

## **Physiochemical properties of crystalline etch products for CR-39 track detector after $\alpha$ -particles irradiation**

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### **ABSTRACT**

*The effect of  $\alpha$ -particles from radiation source Am-241 with activity  $1\mu\text{Ci}$  on the nuclear track detector - NTD type CR-39, dimensions  $(28 \times 28 \times 1.2) \text{ mm}^3$  were measured. The measurements included the changes in physical and chemical properties for the solution of etch crystalline products for CR-39 after irradiation by  $\alpha$ -particles irradiation at two periodic times 0.3 h and 23 h for low and high doses respectively. The changes in organic compounds for etch products were measured by using of FTIR - spectroscopy and transmission electron microscope - TEM. FTIR - spectroscopy shown there was decrease in the values of transmission percent ratios - Tr of wave numbers 1787, 2800, 839 and  $1650 \text{ cm}^{-1}$  relative to wave number  $792 \text{ cm}^{-1}$  respectively with increase of irradiation time of  $\alpha$ -particles. The etch products after  $\alpha$ -particles irradiation of CR-39 detectors were change to low molecular weight organ compounds as a results of degradation bonds and may be formed compounds 2,2-oxy diethanol and 2-propen as the reaction products. Also, shown from transmission electron microscope - TEM there was increase in the degradation amount of etching organic compounds with increase of irradiation time of  $\alpha$ -particles. While the change in inorganic compounds for etch crystalline products after  $\alpha$ -particles irradiation of CR-39 detectors were describe by using of polarized optical microscope - POM, which shown from its images there was broken in crystalline layers to small crystal with increase of irradiation time. The physiochemical properties which measured by FTIR spectroscopy and transmission electron microscope - TEM for organic etching products were better than the analysis of inorganic crystalline etching products by Polarized microscope - PM.*

**Keywords:** CR-39, nuclear track detector-NTD, crystalline etch products,  $\alpha$ -particles.

### **INTRODUCTION**

CR-39 represent of one type of polymers which employed as a detectors in field of science and technology [1, 2, 3]. one of these field was measurement the radiation effects for non-particle radiation as ionizing radiation[4] and particle radiation as ion beam, neutron and  $\alpha$ -particle[5], and so named since named nuclear track detector-NTD[6,7]. The main strength of these detectors is that the damage produced by the ionizing particle can be enlarged through chemical etching [8]. These enlarged tracks, and physical properties can be viewed under the optical microscope and fourier transform infrared spectroscopy - FTIR [9,10].

Nuclear track detector - NTD type CR-39 is one of the trade names of the family of Poly Allyl Diglycol Carbonate - PADC etch track detectors. When it is etched in sodium hydroxide - NaOH solution, a variety of inorganic and organic compounds are formed as the reaction products, which also effect on the thickness of CR-39 [11, 12]. Many products were formed after CR-39 etched in NaOH solution as poly allyl alcohol, 2,2-oxy diethanol [13], allyl alcohol, isopropyl alcohol, sodium carbonate, sodium bicarbonate, and  $\text{CO}_3^{2-}$  ions [14, 15] as well as crystalline products as nahcolite ( $\text{NaHCO}_3$ ), natrite ( $\text{Na}_2\text{CO}_3 \cdot 7\text{H}_2\text{O}$ ), thermonatrite ( $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$ ), natron ( $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ ) and trona ( $\text{Na}_2\text{CO}_3 \cdot \text{NaHCO}_3 \cdot 2\text{H}_2\text{O}$ )[14]. When the physical and chemical products of crystalline etch products is changes with type of radiation damage. So, in the present work, we examined the

properties of crystalline etch products of CR-39 after irradiation with  $\alpha$ -particles. The effect of  $\alpha$ -radiation on CR-39 detector through their crystalline etch products was measured by analysis of organic compounds of these products from using of FTIR – spectroscopy [9] and transmission electron microscope –TEM [16], when the analysis of inorganic compounds [17] of these products from using of polarized microscope.

