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Effects of Anthracene Doping Ratio and UV Irradiation Time on Photo-Fries Rearrangement of Polycarbonate

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Abstract:

Thin films of pure polycarbonate (PC) with anthracene doping PC films for different doping ratios (10, 20, 30, 40, 50 and 60 ml) were prepared by using a casting method. The influence of anthracene doping ratio on photo-fries rearrangement of polycarbonate was systematic investigated. Furthermore, pure PC and anthracene doping PC films were irradiated via UV light at a wavelength (254 nm) for different periods (5, 240, 288, and 360 hrs). The photo-fries rearrangement occurring in pure PC and anthracene doping PC films were monitored using UV and FTIR spectroscopies. The photo-fries rearrangement leads to scission the carbonate linkage and formation phenylsalicylate and dihydroxybenzophenones. The result of the UV spectrum confirms disappear of polycarbonate peaks, while phenylsalicylate and dihydroxybenzophenone peaks appear at (320 nm) and (360 nm), respectively. The formation of a dihydroxybiphenyl compound reveals when the UV peak distinguishes at (340 nm). FTIR spectroscopy supported forms of phenylsalicylate and dihydroxybenzophenone compounds which appear in carbonyl region at (1689 cm^{-1}) and (1630 cm^{-1}), respectively. It founds that anthracene accelerates the photo-fries rearrangement of polycarbonate in the anthracene doping PC films because anthracene leads to formation of excited singlet state oxygen ($^1\text{O}_2$). Singlet oxygen ($^1\text{O}_2$) leads to the formation of a hydro peroxide, which could decompose and cause to chain scission and formation of a terminal of a carbonyl group. The presence of the carbonyl groups in the polymer makes it photo-labile, also warns that the polymer is vulnerable to deterioration.

Key words: Anthracene, Effect of doping ratio, FTIR Spectrum, Photo-Fries rearrangement, Polycarbonate.

Introduction:

Polycarbonate is a kind of polyester where the carbonate ester groups have been connected with an aromatic group (1-4). The basic chemical structure of polycarbonate is shown in Fig. 1, and the name of polycarbonate came from its carbonate backbone (5, 6). When polycarbonate irradiation with UV lights at short wavelength (254 nm) the photo-fries rearrangement is taken place (7). The UV irradiation leads to scission the carbonate linkage and the formation of two free radicals. These free radicals are rearranged in order to form phenylsalicylate and dihydroxybenzophenone, as well as another group such as dihydroxybiphenyl compounds.

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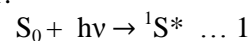
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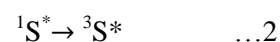
Furthermore, the photo-fries products can also be photo-oxidation to product the secondary and tertiary product of lower molecular weight (6, 8).

Anthracene is a polycyclic aromatic hydrocarbon (PAH) consisting of three benzene rings (9). Excited state polycyclic aromatic hydrocarbon (PAH) can act as a photosensitizer. That is commonly used in polymer chemistry; in reactions such as photo polymerization, photo crosslinking, and photo degradation (10). Photosensitization by a chemical can be result in formation of excited singlet state oxygen ($^1\text{O}_2$) (11).The reaction steps concerned in photosensitization are as follow (12):

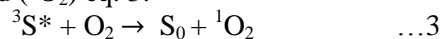
The sensitizer molecule in the ground state (S_0) absorbs a photon to reach the first singlet excited state ($^1S^*$) eq. 1:



Intersystem crossing generate the excited triplet ($^3S^*$) eq. 2:



The excited triplet ($^3S^*$) can be quenched by a stable oxygen molecule to form the ground state sensitizer (S_0) and (1O_2) eq. 3:



Singlet oxygen (1O_2) exhibits several particular reactions. One of the most important reactions of singlet oxygen in photodegradation of polymers is the formation of a hydro-peroxide. This product could decompose and cause to chain scission and formation of a terminal of a carbonyl group (13).

The presence of carbonyl groups in a degraded polymer indicates that oxidation has taken place and warns that the material is vulnerable to further deterioration because they are photo-labile (14).

