

Using (Methanol + NaOH + Water) solution as an Etchant for CR-39 Detector

S. F. AL-Najjar

Department of Basic science, Dental College / University of Mosul, Iraq

ABSTRACT

Aims: In this work have been used new etchants for which CR-39 detector has relatively higher efficiency and shorter etching time. These included (Methanol + NaOH + Water) solution, at different concentration, etching temperatures and etching time.

Materials and methods: Efforts were made to different etchants for the CR-39 detector which have a higher efficiency and shorter etching time, this was desirable because the chemical etching techniques are use longer time. In this study we use an etchant The concentration of methanol 40% to 80% and water 10% to 50% and NaOH was 10% the etching time between was 5 to 30 min at three different temperature 55, 60 and 65 °C.

The results: obtained that track diameter increased linearly with an increasing percentage of methanol concentration in etchant solution consisting (methanol 70% + water 20% + NaOH 10%) at 55°C with the was observed maximum efficiency of 83%, The bulk etch rate V_B and track etch rate V_T and the critical angle θ_c were calculated.

Keywords: CR-39 Detector, chemical etching techniques, new etchant, Methanol.

INTRODUCTION

CR-39 is one commonly used as solid-state nuclear track detector SSNTD ($C_{12}H_{18}O_7$, $\rho = 1310 \text{ kg/m}^3$) is widely used in the field of health physics, such as for radon monitoring and has been used in many fields of science and technology. Basic operation of the CR-39 is based on the fact that charged particles such as alpha particle will cause ionization for almost all molecules close to its path through a medium, this primary ionizing process triggers a series of new chemical processes that result in the creation of a zone enriched with free chemical radicals and other chemical species, this damaged zone is called a latent track. then formed etch tracks in the CR-39 can seen under an optical microscope different etchants can be used for the growth of the tracks for different detectors, but for plastics, the aqueous solution of NaOH and KOH with different concentrations 4-8 normality is the most frequently used, at temperature employed for it is 70°C. It is necessary to control the temperature of the etchant ($\pm 1^\circ\text{C}$). The time for etching varies from 3 hours to 12 hours, but for high efficiency and accuracy long etching time is preferable^(1,2). Aqueous solution of the hydroxides of alkali viz. NaOH, LiOH, and KOH have been extensively studied as etchants of CR-39 detector as etchant⁽³⁾.

A new etchant were studied with an aim to find etchants that would yield desirable results, As a need four new etchants were discovered, these included NaOH dissolved in 1-propanol, NaOH dissolved in Ethanol, NaOH dissolved in Methanol + Water⁽⁴⁾.

Chemical etching is the most common method used for enlarging the size of the latent tracks, the solution attacks the damaged core of the track and penetrates along its length with a velocity V_T , while the surrounding undamaged material is attacked at a lower rate of V_B , the bulk etch rate V_B is generally constant for given etching conditions, the detectors track registration efficiency larger depends on the composition and concentration of the chemical etchant, temperature, etching time. Various methods have been proposed or employed for determination of the bulk etch rate of SSNTDs⁽⁵⁾.

For example, one relied on the difference between the detector masses before and after etching, and another was based on measurements of track diameters, generally, the following techniques are employed for the bulk etch rate of the detector, thickness measurement method, Track diameter method, and Change in detector mass method^(6,7). In this work have been used new etchants for which CR-39 detector has relatively higher efficiency and shorter etching time. these included (Methanol + NaOH + Water) solution, at different concentration, etching temperatures and etching time, In order to find an optimum values of etching temperature and to determine V_B , V_T , V and etching efficiency η .

MATERIALS AND METHODS

In the present work, CR-39 detectors samples of dimensions 0.5 x 0.5 cm² and a thickness 1.5mm cut from large sized material delivered by Tasc Ltd UK, were employed. The irradiated CR-39 SSNTDs were prepared by irradiation with 3.5MeV alpha particles from an ²⁴¹Am alpha-particle source for 3 min using system resolute for this purpose. For good results, it is important to control the quality of the etchant. High concentration of the etch product is built up after prolonged use of the etchant and it is therefore, necessary that fresh etchant should be used frequently. After etching the bulk etch rate V_B was measured from the change in thickness method for all the detector⁽⁸⁾.