### MATERIALS AND METHODS

Three pieces sample of nuclear track detector-NTD type CR-39 was munched from Xuchang Tianhe Welding Products Co., Ltd. Having dimensions (28 x 28 x 1.2) mm<sup>3</sup>. Two pieces samples of CR-39 were irradiated by  $\alpha$ -particle from Am-241 source with activity 1 $\mu$ Ci. One of these samples irradiated at 0.3 h (for low radiation dose) and another sample irradiated at 23h (for high radiation dose). Third sample of CR-39 was un-irradiated as a control.

Three sample above were dissolved in 200 ml of 6:0 M sodium hydroxide - NaOH solution contained in a 300 ml Pyrex beaker. The beaker was placed in a water bath maintained at 70.2 - 70.3°C and shaken for 70 h to prepare a super-saturated solution of the etch products. The separation of organic compounds solutions for CR-39 during etching process for three sample above were sampled as EC, ER<sub>1</sub> and ER<sub>2</sub> for un-irradiated, 0.3 h  $\alpha$ -irradiated and 23 h  $\alpha$ -irradiated respectively.

EC, ER<sub>1</sub> and ER<sub>2</sub> were separated in pure forms by solvent extraction with 50 ml diethyl ether with the aid of 250 ml separating funnel for three steps, then the organic extracting evaporated at room temperature up to 2 ml concentrate sample, and the FTIR spectra in the range (4000 – 400) Cm<sup>-1</sup> were recorded using NaCl sandwich cell on FTIR instrument, model - 8000 Shimadzu spectrophotometer. Separation of organic compounds was also analysis by transmission electron microscope - TEM, Model - Philips CM10 with an optimal operating voltage of 200 keV, to show the images of organic compounds after irradiation comparing with un-irradiated samples. The separation of inorganic compounds for EC, ER<sub>1</sub> and ER<sub>2</sub> by splitting the aqueous layer samples and left this layer to evaporate at room temperature to grow the formed inorganic products for 2 months, different needle crystalline for each sample were separated manually upon a glass microscope for image analysis by polarized optical microscope – POM, model Meiji MT9000. The texture of the compounds were observed using polarized light with crossed polarizer, the sample being prepared as a thin film sandwiched between a glass slide and a cover slip. A camera –Lumenera was installed on the polarizing microscope.

### RESULTS AND DISCUSSION

Figure 1 shown the FTIR spectrum at the wave number rang 4000-400 Cm<sup>-1</sup> for chemical etching products of EC, ER<sub>1</sub> and ER<sub>2</sub> samples for un-irradiated, 0.3 h irradiated and 23 h irradiated of CR-39 respectively. The FTIR spectrum for EC sample, show the presence of band at 1787.9 Cm<sup>-1</sup> due to the stretching of carbonyl group of ester and band at 839 Cm<sup>-1</sup> for out of plane bending, these two band also appeared for ER<sub>1</sub> while this two bands disappeared for ER<sub>2</sub>, which can attributed to the decomposition of compound ER<sub>2</sub> by the action of radiation and converted to another compounds (alcohol and ether). Other bands like aliphatic C-H stretching at 2800 Cm<sup>-1</sup> and C = C stretching at 1650 Cm<sup>-1</sup> having small effect relative to wave numbers 1787.9 Cm<sup>-1</sup> and 839 Cm<sup>-1</sup>.

As we see from figures all the functional group decreases this due to as we said the decomposition of compound to lower molecular mass alkane during etching of CR-39, as shown in figure 2.

The FTIR spectra show the appearance of OH stretching group for samples EC, ER<sub>1</sub> and ER<sub>2</sub>, because the reactant and the products contain hydroxyl group. Also from the spectra we have bands for carbonyl stretching at 1787.9 cm<sup>-1</sup> for sample EC and ER<sub>1</sub> and disappeared for sample ER<sub>2</sub> this can be explain as follows: sample EC contain two carbonyl groups, after radiation produce sample ER<sub>1</sub> which contain one carbonyl group, while sample ER<sub>2</sub> don't have carbonyl group as show in figure 2.

Table 1 show the wave numbers and assignment for crystalline etch products for EC, ER<sub>1</sub> and ER<sub>2</sub> of CR-39 detector measured by FTIR spectroscopy, which shown the mains bonds still appear with increase of irradiation time at wave numbers 2800, 1787, 1650, 839 Cm<sup>-1</sup> as wall as to another wave numbers 3300, 1402, 1109 Cm<sup>-1</sup>.