Rivaton *et al.*, (15) studied a comparison of the photochemical and thermal degradation of thin films of bisphenol-A polycarbonate and trimethylcyclohexane-polycarbonate. The results reveal that thin films with a thickness of (20 μm) were obtained by evaporation of polymer solution in CHCl_3 compound. It founded that when samples irradiation at short wavelength (254 nm), the photo-fries rearrangement was taken place and led to form phenylsalicylate and dihydroxybenzophenone composites. Since, the UV bands of phenylsalicylate and dihydroxybenzophenone seemed at (320 nm) and (355 nm), respectively. Examine of FTIR spectra confirmed that formation of phenyl salicylate and dihydroxybenzophenone in carbonyl region at (1689 cm^{-1}) and (1629 cm^{-1}), respectively.

Moreover, Salh (16), investigated the spectroscopic properties of HALS (HALS: Tinuvin 770 and Tinuvin 123) doped polycarbonate films with a thickness of (2 mm). The UV irradiation of as- grown samples is achieved by using UV source for (48 hours) under dry condition (i.e. room relative humidity). The distance between the lamp and the sample is (28 cm). The UV-Vis spectroscopy showed that the absorbance for wavelengths below (400 nm) is increasing with increasing (Tinuvin 770) concentration. Two peaks were appeared at wavelengths (320 nm) and (355 nm), which are attributed to phenylsalicylate and dihydroxybenzophenone composites, respectively.

The major aim of the present work is to investigate the photo-fries rearrangement of pure PC and anthracene doping PC films for different doping ratios 10, 20, 30, 40, 50 and 60 ml. In addition, study the effect of anthracene addition on the photo-fries rearrangement of polycarbonate compound. The product material could be beneficial for various applications such as safety helmets, lenses light globes and base for photographic film.

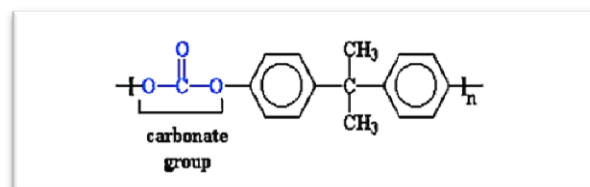


Figure 1. The basic chemical structure of polycarbonate (PC) compound (19)

Materials and Method:

Polycarbonate with common brand name Lexan (provided by Sabic company) and anthracene with the chemical formula ($\text{C}_{14}\text{H}_{10}$) (Treated by the company Chemical Point) used in this work. Pure PC films were doping via different ratios of anthracene include 10, 20, 30, 40, 50 and 60 ml by using the casting technique. In briefly, pure PC film was prepared by dissolve 0.3 g of PC in 10 ml Chloroform. Then, the as-synthesis PC solutions cast onto glass petri dish with a diameter 10 cm and leave it dry at room temperature for 24 hrs to acquire homogeneous pure PC films. Furthermore, the anthracene solution with a concentration of (1×10^{-4} mol /L) was prepared according to the procedure explained in the previous study (17). Different ratios of anthracene solution 10, 20, 30, 40, 50 and 60 ml were added to the PC solution as mentioned earlier and mixed very well for 30 min. The mixture cast onto a glass petri dish with 10 cm diameter and left to dry at room temperature for (24 hrs) to get homogeneous doping PC films. Irradiate aging was carried out by UV-cabinet using a mercury lamp (Leicarm 2145; Germany). The as-prepared pure PC film and anthracene doping PC film were irradiated by UV light at wavelength 254 nm for various periods 5, 240, 288, and 360 hrs . Finally, the UV-Visible spectrophotometer type (T70/T80 Series UV/VIS Spectrometer) and FTIR spectroscopy (FTIR-600 biotech engineering management) used to characterize the photo-fries rearrangement products.

Results and Discussions:

Absorption Spectrum

The absorption spectra of pure PC film as well as, PC films doping by the various ratio of anthracene 10, 20, 30, 40, 50 and 60 ml were examined as presented in Fig. 2. Results show that the spectrum of pure PC film exhibit two major peaks at 305 nm and 345 nm, it's referring to the carbonyl group of polycarbonate. Meanwhile, anthracene doping PC films exhibited red-shift in absorption peaks at wavelengths 360 nm and 380 nm, respectively. These results could be attributed to the anthracene compound.

