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# Low Energy X-ray Photons Induced Changes in Lexan Films

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40 keV Synchrotron X-ray photons were made to fall on Lexan polycarbonate films at different exposure time. The low energy X-ray photons assisted modification in terms of physico-chemical properties of Lexan films were studied using Fourier Transform Infrared (FTIR) spectrophotometer, X-Ray Diffractogram (XRD), Vicker's Microhardness Tester, Scanning Electron Microscope (SEM) and Contact Angle Meter. FTIR result mainly includes decrease in the C=C stretching and C-C stretching of aromatics after irradiation. XRD analysis shows a slight decrease in the crystallinity after irradiation. Vicker's microhardness test reveals the decrement in microhardness of Lexan films after irradiation. SEM result shows irradiation induced changes in the surface morphology. Contact angle measurement shows increase in the water contact angle in irradiated Lexan films.

**Keywords:** Lexan films, X-ray photons, XRD, FTIR, Microhardness, SEM, Contact angle.

## 1 Introduction

Modification of polymer properties has become an emerging field of interest due to increased usage of polymers in different fields such as microelectronics[1], anti-bio films applications[2], space applications[3] and other corresponding surface coating applications. The modification of different properties can be done by various methods such as chemical method, flame treatment, doping method or by irradiation *etc*. Irradiation method will bring changes in the material by energy deposition, molecular bond breaking, creating free radicals *etc*. The changes in polymer properties can be finely tuned with the selection of appropriate energy, dose and species of radiation. Among different types of radiation, Synchrotron X-rays have found many interesting applications due to their ability to change the characteristics of the materials[4].

Polymer modification by irradiation method would cause scissioning of polymer chains, cross-linking among them resulting the formation of new polymer network, *etc*. This would result in the modification of physico-chemical and other corresponding properties of the polymer. The present study is based on the Synchrotron X-ray irradiation effects on Lexan polycarbonate films as it is a promising material with important applications in different fields[5,6]. The irradiated polymer would have changes in its molecular bonds and functional groups. However, the change is directly depending on the energy and time of exposure. The mechanism of modification of the polymer results in the chemical, structural, mechanical and other corresponding properties. In this study, the effect of 40 keV Synchrotron X-ray photons on the thin, transparent Lexan polymer films has been studied by comparing the physico-chemical properties before and after irradiation.

## 2 Experimental details

The material used for the study was thin and transparent Lexan polycarbonate film with 200 μm thickness. It was cut into 2cm x 2cm size for the purpose of irradiation. The irradiation was carried out using 40 keV X-ray photons from Synchrotron Accelerator Facility available at Raja Ramanna Centre for Advanced Technology (RRCAT) Indore. The details of the facility were reported elsewhere[7]. Irradiation induced changes in the molecular bonds and functional groups of polymer were studied using a JASCO V-670 FTIR Spectrophotometer. Structural changes were studied using a Bruker AXS D8 Advance X-Ray Diffractometer. The microhardness of Lexan films were tested using Vickers' Microhardness tester. The surface morphology was

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studied using a JEOL JSM 6390LV SEM instrument. The wettability test was performed using a contact angle meter.

## 3 Results and Discussion

### 3.1 FTIR Studies

Figure 1 shows FTIR spectra of pristine and irradiated Lexan films. The approximate characteristics of the vibrations and corresponding wave numbers and functional groups of characteristic bands are listed in Table 1[8]. There are no major changes in the spectra of both pristine and irradiated PC films. But for the subtle observation, minor decrement in the position of absorption bands especially in C=O stretching, C-C stretching, etc were observed.

This kind of minute change ascertains the chain scission in the polymer matrix with the release of volatile gases such as carbon monoxide/dioxide and with the formation of hydroxyl groups. The result of this chemical modification may alter the structural and mechanical behavior of Lexan films.

