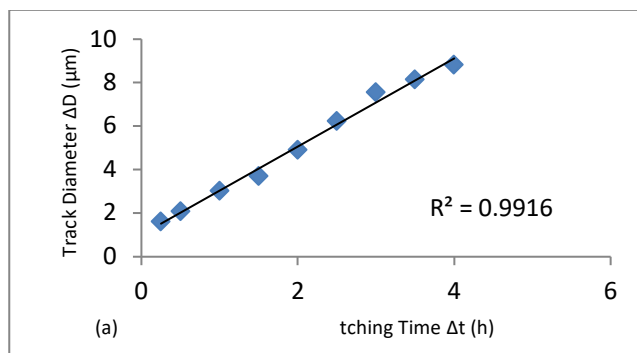


Figure 6. Slope between track diameter and etching time for CR-39 detector exposed at: (a) 5sec and (b) 15 sec etched with ultrasound.

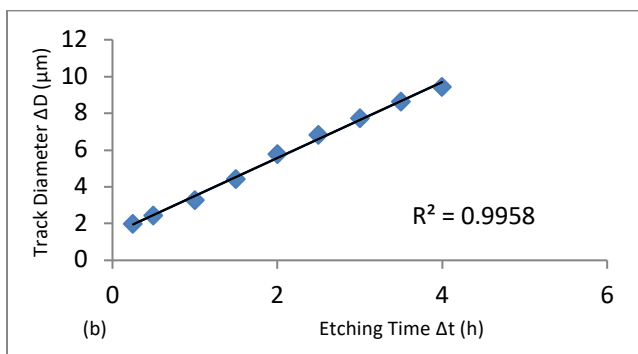
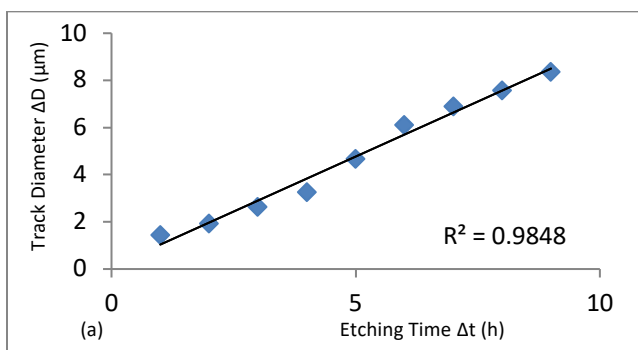


Figure 5. Slope between track diameter and etching time for CR-39 detector exposed at: (a) 5sec and (b) 15sec etched with water bath.

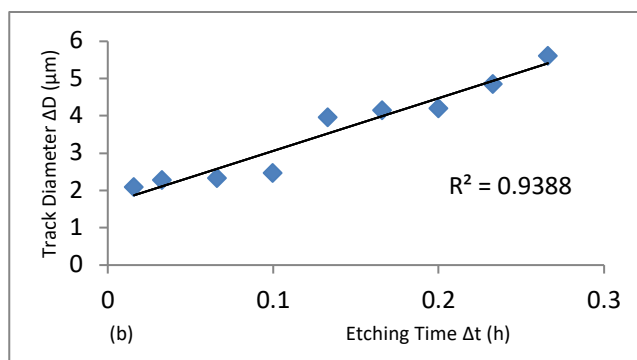
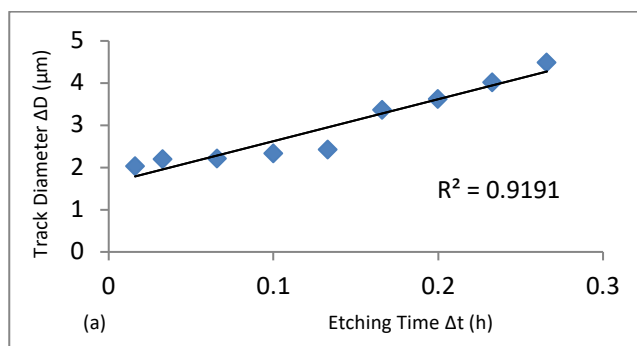


Figure 8. Slope between track diameter and etching time for CR-39 detector exposed at: (a) 5sec and (b) 15sec etched with microwave.

Table 6. Etching parameters of CR-39 detector exposed at 5sec.

Etching Method	ρ_{\max}	V_B	V_T	V	η
Water Bath	5659	1.434	4.946	3.451	0.711
Ultrasound	5577	1.561	6.122	3.922	0.745
Micro Wave	7051	18.995	33.357	1.756	0.431

Table 7. Etching parameters of CR-39 detector exposed at 15sec.

Etching Method	ρ_{\max}	V_B	V_T	V	η
Water Bath	12976	1.786	12.435	6.959	0.856
Ultrasound	11660	1.803	10.866	6.024	0.834
Micro Wave	13333	20.996	55.891	2.661	0.624

The two tables above clarify that the parameters of etching (V_B , V_T , and V) of CR-39 detector had a significant increase when etching with microwave relative to ultrasound and water bath, this is attributed to the very short time (10min) for both two times of exposure (5 and 15sec) at which the latent tracks were developed comparing to the relatively long time (90 and 120min) for two times of exposure (5 and 15sec) respectively at which the tracks were developed when etching with water bath and ultrasound. The most important parameter is the etching efficiency of the detector, as its value for microwave was less than its values for water bath and ultrasound, where (0.431 and 0.624) for microwave at (5 and 15sec) respectively, and for water bath and ultrasound at (5sec) were (0.711 and 0.745) respectively, and were (0.856 and 0.834) for water bath and ultrasound at (15sec) respectively. And because the increased of the track etch rate to the bulk etch rate in the microwave is less than that in the water bath and ultrasound, the efficiency of the detector (η) using the microwave technique is less than the other two techniques.

4 Conclusions

From the results of this research, we obtain the following conclusions:

1. There is no effects of the exposed time on the etching speed in CR-39 detector with the three used heating techniques in this work.
2. The first appear observe for tracks faster when etching with microwave, which about 1 minute at the both exposure times (5 and 15sec).
3. Tracks are appearing in ultrasound clearly more than in water bath and start at the same etching time (15min) at the both exposure times (5 and 15sec).
4. In this work, the effects of the exposed time on the tracks density was observed, where the densities increase with the alpha particles exposure time increase.
5. The maximum value of track densities appear when

etching with microwave, water bath and ultrasound respectively.

6. The optimum etching time for CR-39 detector is same for water bath and the ultrasound which are (120min at exposure time 5sec and 90min at exposure time 15sec), and for microwave is (10min at both exposure times 5 and 15sec).

7. The tracks size (tracks diameter) are increasing with etching time for the detector and with the exposure time increase.

8. In this research, it was found that the values of the etching parameters when using the microwave for heating are higher than about ten times using ultrasound or water bath, which confirms the conclusion (2.).

9. The efficiency of the detector (η) using the microwave technique is less than the other two techniques.

10. Heating glass volumetric flask is suitable container for chemical etching with water bath and ultrasound, but it's damaged when using with microwave. Quartz container manufactured in this study is suitable for chemical etching CR-39 detector with microwave.

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