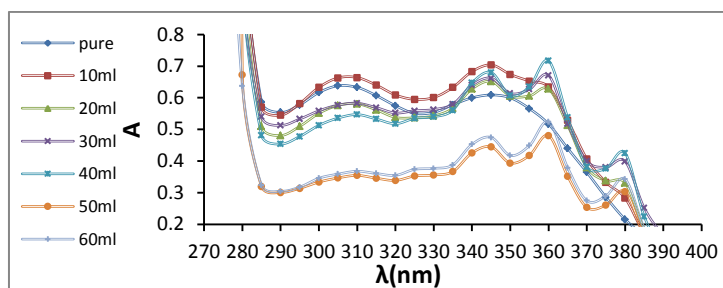


Figure 2. Absorption spectra of pure PC film and anthracene doped PC films for different doping ratio

The influence of UV radiation (254 nm) for irradiation times in the range of (5, 240, 288, 360 hrs) on the UV-Vis absorption properties of as-grown pure PC film and PC films doping with anthracene for the different ratio as illustrated in Table 1.

Figure 3 shows the effect of UV irradiation at 254 nm for 5 hours on the absorption properties of pure PC film and anthracene doping PC films. Due to the irradiation process, it seems that the second

peak of polycarbonate at (345 nm) disappear for pure PC film, which could be attributed to the variation in the structure characteristics. Spectra analyses of anthracene doping PC films for doping ratio 10, 20 and 30 ml were exhibited similar behavior. Besides that, a single absorption peak appears at the wavelength 325 nm of anthracene doping PC films for doping ratio 50 and 60 ml, which refers to the formation of phenylsalicylate (15, 16).

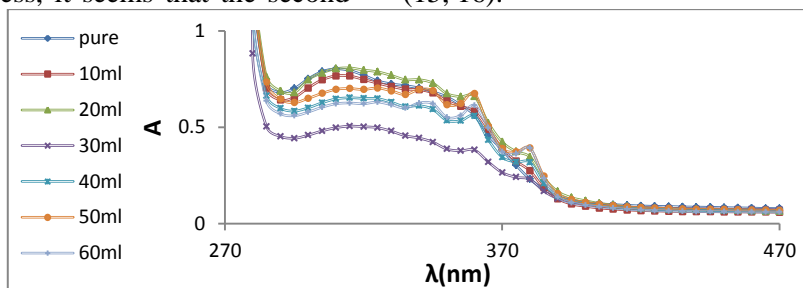


Figure 3. Effect of UV radiation on the absorption spectrum of pure PC film and anthracene doping PC films for different doping ratio for irradiation time (5 hrs)

For further study, the UV irradiation effect on the absorption spectra of pure PC and anthracene doping PC films for irradiation time 240 hrs was also examined as shown in Fig. 4. Figure 4 reveals that the polycarbonate absorption peaks around 305 nm and 345 nm were disappeared, that endorsed to

scission of carbonate linkage and phenylsalicylate appears at the wavelength 320 nm (15, 16). Furthermore, absorption peaks of anthracene at 360 nm and 380 nm were vanished due to the irradiation process for (240 hrs).

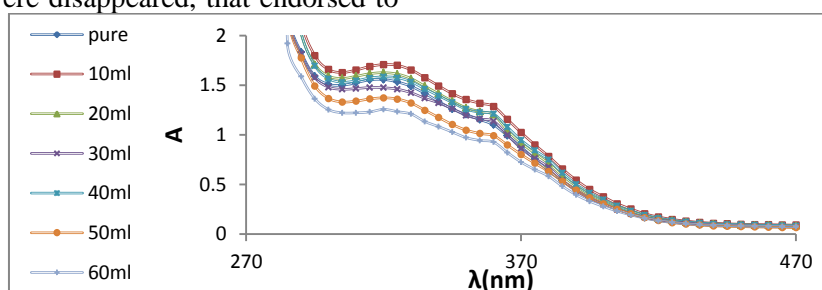


Figure 4. Influence of UV radiation on absorption spectra of pure PC film and anthracene doping PC films for altered doping ratio for irradiation time (240 hrs)