Table 1 — Approximate characteristics of the vibrations and corresponding wave numbers

| Position of bands (in $cm^{-1}$) | Assignment of bands |
|---|---|
| 1767 | C=O stretching of carbonyls |
| 1500 | C-C stretching of aromatics |
| 1220 | C-H wagging of alkyl halides |
| 1185 | C-H wagging of alkyl halides |
| 1156 | C-H wagging of alkyl halides |
| 1010 | C-O stretching of carboxylic acids |

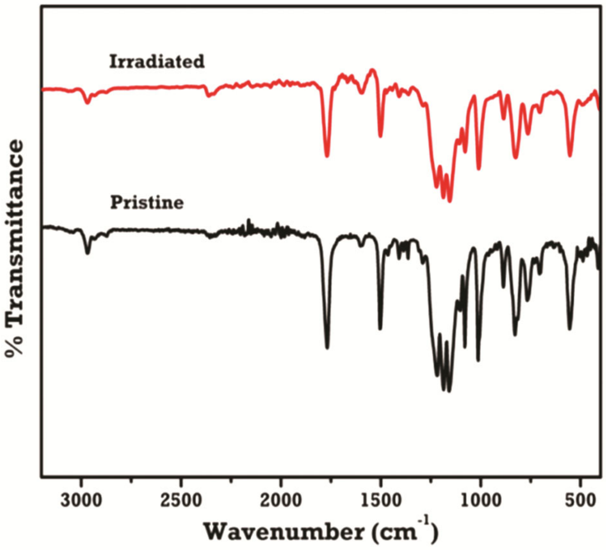

Fig. 1 — FTIR spectra of Lexan films.

### 3.2 XRD Analysis

The structural parameters of Lexan films for both pre-irradiation and post-irradiation were determined using XRD analysis. The X-ray diffractogram is as shown in Fig. 2. The main peak of the pristine PC is at the 2θ value $16.60^0$. Irradiation has introduced small changes in the positions of the peaks but a significant reduction in the peak intensity with narrowing of full width at half maximum has been observed until 45 minutes of exposure. This could be attributed to the enhancement of amorphous nature and the degradation of polymer chains. The peak intensity starts to increase in the case of samples exposed for 60 minutes. This may be due to the rearrangement of polymer chains and occurrence of cross linking process.

The structural parameters in response to the irradiation would have also changed by the rearrangement of cross linking density. The inter-planar distance (d) was calculated using the Braag's equation.

$$n\lambda=2d\sin\theta \quad \dots (1)$$

Where, n is the order of reflection, λ is the wavelength of the X-ray used (1.5406Å), d is the inter-planar distance and θ is the diffraction angle. The crystallite size (L) and percentage of crystallinity (X%) were calculated using the following equation[9].

$$L=\frac{K\lambda}{B\cos\theta} \quad \dots (2)$$

Where K is the Scherer constant (0.9) and B is the full width at half maximum.

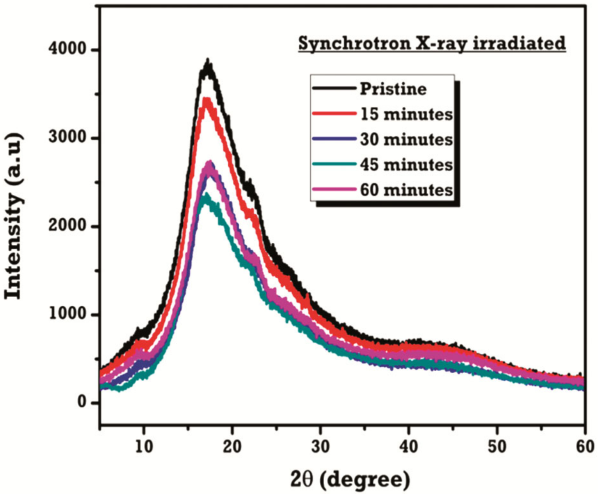

Fig. 2 — X-ray Diffractogram of Lexan films.

$$X=\frac{Ac}{Ac+A_a}\times 100 \quad \ldots (3)$$

Where $A_c$ and $A_a$ are the area under crystalline and amorphous halos respectively.