The etchant solution was (Methanol + Water + NaOH), this solution contain 10% NaOH dissolved in X% methanol and (90-X)% water, after each 5 minutes etching step, detectors was washed under running tap water and then with distilled water⁽⁴⁾. Diameters of the alpha particle tracks were measured using an optical microscope (Nikon) with magnification of 400X, track measurement were recorded for track diameter used a digital camera (Digital Camera) type MDCE-5A.

For determination of V_B the thickness measurement method was used, the bulk etch rate is given from eq.1⁽¹⁰⁾. Where ΔX is the thickness variation after etching time Δt . It is assumed that the bulk etching is the same on both sides of the detector by plotting ΔX as a function of etching time t and by a given fit of the data point the bulk etch rate be determined. The formation of teachable tracks in a detector depends on certain critical etching parameters like bulk etching rate V_B and track etch rate V_T which must be experimentally determined. From the knowledge of V_B and V_T etching efficiency (η) for etchant used can be calculated. To determine V_B , V_T , V_D , V , θ_c and η , the following equations were used. Where Δx is the thickness removed from both sides of the detector during an etching time t and θ_c the critical angle of etching and V_D ^(11,12,13)

$$V_B = \frac{1}{2} \frac{\Delta x}{\Delta t} \text{ --- (1)}$$

$$V_T = V_B \frac{(4V_B^2 + V_D^2)}{(4V_B^2 - V_D^2)} \text{ --- (2)}$$

$$V_D = \frac{D}{t} \text{ --- (3)}$$

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

$$\theta_c = \sin^{-1} \left(\frac{1}{V} \right) \text{ --- (5)}$$

$$\eta = 1 - \sin(\theta_c) \text{ --- (6)}$$

RESULTS AND DISCUSSION

Fig's (1), (2) and (3) shows the average track diameter for CR-39 detectors that were etched in five different normality of 40%-80% methanol and 50%-10% water and 10% NaOH solution the etching time 5-30min at (55,60 and 65) °C, it can be seen that the track diameter increased with the increasing etching time and increasing the concentration of the methanol in the etching solution, The effect of increasing temperature of etching solution can also be seen upon the track diameter. The fitting of experimental data plotted in Figs(1,2,3) explained the sharply increasing of track diameter with an increase etching time and the etching temperature, the track apparent time was 15min when the concentration of methanol was 50%, but when the methanol concentration reached 80% in the etching solution the track apparent time was 5min and disappearance time of tracks was 30min for three etching conditions that means the precipitate formed at the surface of CR-39 detectors during etching in (Methanol + Water + NaOH) has also shown that sodium carbonate is present in the precipitate. Sodium carbonate is the disodium salt of carbonic acid. It is a gray-white powder that is soluble in water but not in organic solvents. Comparing the bulk etch rates of CR-39 detector in sodium methoxide (Methanol + Water + NaOH), the production rate of sodium carbonate in the latter one was larger. Due to the insolubility of sodium carbonate and its saturation in Methanol, a layer of precipitate was accumulated on the surface of CR-39, which was absent when the etchant NaOH/H₂O was used⁽¹²⁾.

Fig's(4,5,6) shows the average removal thickness as a function of etching time for CR-39 detectors were etched with different normality, as expected V_B obtained by linear fitting of experimental data obtained for thickness-measurement method, we are seen increase V_B with etching concentration of methanol in the etching solution and etching time, and we can see the bulk etch rate increased with increasing as the temperature of etching solution increased. Fig(7) shows the change in etch rate ratio V with methanol concentration in the etching solution at (55,60,65)°C the etch rate ratio grows gradually up to maximum value and then decreases with increases the methanol

concentration. Other, important parameters critical angle of etching have the inverse behavior of the etch ratio V show in fig(9). From fig(8) observe the removal thickness increase with methanol concentration at time etching 30 min and increase with increase temperature.

The Efficiency η are the most important parameters in choose of an etchant for CR-39 detector, fig (10) show the Efficiency η as a function of methanol concentration in the etching solution of CR-39 detectors at different temperature. It is interesting to note that the maximum Etching Efficiency $\eta\%$ found on approximately 84% at 70% methanol concentration in etching solution when etching at 55 °C, the optimum etching condition of the solution using in this research (methanol + water + NaOH) with five different concentration and is when etching in (70% methanol + 20% water + 10% NaOH) and the solution temperature 55°C with time of etching 15min. over 6.25M NaOH aqueous solution where several hours of etching would have been required to get similar track diameter.

