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Comparison and Study of the Bulk Etch Rate of the Detector CR-39 using Two Etching Solutions

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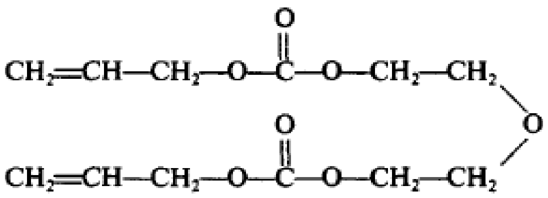
Abstract

In this research, we studied the effect of two types of etching solutions on the bulk etch rate(V_B)of the nuclear track detector CR-39. The first type of solution was prepared from dissolving NaOH in (ethanol+water) by different normalitiesat temperature 55ºC and compared with the second type of the solution prepared from dissolving NaOH in (methanol+water) by different normalities at temperature 55ºC.The method of direct measurement of the thickness of theremoved layer by the chemical etching of the detector was used in finding the bulk etch rate values. It was found the values of the bulk etch rate by using the etching solution NaOH/methanol+water greater than the values of the bulk etch rate by using the etching solution NaOH/ethanol+water, Their values have ranged between (4.48-10.62)um/hr for NaOH/ethanol+water and (15.64-35.51)um/hr for NaOH/methanol+water.

Keywords: *CR-39;Bulk etch rate;NaOH+ethanol+water;NaOH+methanol+water.*

1. Introduction :

CR-39 detector belongs to organic detectors, it is the most popular member of the SSNTD's family. This plastic is thermoset, cross-linked, totally amorphous and very sensitive to heavy ion damage [6,7]. CR-39 is prepared by the polymerization of a liquid monomer diethylene glycol bis(allyl carbonate) [29]



Diethylene glycol bis(allyl carbonate)

Also CR-39 are characterized by being hard, infusible and insoluble in all solvents. In addition to the general thermoset characteristics , CR-39 is a high-grade optical plastic whose refractive index is just slightly less than that glass. It is extremely hard yet brittle, resistance to almost all chemicals except strong bases and highly oxidizing acids[4,10,19].

Chemical etching is the most common method being used in various studies of surfaces and technical applications[15]. The response of the solid state nuclear track detectors(SSNTDs) depends strongly on the etching condition and etchant characteristics. Since then, efforts have been made to introduce a better etchant having improved etching parameter such as V_T, V_B, E_T, E_B, etch induction time [1].

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The bulk etch rate V_B is the rate of removing of the undamaged surface of the detector due to the chemical reaction between the etching solution and the detector material, Which reduces the thickness with the progress of the process of etching [25,33]. Since V_B is one of the most important parameters that control the formation and development of tracks, It has been shown that V_B depends on many factors like the purity of the basic substances. The homogeneity of the detector material and its isotropic, the molecular structures of polymers, polymerization conditions as well as its dependence on a number of environmental factors during detector irradiation. The process of swelling is considered due to the absorption of detector to the solution during the process of etching. one of the important factors that affects the measurement of the bulk etch rate. The etching materials not only remove the general surface, but also cause swelling in the detector. The amount of swelling depends on the type of polymeric detector and its polymerization conditions as well as weather factors such as humidity, dehydration and temperature change during storage [30,4,20,24,12,7,22].In addition to the V_B is being detecting parameter, it is also an important etching parameter, It also depends on the type of chemical solution and its normalities and its temperature[3,25,31].

It is known that the value of V_B does not depend on the etching direction under experiment conditions in homogeneous and isotropic materials such as the CR-39, PM-355, LR-115, CN-85, etc., because the V_B value is constant for all etching directions on the general surface of the detectors, have two values depending on the direction of the non-homogenous and non-isotopic detection surface[2].There are different methods have been established for the determination of the bulk etch rate: One of the methods is used in the measurement of V_B is the method of removed thickness from the surface of the detector. this method is called direct method, and is done by measuring the thickness of the detector before the etching process and after and for successive etching time, the method is used in this research, V_B can be calculated from the relationship[12].

$$V_B = \frac{1}{2}\frac{\Delta h}{\Delta t} \quad (1)$$

Where: h is the removal layer thickness (µm), t is etching time(hr).