Figure 5 shows the absorption spectra of UV irradiation for 288 hrs of pure PC film and anthracene doping PC films. The absorption peak of a dihydroxybenzophenone compound at a wavelength 360 nm in anthracene doping PC film for doping ratio 60 ml was toward to appear (15, 16), as confirmed in Fig. 5. Due to the photo-fries

rearrangement in PC film; this leads to the construction of phenylsalicylate and dihydroxybenzophenone. Based on the UV irradiation, the oxygen from the weak C-O bond reorganizes its position to shape a stronger O-H bond which resulting in phenylsalicylate form. Furthermore, when the oxygen of the C-O group in

phenylsalicylate structure suffer from further reorganization to form strong O-H bond then it results in a dihydroxybenzophenone configuration.

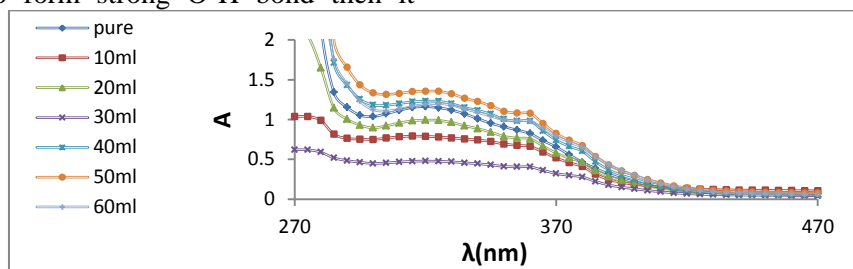


Figure 5. The UV irradiation effect (wavelength 254 nm for 288 hrs) on absorption spectra of pure PC film and anthracene doping PC films for various doping ratio

Moreover, the influence of irradiation time (360 hrs) via UV radiation on the absorption characteristics of pure PC sample and anthracene doping PC samples were displayed in Fig. 6. The dihydroxybenzophenone compound appears in anthracene doping PC films for doping ratio 30 ml, 40 ml, 50 ml, and 60 ml at wavelength 360 nm (15,

16). As well as, the new absorption peak appears at wavelength 340 nm in anthracene doping PC film for doping ratio 60 ml. Formation of phenylsalicylate and dihydroxybenzophenone compounds refers to the photo-fries rearrangement is take place (15).

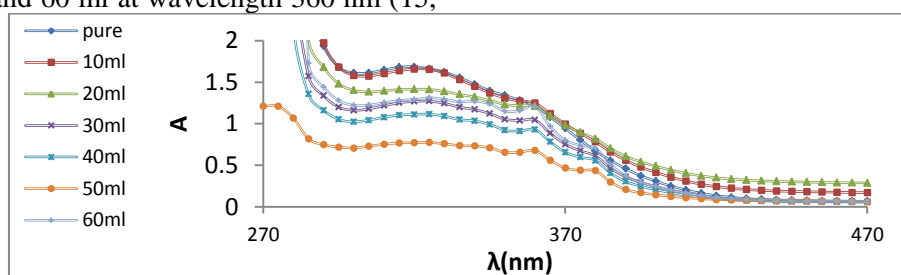


Figure 6. The influence of irradiation time (360 hrs) via UV radiation on the absorption characteristics of pure PC sample and anthracene doping PC samples.