Table(1.2.3) show the values of V_B , V_T , V , θ_C and η for alpha particles in CR-39 detector using various concentrations of Methanol in etching solution at (55,60,65)°C. The relationship for the efficiency of track increase with the increasing of etching time the maximum values at methanol concentration 70% in the etching solution, at etching temperature (55 and 60) °C, but at 65°C the maximum value of efficiency at methanol concentration 60% then the value of efficiency decreased. From fig(11) show typical images of CR-39 detectors etching at 55°C captured under the optical microscope. We can see the diameter increase with increase etching time. fig(11) Typical images of CR-39 detectors etching at 55°C captured under the optical microscope.

CONCLUSION

The etchant solution using in this research (methanol + water + NaOH) are more efficient than normally used 6.25M NaOH at 70°C, this etchant also have the advantage that they require much shorter time. The foreground of these etchants in usual dosimeter will processing time from hours to minutes for the CR-39 detector.

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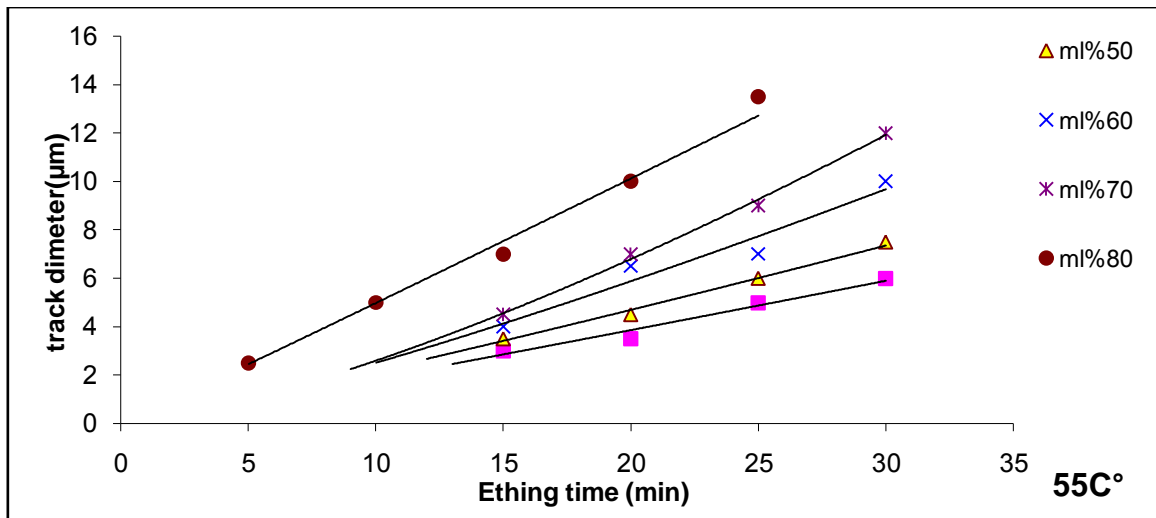


Fig (1) Alpha particle track diameter as a function of etching time for CR-39 detectors were etched in Methanol+ Water+ NaOH with different normality at 55°C.

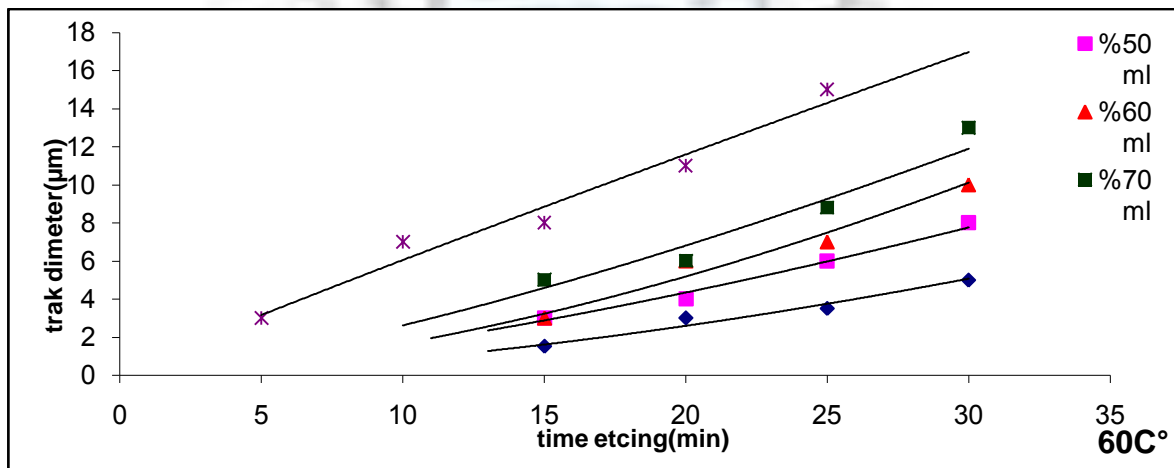


Fig (2) Alpha particle track diameter as a function of etching time for CR-39 detectors were etched in NaOH+Methanol+Water with different normality 60°C.