There is another method called method of gravimetric, and depends on the measurement of the detector mass before and after etching, V_B can be calculated from the relationship:

$$V_B = \frac{1}{2\rho A}\frac{\Delta m}{\Delta t} \quad (2)$$

where Δm is mass difference, A the etched surface area, ρ the density of the detector and t is the etching time.

In addition to that, there are other methods such as the method of fission fragments using the irradiated source ^{252}Cfand the rate of etching average is high in this case, V_B can be found by direct measurement of fission fragments D_f(µm) at the time of etching t(hr) using the following relationship[12]:

$$V_B = \frac{D_f}{2t} \quad (3)$$

There is another method to measure the bulk etch rate , It is a method of measuring the track's diameter-length(L_e-D), V_B which can be calculated from the relationship[23,5]:

$$V_B = \frac{D^2}{4tL_e}\left[1+\sqrt{1+\frac{4L_e^{\,2}}{D^2}}\right] (4)$$

$$D = 2\ V_B\ t\sqrt{\frac{(V_T - V_B)}{(V_T + V_B)}} \ (5)$$

Where L_e is track length, D is the cone diameter, t is the etching time.

It was found that the bulk etch rate V_B and track etch rate V_T are affected by the change in the normality of the etching solution depending on the relationship[27]:

$$V_B = f_B C^n e^{(-\frac{E_k}{kT})} (6)$$

Where $f_B$is the constant depends on the type of material, C is the concentration of the etchant in the unit of normality, k is the Boltzman's constant, T is the temperature in K.

2. Methodology

CR-39 detector of 200µm thickness was used from TASKRAK (Track Analysis System Ltd.(TASL), Bristol, UK)and have been prepared in pieces of 1×1 cm^2.The edges are fine-polished to get rid of the scratches and to make them sharp and clear for precise measurements of the detector thickness and then the bulk etch rate(V_B). These detectors were then etched in NaOH/70% ethanol+30%water and NaOH/70% methanol+30%water at normality of (1.5 , 2 ,2.5 ,3)N ,at a temperature of 55ºC.The etching was done in a bottle with a tight cover to prevent change in the concentration of the etching solution due to vaporization of ethanol and methanol and absorption of moisture. For an etching process, a water bath (Memmert type W200)of stabilizing temperature with accuracy ±1C was used. After each etching step, detectors were washed with distilled water and dried using a thermal oven type (elektro.mag – M 420)at 50 ° C for a quarter of an hour. To begin the process of microscopic observation to measure the removal layer thickness with a magnification of 100× attached to digital camera [MDCE-5A] connected to a computer. The bulk etch rate V_B is measured by the change of the thickness of the detector(direct measurement) using the relation(1).

3.Results and discussion

The Bulk etch rate (V_B)is considered a fundamental parameter in the formation of the track, where it is an etching and detecting parameter ,The final effect of the etching process is the removal of the material from the detector surface[3,15,25]. In our study, we used new etching solutions NaOH/ethanol+water and NaOH/methanol+water to reduce etching time compared to the conventional etching 6 N NaOH/water at 70ºC and use these solutions in many applications.

3.1.Bulk etch rate of the CR-39 in NaOH/ethanol+water :

Bulk etch rate of CR-39 was determined in different concentrations of NaOH/ethanol+water (1.5,2,2.5,3)N at temperature 55ºC.To avoid evaporation of ethanol due to its lower boiling point, etching was carried out at lower temperatures[26].