Table 1. The absorption peaks for un-irradiated and irradiated pure PC and anthracene doping PC films with UV radiation at wavelength (254 nm)

Time	Doping Ratio of anthracene -PC Films							
		PC	10ml	20ml	30ml	40ml	50ml	60ml
0 hrs	λ_{max} (nm)	305	310	310	310	310	310	310
	Absorbance	0.638	0.664	0.581	0.583	0.547	0.355	0.368
	λ_{max} (nm)	345	345	345	345	345	345	345
	Absorbance	0.609	0.704	0.652	0.662	0.68	0.445	0.475
	λ_{max} (nm)			360	360	360	360	360
	Absorbance			0.628	0.671	0.717	0.481	0.524
	λ_{max} (nm)				380	380	380	380
	Absorbance				0.399	0.425	0.304	0.343
	λ_{max} (nm)	310	310	315	315	315	315	315
	Absorbance	0.803	0.767	0.808	0.506	0.653	0.701	0.626
	λ_{max} (nm)						325	325
	Absorbance						0.704	0.631
5 hrs	λ_{max} (nm)					340	340	340
	Absorbance					0.612	0.696	0.626
	λ_{max} (nm)			360	360	360	360	360
	Absorbance			0.66	0.382	0.558	0.675	0.614
	λ_{max} (nm)						380	380
	Absorbance						0.392	0.392
240 hrs	λ_{max} (nm)	320	320	320	315	320	320	320
	Absorbance	1.557	1.708	1.626	1.477	1.584	1.373	1.257
	λ_{max} (nm)	320	315	320	320	320	320	325
	Absorbance	1.163	0.792	0.994	0.479	1.232	1.358	1.201
288 hrs	λ_{max} (nm)							360
	Absorbance							0.983
	λ_{max} (nm)	320	320	320	325	325	325	325
	Absorbance	1.686	1.659	1.416	1.271	1.116	0.774	1.312
360 hrs	λ_{max} (nm)							340
	Absorbance							1.27
	λ_{max} (nm)				360	360	360	360
	Absorbance				1.045	0.929	0.678	1.209

FTIR Spectra Analysis

Figure 7 shows the FTIR spectra of pure PC and anthracene doping PC films with different doping ratios 10, 20, 30, 40, 50 and 60 ml. It can be

easily seen that no observable peaks are seen to be appearing or disappearing but an only slight change in the position of bonds in anthracene doping PC films for different doping ratios.