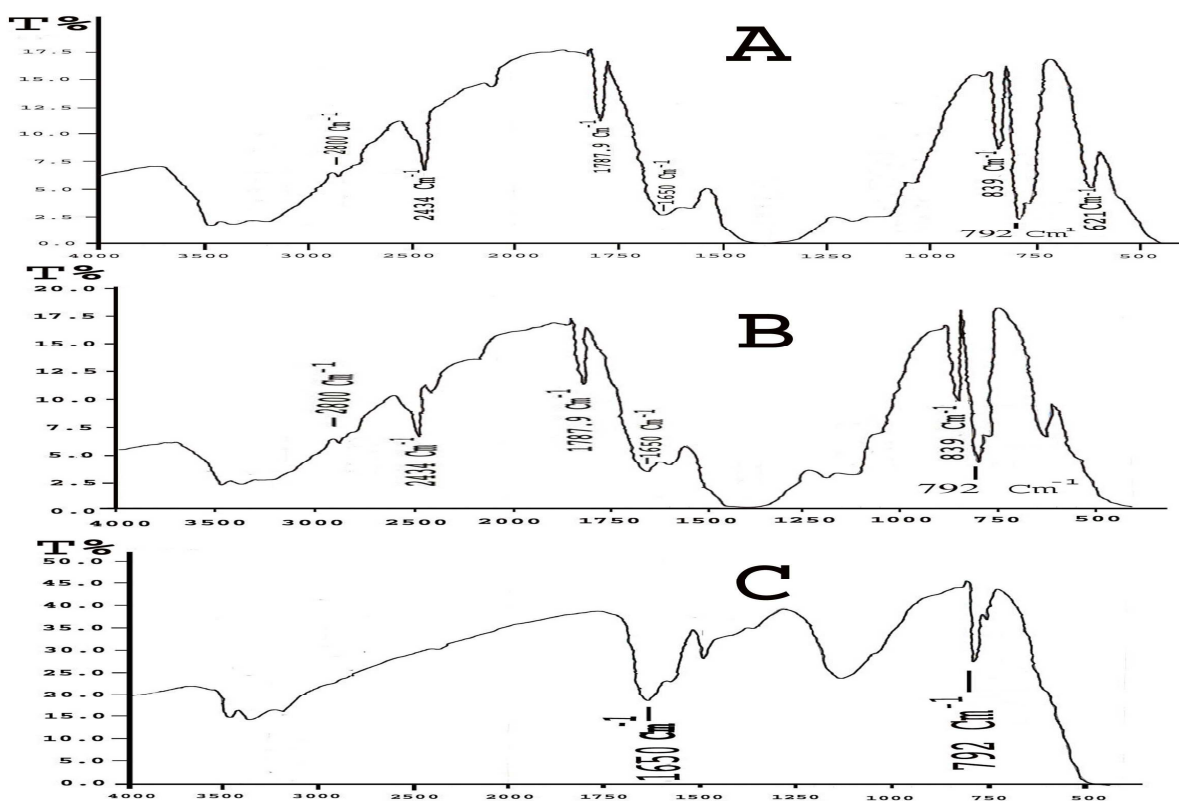


Fig. 1 : Transmission percent  $-T\%$  from FTIR – spectrum at wave number range  $4000-400\text{ cm}^{-1}$  for organic etching products of EC,  $\text{ER}_1$  and  $\text{ER}_2$  samples after  $\alpha$ -particle irradiated of CR-39 detector respectively. A: un-irradiated, B: 0.3 h irradiated, C: 23 h irradiated