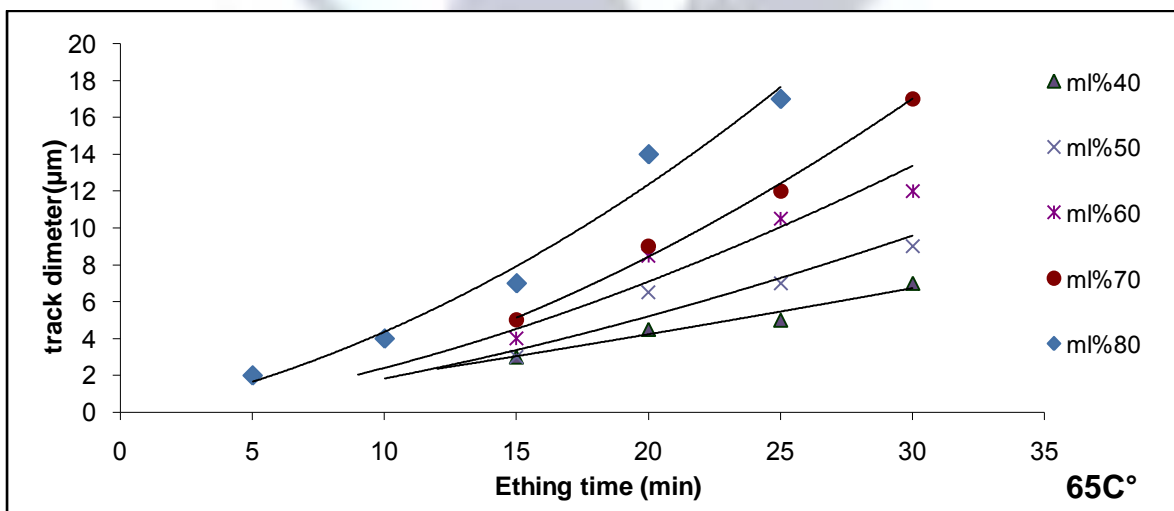


Fig (3) Alpha particle track diameter as a function of etching time for CR-39 detectors were etched in Methanol+ Water+ NaOH with different normality at 65°C.

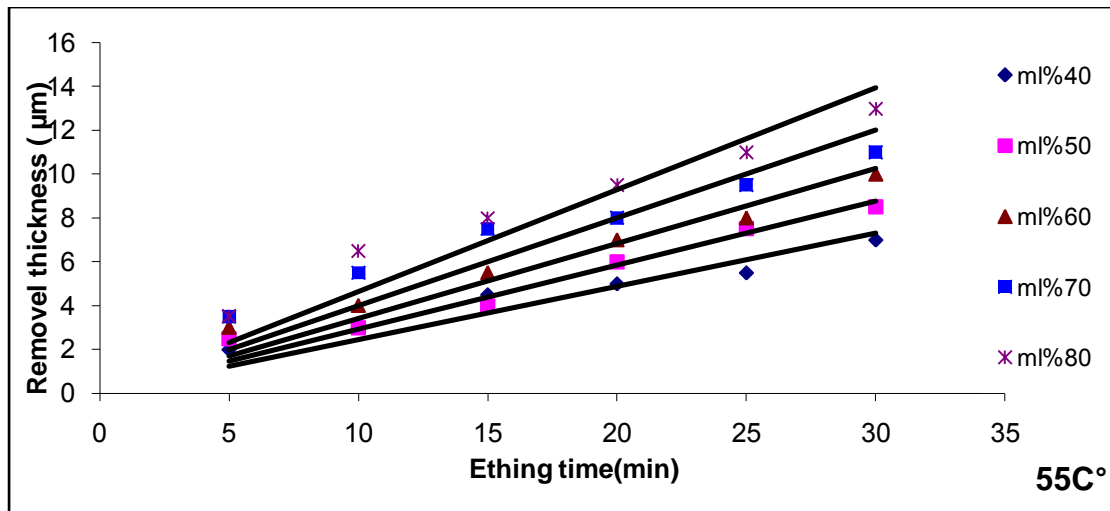


Fig (4) Removal thickness as a function of etching time for CR-39 detectors were etched in Methanol+ Water+ NaOH with different normality at 55°C.