As previously mentioned, there are different methods to measure bulk etch rate (V_B),we are used a direct measurement of thickness, the removal layer thickness was measured for successive etching times and for each normality of solution. Figure (I) represents the removal layer thickness from the detector surface for successive etching times for each concentration of the etching solution. A linear relationship was found between the removal layer thickness and etching time. Increasing the time of etching, increases the reaction time between the detector and the etching solution and thus increases the amount of degradation of the detector material from the surface, thereby increasing the thickness of the layer removed from the surface of the detector.

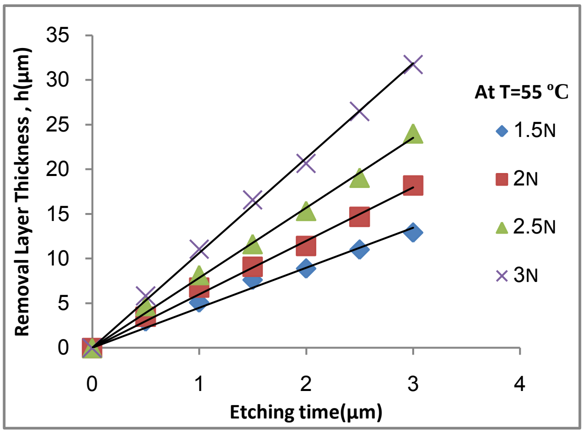
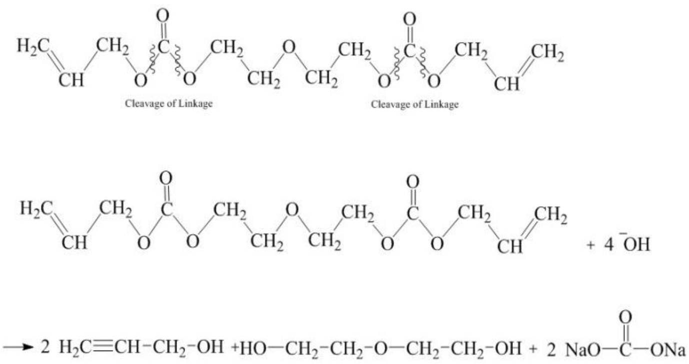


Figure I
Change the removal thickness layer with etching time in CR-39 detectors etched in NaOH/ethanol+water at 55°C with different concentration

Although both etching solution NaOH/water and NaOH/ethanol+water has the same etching mechanism, The treatment of the nuclear track detector CR-39 with basic solutions leads to scission of the carbonate ester bond by hydroxide ion through hydrolysis of ester, The following reaction shows the position of cleavage by hydroxide ion (OH^-)[16,32] :



The large increase in the bulk etch rate using NaOH/ethanol+water solution compared to the bulk etch rate using NaOH/water, this is due to the miscibility of ethanol with the organic products of the CR-39 during the etching solution. Also the production rate of sodium carbonate in NaOH/ethanol+water during the etching process is greater than NaOH/water and a layer of sodium carbonate precipitate was accumulated on the surface of the CR-39 due to the insolubility of sodium carbonate and saturation in ethanol+water, which increases the amount of removal material from the surface of the detector[9,32,8].

Table 1
V_B for CR-39 as a function of normality of NaOH/ethanol+water

Normality	V_B
1.5	4.48
2	5.99
2.5	7.83
3	10.62

Figure II show values of V_B increase with the increasing concentration of the etching solutionNaOH/ethanol+water and note this increase is exponential follows the equation(6).

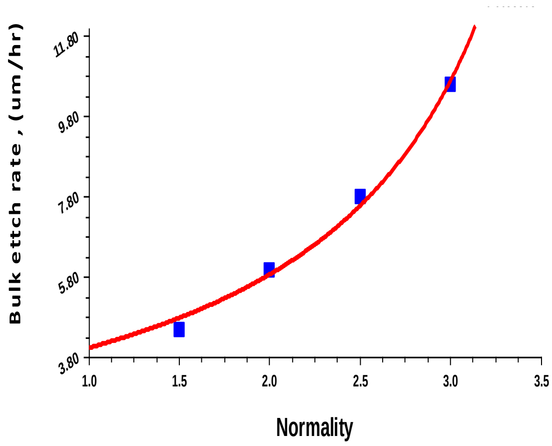


Figure II
Change the bulk etch rate VB with the concentration of the etching solution of the NaOH/ethanol+water at 55°C