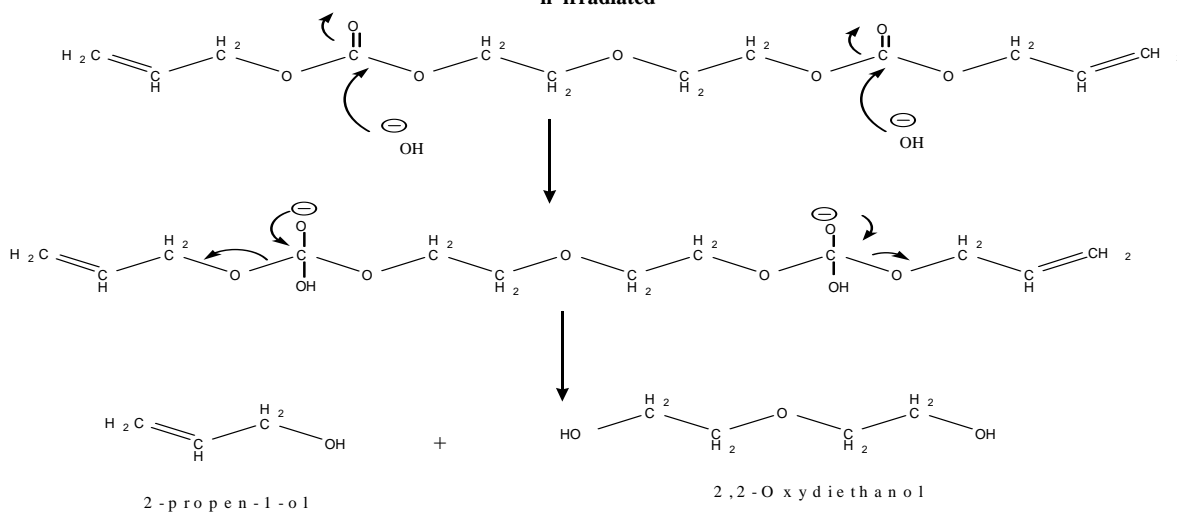


Fig. 2 : Scheme for the possible positions which are subjected to cleavage by the hydroxide ion [ 13] .

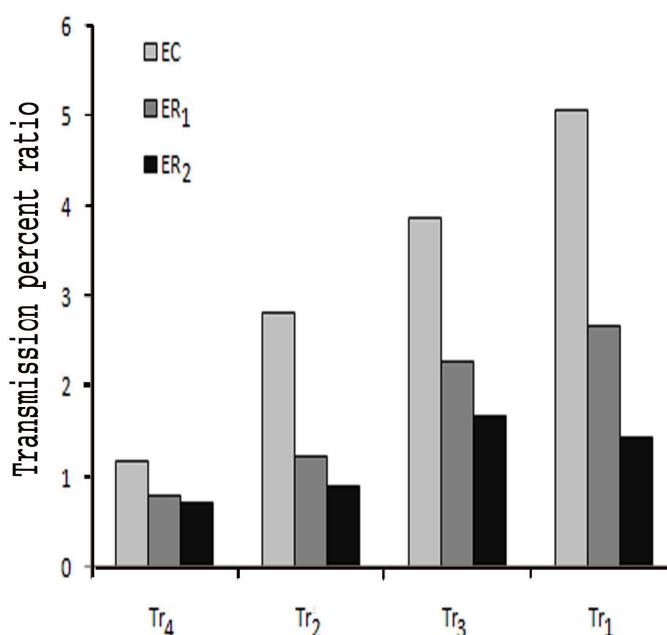
**Table 1 : Wave numbers –  $\text{Cm}^{-1}$  and assignment crystalline etch products of for chemical etching products of CR-39 detector measured by FTIR spectroscopy as per Figure 1**

ESC ( $\text{Cm}^{-1}$ )	ESR <sub>1</sub> ( $\text{Cm}^{-1}$ )	ESR <sub>2</sub> ( $\text{Cm}^{-1}$ )	Assignment
3300	~ 3300	3348	OH stretching
3193	~3192	3190	C=C-H stretching
2800	2800	~ 2860	Symmetric CH <sub>2</sub> stretching
2761	2761	-----	Symmetric CH <sub>2</sub> stretching
1787.9	1787.9	-----	C=O stretching
1650	1650	~ 1635	C=C stretching of vinyl group
-----	-----	1577	OH bending
1402	1400	~ 1492	C-H bending
		1367	C-H bending
1109	1109	-----	C-O-C stretching
1043	1043	~1134	C-O-C stretching
839	839	-----	C-H rocking out of plane
769	769	794.6	C-H rocking out of plane