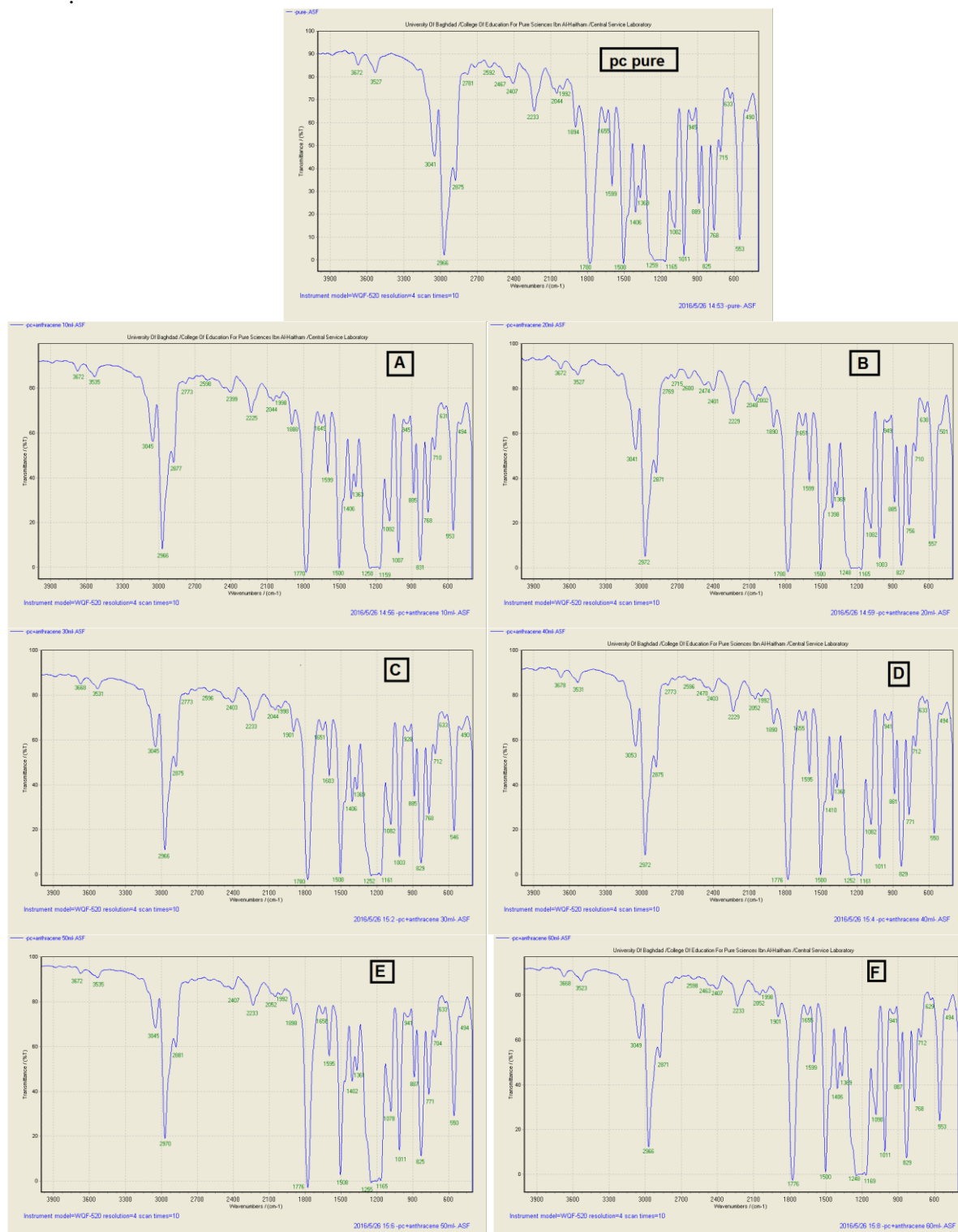


Figure 7. FTIR spectra of pure PC and anthracene doping PC films for different doping ratios: (A) 10 ml, (B) 20 ml, (C) 30 ml, (D) 40 ml, (E) 50 ml and (F) 60 ml

The UV irradiation influence at the wavelength 254 nm and irradiation time 5 hrs on FTIR spectra of pure PC and anthracene doping PC

films for various doping ratios were presented in Fig. 8. The phenylsalicylate appears in the C=O stretching region at 1685 cm^{-1} of anthracene doping

PC films for doping ratio (50 and 60 ml), this result supports by literature (15).