3.1.Bulk etch rate of the CR-39 in NaOH/methanol+water :

Bulk etch rate of CR-39 was determined in different concentrations of NaOH/methanol+water (1.5,2,2.5,3)N at temperature 55ºC, Figure III show the removal layer thickness in NaOH / methanol + water is greater than NaOH / ethanol + water. This can be explained by the polar force, which shows that the methanol polarity (5.1) is greater than the ethanol polarity (4.3) [13].

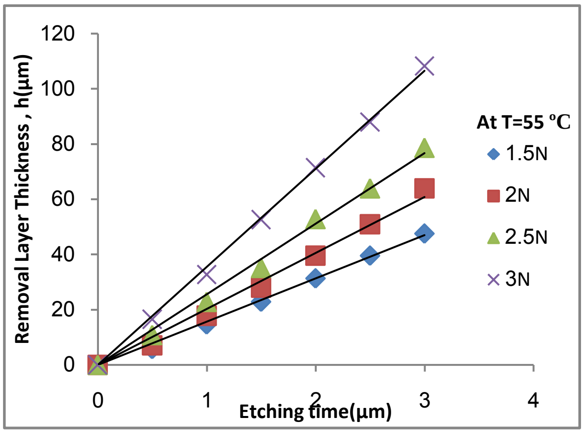


Figure III
Change the removal thickness layer with etching time in CR-39 detectors etched in NaOH/methanol+water at 55°C with different concentration

Table 2
V_B for CR-39 as a function of normality of NaOH/methanol+water

Normality	V_B
1.5	15.64
2	20.28
2.5	25.58
3	35.51

Figure IV show values of V_B increase with the increasing concentration of the etching solution NaOH/methanol+water and note this increase is exponential follows the equation(6).

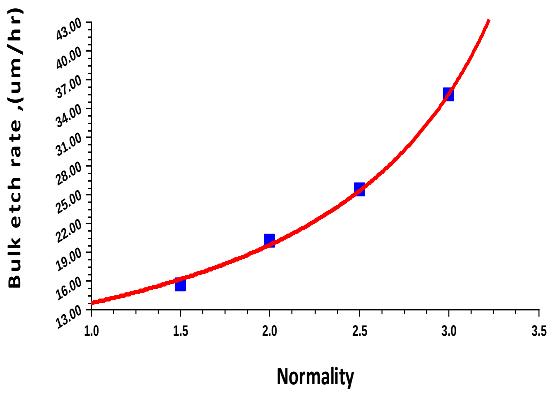


Figure IV
Change the bulk etch rate VB with the concentration of the etching solution of the NaOH/methanol+water at 55°C

3.3 Application:

From tables (1) and (2) we find the that values of the bulk etch rate V_B when using NaOH/ethanol+water and NaOH/methanol+water solutions are large and can be used in many fields; Clean the detector from the background of radiation before use in experiments and measurements, especially in the measurement of radon concentrations.It is also used in the process of thinning the detector to thickness less than (h<20µm) in order to be used in experiments to study the growth of biological cells[8].These solutions can be used prior to electrochemical etching to reduce sharp surface defects[17].These solutions are also used to improve the efficiency of the scanning for ultraheavy cosmic ray pathways as well as cleaning the detector from the background of heavy radiation when the detector exposure to thermal neutrons intensities above 10^{14} n cm^{-2} [4].

Conclusions

Throughout this research, we inferred that using two etching solutions NaOH/ethanol+water and NaOH/methanol+water gives good results for bulk etch rate and with greater values in short duration etching and can be used in many fields: Nuclear physics , radiobiological, space physics, cosmic rays.

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