Figure 3 show the transmission ratio  $Tr$  of the wave numbers 1787 , 2800 , 839 and 1650  $\text{Cm}^{-1}$  relative to wave number 792  $\text{Cm}^{-1}$  for each samples of EC , ER<sub>1</sub> and ER<sub>2</sub> samples of CR-39 detector . Transmission ratios  $Tr$  of the wave numbers 1787 , 2800 , 839 and 1650  $\text{Cm}^{-1}$  named to  $Tr_1$  ,  $Tr_2$  ,  $Tr_3$  and  $Tr_4$  calculated by following equations respectively .

$$*Tr_1 = \frac{[T\%]_{1787}}{[T\%]_{792}}, Tr_2 = \frac{[T\%]_{2800}}{[T\%]_{792}}, Tr_3 = \frac{[T\%]_{839}}{[T\%]_{792}}, Tr_4 = \frac{[T\%]_{1650}}{[T\%]_{792}} \quad (1)$$

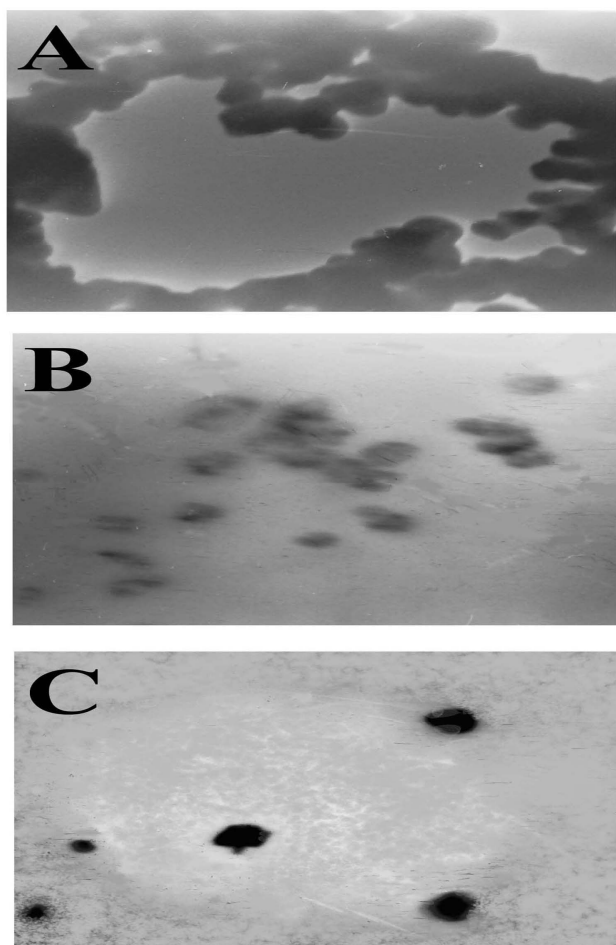
The values of  $Tr_1$  ,  $Tr_2$  ,  $Tr_3$  and  $Tr_4$  were dropping with increase of time of irradiation of  $\alpha$ - particles as shown in fig. 3 .



**Fig. 3 : Transmission percent ratios  $Tr_1$ ,  $Tr_2$ ,  $Tr_3$  and  $Tr_4$  (1) for wave numbers 1787 , 2800 , 839 and 1650  $\text{Cm}^{-1}$  relative to wave number 792  $\text{Cm}^{-1}$  respectively .EC , ER<sub>1</sub> and ER<sub>2</sub> samples to un-irradiated , 0.3 h irradiated and 23 h irradiated for  $\alpha$ -particle irradiation of CR-39 detector respectively**

$Tr_1$  and  $Tr_3$  for C=O group stretching and C-H group rocking respectively were a good agreement for radiation response better than  $Tr_2$  and  $Tr_4$  for CH<sub>2</sub> group starching and C=C respectively , figure 3 .

For high doses of  $\alpha$ - radiation at time of irradiation more than 23 h represent degradation for all chemical etching products to low organic compound as appearing the stability of  $Tr_1$  ,  $Tr_2$  ,  $Tr_3$  and  $Tr_4$  after the irradiation time 23 h . And the degradation of organic compounds with increase of irradiation time was clear appearing in the images of transmission electron microscope - TEM as shown in figure 4 .



**Fig. 4 :** Transmission electron microscope –TEM\* images for organic etching products of EC ,ER<sub>1</sub> and ER<sub>2</sub> samples after  $\alpha$  – particle irradiated of CR-39 detector respectively .A: un-irradiated , B: 0.3 h irradiated , C: 23 h irradiated .