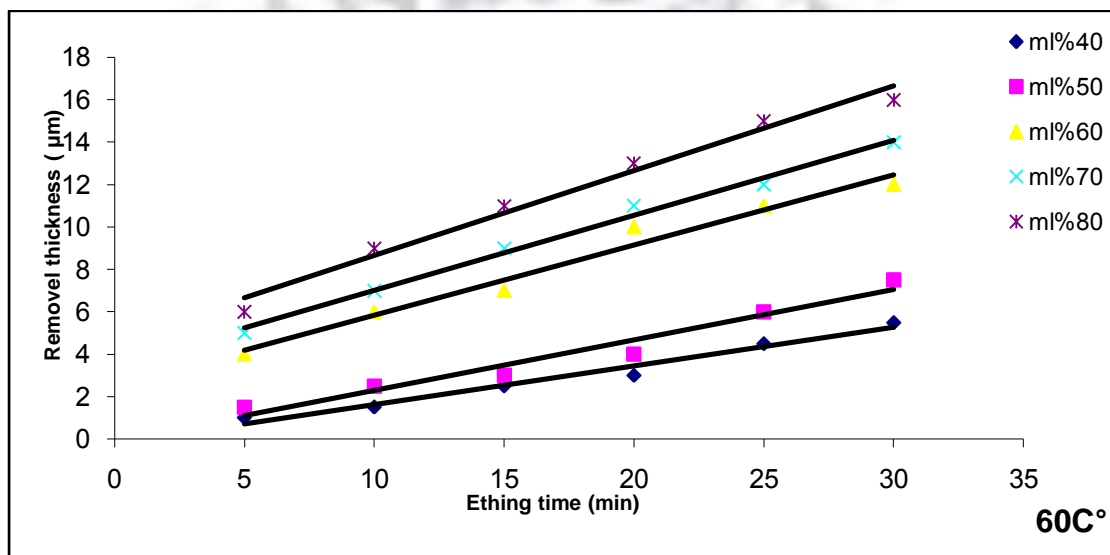


Fig (5) Removal thickness as a function of etching time for CR-39 detectors were etched in Methanol+ Water+ NaOH with different normality at 60°C.

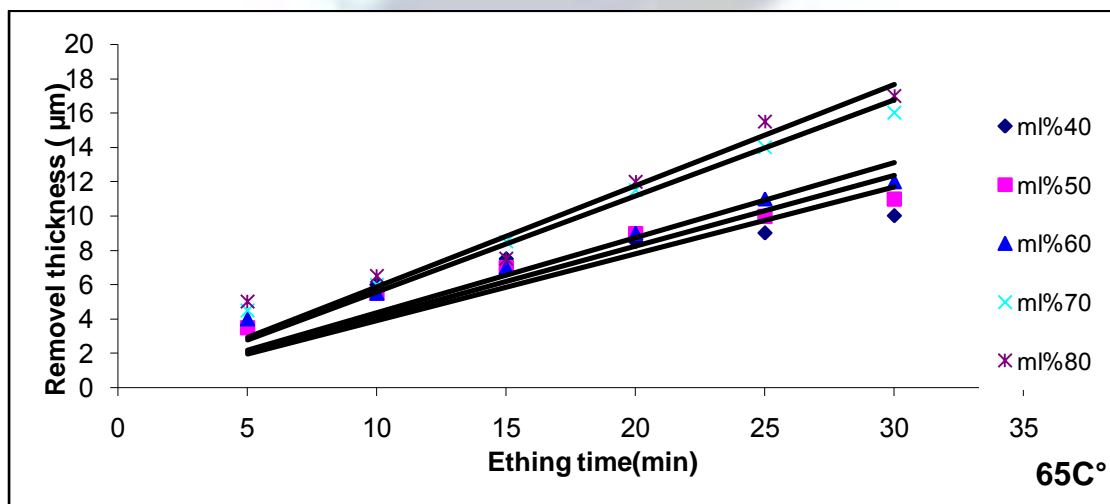


Fig (6) Removal thickness as a function of etching time for CR-39 detectors were etched in Methanol+ Water+NaOH with different normality at 65°C.