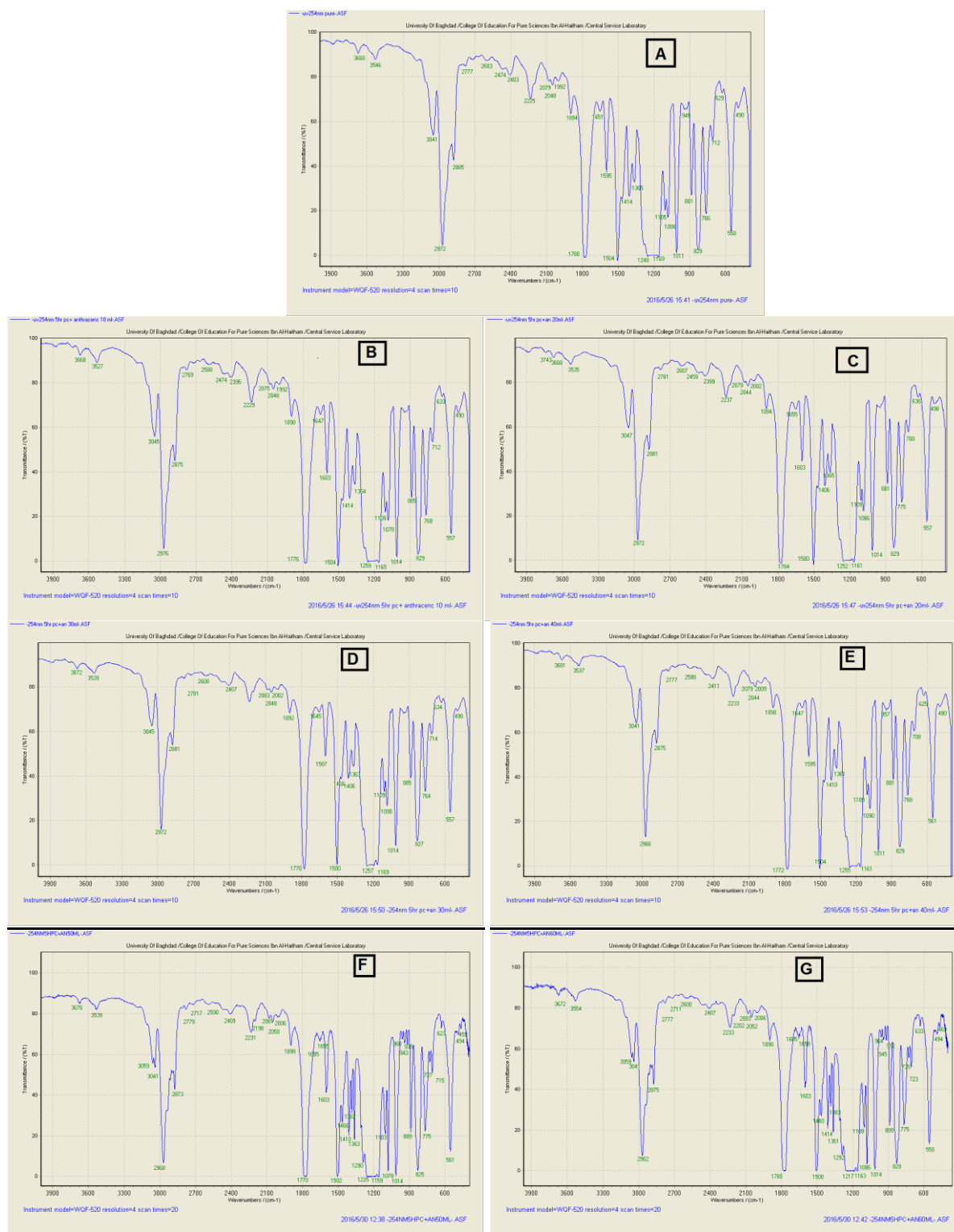


Figure 8. The UV irradiation influence at wavelength (254 nm) and irradiation time (5 hrs) on FTIR spectra of (A) pure PC; and anthracene doping PC films for various doping ratio (B) 10 ml, (C) 20 ml, (D) 30 ml, (E) 40 ml, (F) 50 ml and (G) 60 ml

The effect of UV radiation at the wavelength 254nm on FTIR spectra of pure PC and anthracene doping PC films for different doping ratios for 240 hrs shows in Fig. 9. The phenylsalicylate appear in pure PC at (1685 cm^{-1}) and in anthracene doping PC

films at 1689 cm^{-1} for doping ratio 10 and 40 ml as well as at 1682 cm^{-1} for doping ratio 20 and 30 ml and around 1685 cm^{-1} for doping ratio 50 and 60 ml (15).