Table 2 shows the calculated values of inter planar distance (d), crystallite size (L) and percentage of crystallinity (X %) All these observed changes and variations in their values attribute to the occurrence of chain scission and rearrangement of polymer chains along with cross linking[9,10].

### 3.3 Microhardness Test

The microhardness of Lexan films were measured using a Vickers microhardness tester with diamond pyramidal indenter. The Vickers' diamond pyramidal hardness is the quotient of the indenter load and the pyramidal surface area of the impression which is given by the relation below[2].

$$H_V=\frac{1854.4\times P}{d^2}\left(\frac{kgf}{mm^2}\right) \quad \ldots (4)$$

Where $H_v$ is the Vickers' hardness in MPa, P is the indenter load in mN, d is the average of the two indentation marks having diagonal length in µm.

It is evident from Fig. 3. that, the values of microhardness decreases after irradiation. This attributes the X-ray photon induced chain scission in the polymer matrix and hence the maximum possibility of slipping of polymer chains is high. This will increase the deformation and result in decrease of the hardness value[11,12].

### 3.4 Scanning Electron Microscopy

Irradiation induced changes with regard to the surface morphology of Lexan films were studied using a scanning electron microscope. Fig. 4 shows SEM images of pristine and irradiated Lexan films. The morphology on the surface of pristine Lexan shows a relative smoother surface without any unusual or irregular patterns. But, after irradiation some kinds of broken points with voids & gaps and kink like structures and many more disturbed points were observed on the surface morphology of Lexan films. These irregularities impute the occurrence of chain scission due to impinged X-ray photons. During the time of chain scission there might have been emission of volatile gases as a byproduct of scissioning process[13,14]. As a whole, the surface morphology of the irradiated film changes.

Table 2 — Structural parameters of pristine and irradiated PC films

| Exposure time (min) | 2θ (degree) | d (Å) | L (Å) | X (%) |
|---|---|---|---|---|
| 0 | 16.60 | 5.34 | 11.50 | 42.66 |
| 15 | 16.82 | 5.27 | 8.45 | 37.12 |
| 30 | 16.97 | 5.22 | 8.36 | 35.00 |
| 45 | 17.05 | 5.20 | 7.79 | 31.27 |
| 60 | 16.88 | 5.25 | 7.87 | 36.16 |

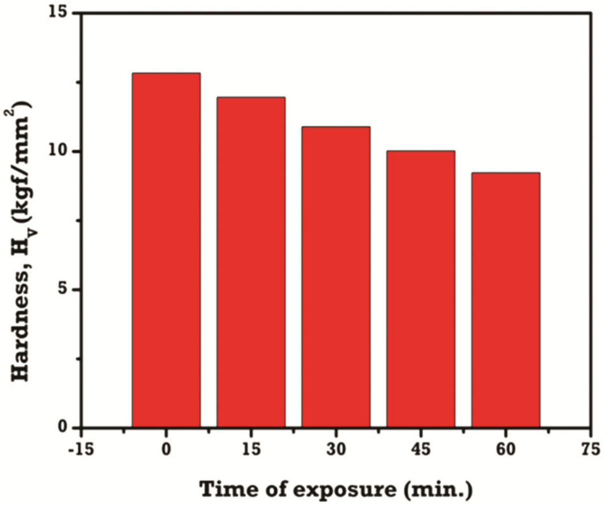

Fig. 3 — Variation of microhardness with exposure time.

### 3.5 Contact Angle Measurement

The changes in the surface morphology bring out changes in terms of water contact angle on the surface of polymer films. The pristine Lexan film is hydrophilic in nature which has got the contact angle value about $70.16^0$. But, irradiated PC films show significant changes in their contact angles. The measurement of strength of the contact between liquid solid interfaces can be explained in terms of work of adhesion.

It can be calculated using the relations[2].

$$W_A=\gamma_{lg}\left(1+\cos(\theta)\right) \quad \ldots (5)$$

Where, $\gamma_{lg}$ is surface tension of liquid gas interface = 72.8 mJ/m2.