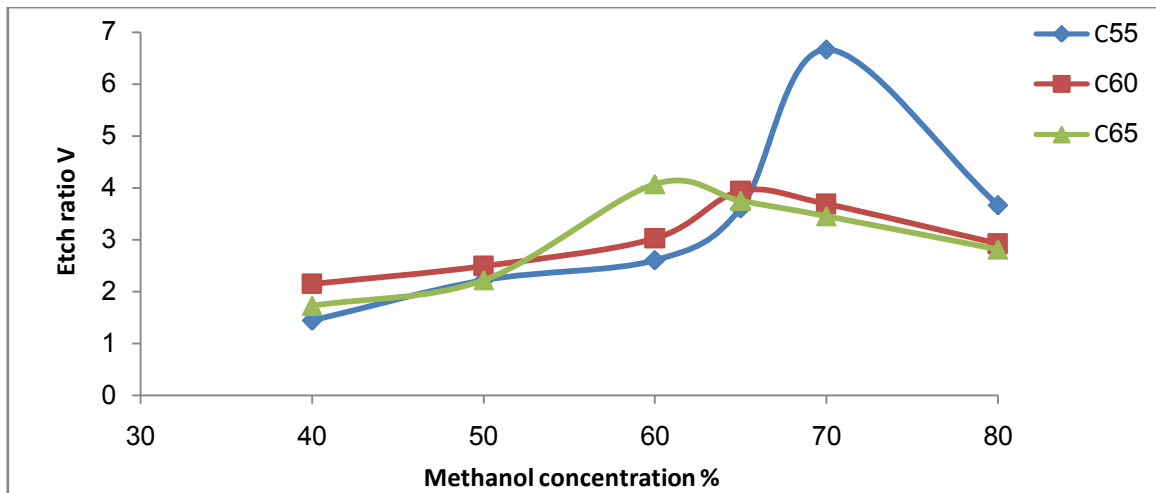


Fig (7) Etch rate ratio V as a function of methanol concentration in the etching solution of CR-39 detectors

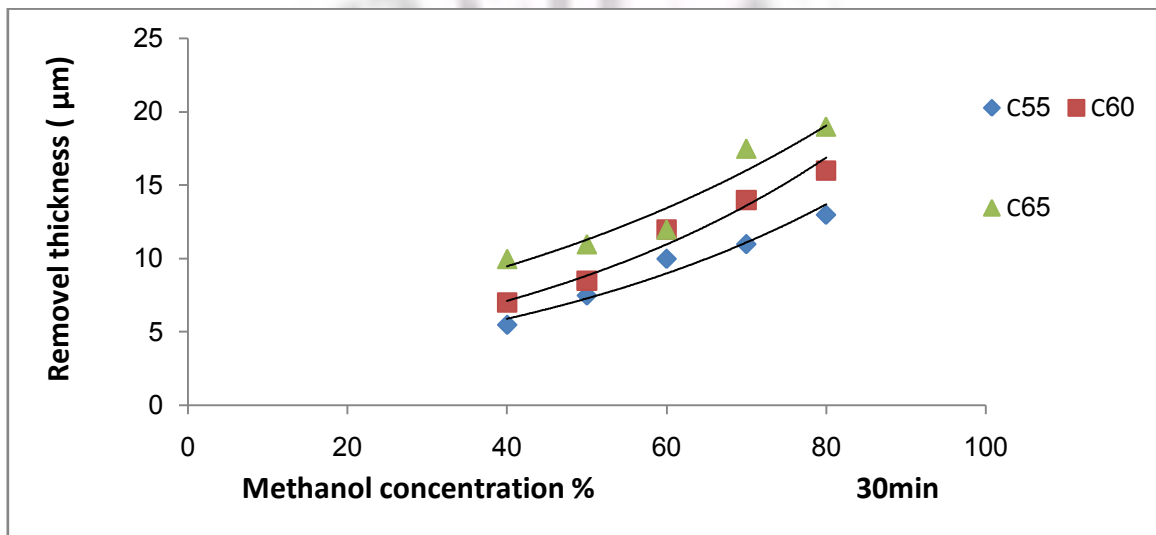


Fig (8) Removal thickness as a function of methanol concentration in the etching solution of CR-39 detectors at etching time 30min.