\* Transmission electron microscope –TEM model Philips CM10

The average diameter ranges of organic compounds which measured by TEM for sample B and C were 70-90 nm . The  $\alpha$ -radiation effect below irradiation time 0.3 h , figure 4 may be assessment the radiation effect for low doses by measured of relative transmission percent  $Tr_1$  and  $Tr_3$  for 1789 and 1650  $Cm^{-1}$  respectively equivalent to image analysis process by programming software for diameter ranges . The POM - analysis of inorganic compounds of chemical etching products were appear these compounds as layers at un-irradiated samples as shown in the image A for EC sample , figure 5 .

When  $\alpha$ -irradiation at time 0.3 h for ER<sub>1</sub> sample the crystalline layers were broke to small crystal as shown in the image B for ER<sub>1</sub> sample . while  $\alpha$ - irradiation at time 23 h these small crystal were degradation to vary small crystal as shown in image C for ER<sub>2</sub> . The degradation of small crystals in image C , Figure 5 also may be measured by image analysis process using programming software to produce good agreements which reflected the effect of  $\alpha$ - radiation on CR-39 detector. From this study shown the properties of chemicals etching products after  $\alpha$ -irradiation CR-39 detector with  $\alpha$ - particles for 0.3 h and 23 h was change depended on the time of radiation , and these properties measured by FTIR spectroscopy and transmission electron microscope –TEM for organic etching products were better than the analysis of inorganic crystalline etching products by Polarized microscope - PM .

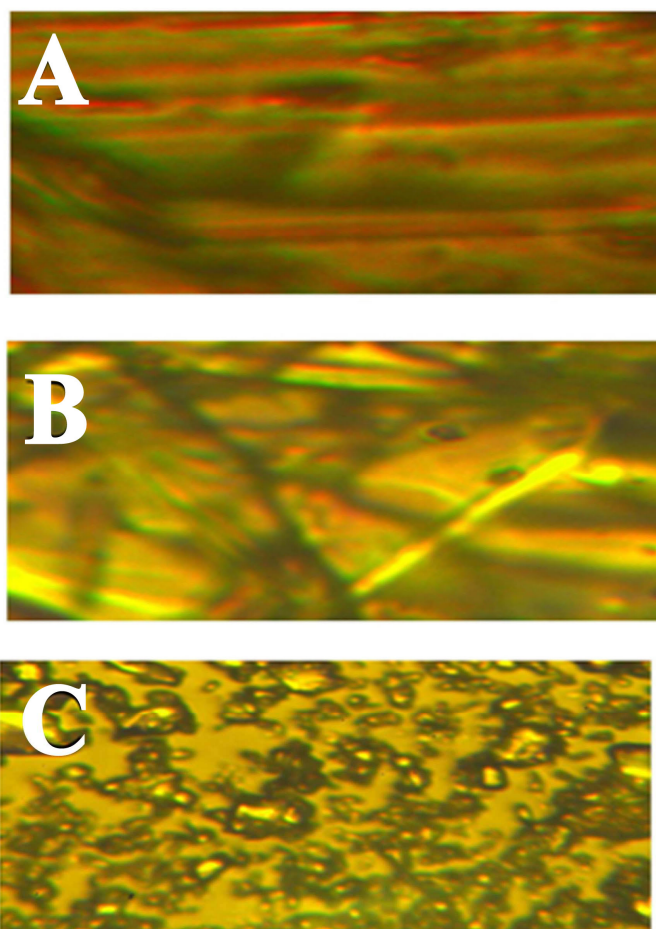


Fig. 5 : Polarized optical microscope – POM\* images for inorganic crystalline etching products of EC, ER<sub>1</sub> and ER<sub>2</sub> samples after  $\alpha$ -particle irradiated of CR-39 detector respectively. A: un-irradiated, B: 0.3 h irradiated, C: 23 h irradiated.

\*Polarized optical microscope–POM, model - Meiji MT9000.

### CONCLUSION

The physiochemical properties which measured by FTIR spectroscopy and transmission electron microscope–TEM for organic etching products for CR-39 track detector after  $\alpha$ - particles irradiation were better than the analysis of inorganic crystalline etching products by Polarized microscope-PM.

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