The area fraction of the liquid solid interface (f) was also calculated using Classies law of contact angle[2].

$$\text{Cos}\theta_c=f\left(\cos\theta_1+1\right)-1 \quad \ldots (6)$$

Where, $\theta_c$ and $\theta_1$ are the contact angles of irradiated and pristine films.

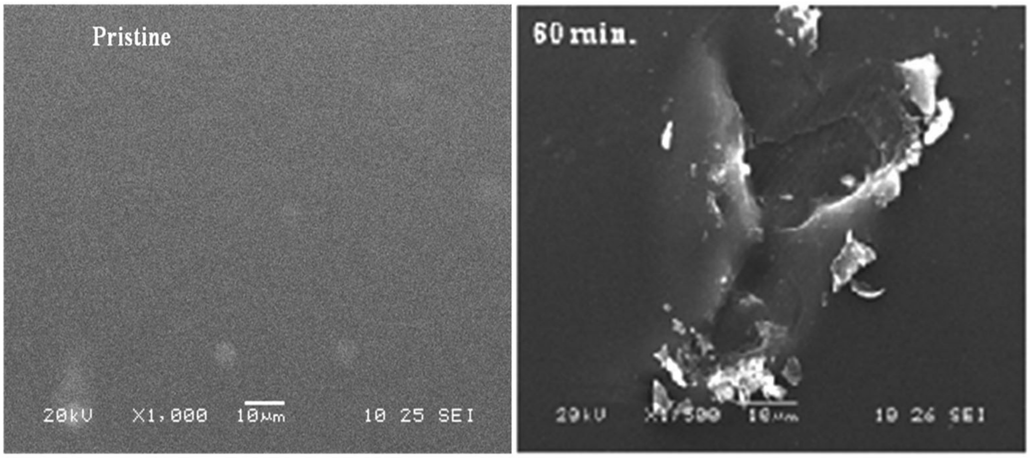

Fig. 4 — SEM images of pristine and irradiated (60 min) Lexan films.

Table 3 — Contact angle parameters of Lexan films at different exposure time.

| Exposure time (min) | Contact angle (degree) | $W_A$ (mL/$m^2$) | f |
|---|---|---|---|
| 0 | 70.16 | 97.51 | 1 |
| 15 | 71.15 | 96.32 | 0.99 |
| 30 | 74.98 | 91.67 | 0.94 |
| 45 | 76.82 | 89.40 | 0.92 |
| 60 | 79.74 | 85.77 | 0.88 |

Table 3 gives all the values which are related to contact angle measurement. It can be observed that by increasing the time of exposure, the water contact angle of Lexan films gradually increases and heads towards hydrophobic nature. However, the results obtained are sufficient to understand the change in surface morphology of Lexan films. In addition to these, the change in the strength of the contact between liquid and solid interfacing surfaces in terms of work of adhesion and area fraction was observed. All these changes attribute that, even 40 keV of X-ray photons have also made significant changes on the surface properties of Lexan films. Altogether, it can be concluded that the low energy X-ray photons are capable to increase the contact angle of Lexan films to make them hydrophobic.

## 4 Conclusions

Lexan films were irradiated by 40 keV X-ray photons and the changes were observed in terms of their chemical, structural, mechanical and surface related properties. FTIR results have shown minor decrement in the carbonyl stretching at 1767 $cm^{-1}$, C-C stretching at 1500 $cm^{-1}$, *etc*. From XRD study, the decrease in the crystallinity after irradiation has been notified. The Vicker's microhardness test has shown decrease in the microhardness after irradiation. SEM results revealed the changes in surface morphology and the wettability test showed that irradiation has induced hydrophobicity in Lexan films. Altogether, the study has revealed the occurrence of chain scission which is responsible for variations physico-chemical behavior of irradiated Lexan films. According to the application point of view, these modifications can be perfectly tuned by selecting appropriate energy, exposure time and other corresponding irradiation parameters.

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