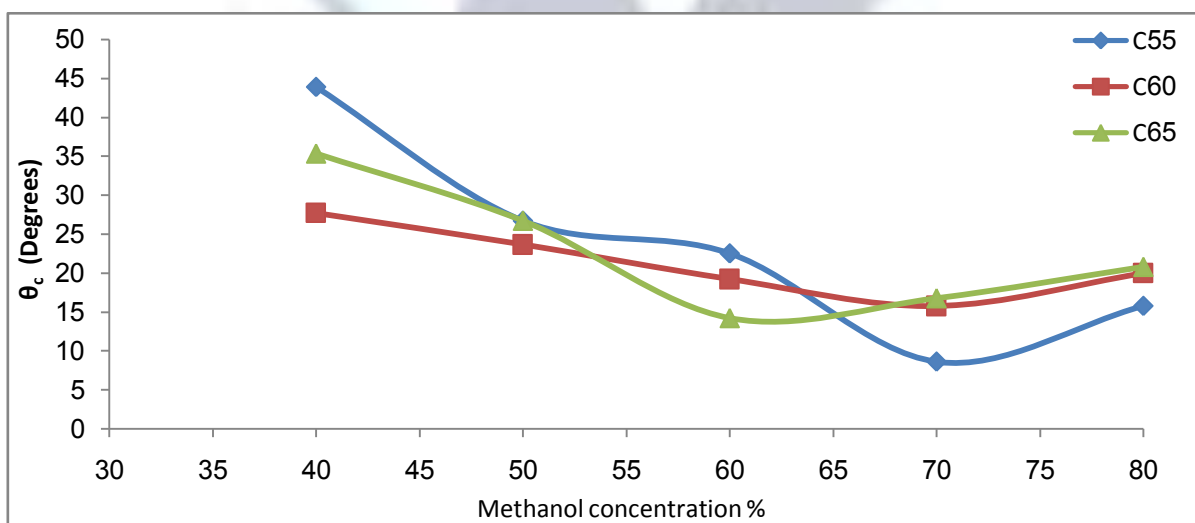


Fig (9) Critical angle θ_c as a function of methanol concentration in the etching solution of CR-39 detectors

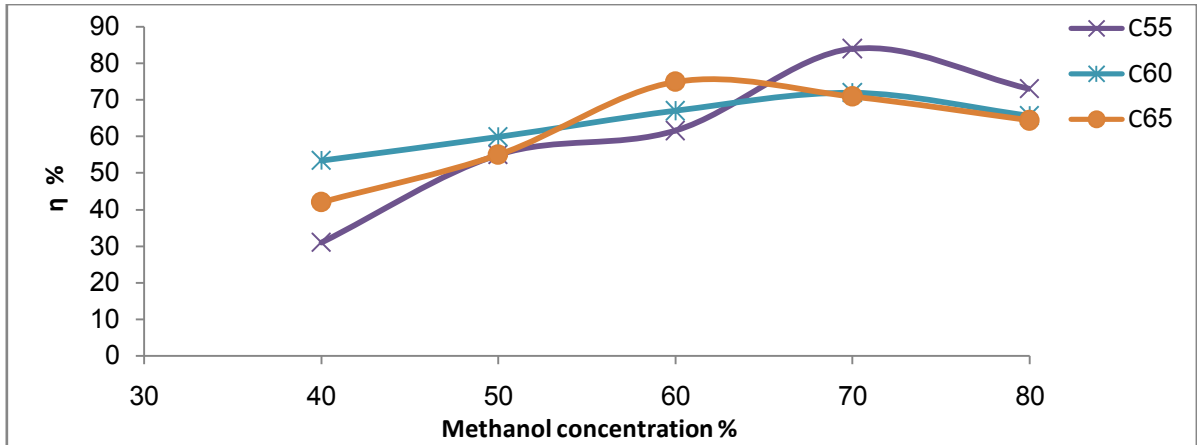


Fig (10) Efficiency η as a function of methanol concentration in the etching solution of CR-39 detectors

Table 1- Values of V_B, V_T, V, θ_c and η for alpha particles of CR-39 detector for various concentrations of Methanol in etching solution at 55°C.

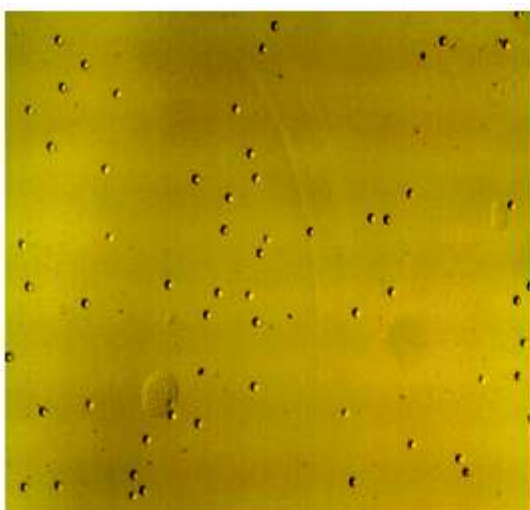
Methanol%	VD	$V_B (\mu\text{m}\cdot\text{h}^{-1})$	$V_T (\mu\text{m}\cdot\text{h}^{-1})$	V	θ_c	η %
40	0.16	11.28	16.2	1.44	43.89	31
50	0.32	15.6	34.6	2.21	26.74	55
60	0.37	16.6	34	2.61	22.5	61
70	0.49	17	113	6.66	8.6	84
80	0.54	21.4	78	3.67	15.8	73

Table 2- Values of V_B, V_T, V, θ_c and η for alpha particles of CR-39 detector for various concentrations of Methanol in etching solution at 60°C.

Methanol%	VD	$V_B (\mu\text{m}\cdot\text{h}^{-1})$	$V_T (\mu\text{m}\cdot\text{h}^{-1})$	V	θ_c	η %
40	0.22	10.9	23.4	2.15	27.7	53..5
50	0.34	14.22	44.4	3.12	18.6	67
60	0.44	18.6	56.4	3.02	19.2	67
70	0.53	19.2	77.4	3.68	15.74	72
80	0.56	24	70.2	2.92	20.015	65

Table 3- Values of V_B, V_T, V, θ_c and η for alpha particles of CR-39 detector for various concentrations of Methanol in etching solution at 65°C.

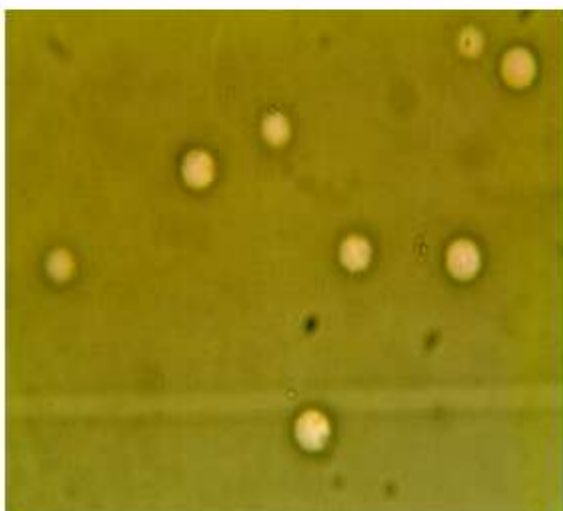
Methanol%	VD	$V_B (\mu\text{m}\cdot\text{h}^{-1})$	$V_T (\mu\text{m}\cdot\text{h}^{-1})$	V	θ_c	η %
40	0.25	14.5	25.2	1.72	35.36	42
50	0.37	18	40.1	2.22	26.7	55
60	0.52	20	81.6	4.07	14.2	75
70	0.78	31.5	109.2	3.46	16.78	71
80	0.8	34.8	97.8	2.81	20.8	64



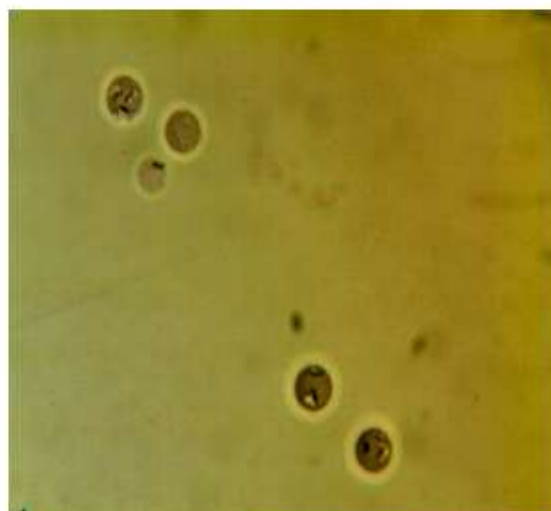
Methanol 70%, 15min, 55°C



Methanol 70%, 20min, 55°C



Methanol 70%, 25min, 55°C



Methanol 70%, 30min, 55°C