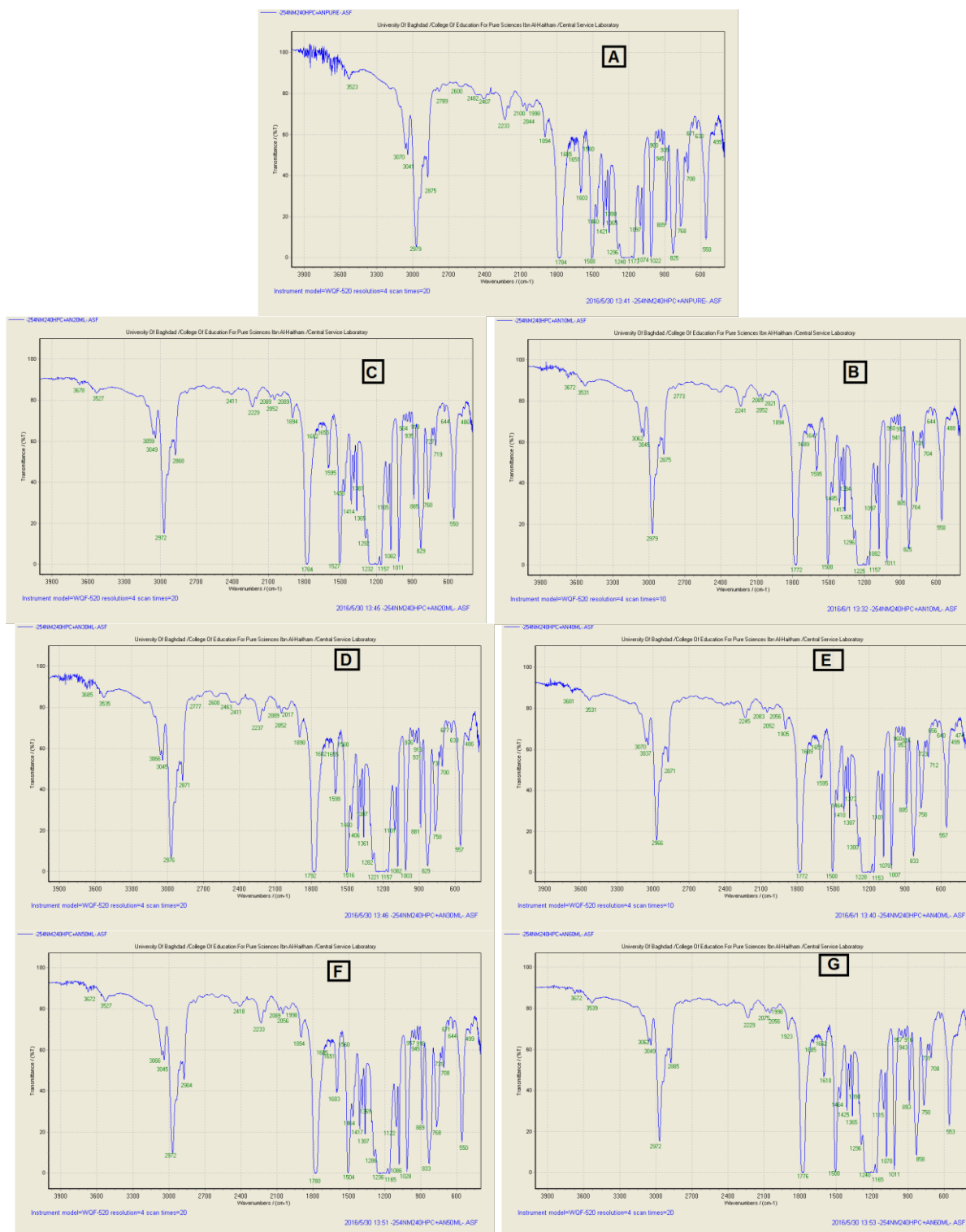


Figure 9. The effect of UV light at wavelength (254 nm) for (240 hrs) on FTIR spectra of (A) pure PC sample; and PC/anthracene films for different doping ratio (B)10 ml, (C) 20 ml, (D) 30 ml, (E) 40 ml, (F) 50 ml and (G) 60 ml

Furthermore, the special effects of UV radiation at the wavelength 254 nm for 288 hrs on FTIR spectra of pure PC film and anthracene doping PC films for diverse doping ratio demonstrated in Fig. 10. The phenylsalicylate complex appears in pure PC at 1685 cm^{-1} and in anthracene doping PC films at 1685 cm^{-1} for doping

ratio 10 and 20 ml. While, FTIR peaks at 1689 cm^{-1} were revealed for doping ratio 30, 50, and 60 ml and at 1682 cm^{-1} for doping ratio 40 ml. The dihydroxybenzophenone appears in anthracene doping PC films for doping ratio of 60 ml at 1626 cm^{-1} as supported in previous study (15). These results can be ascribed to the absorption spectrum of

a polymer is generally dependent on the chromophores existing in its structure. The presence of a substituent group in a chromophore can shift

the absorption band to either shorter wavelength or longer wavelength.

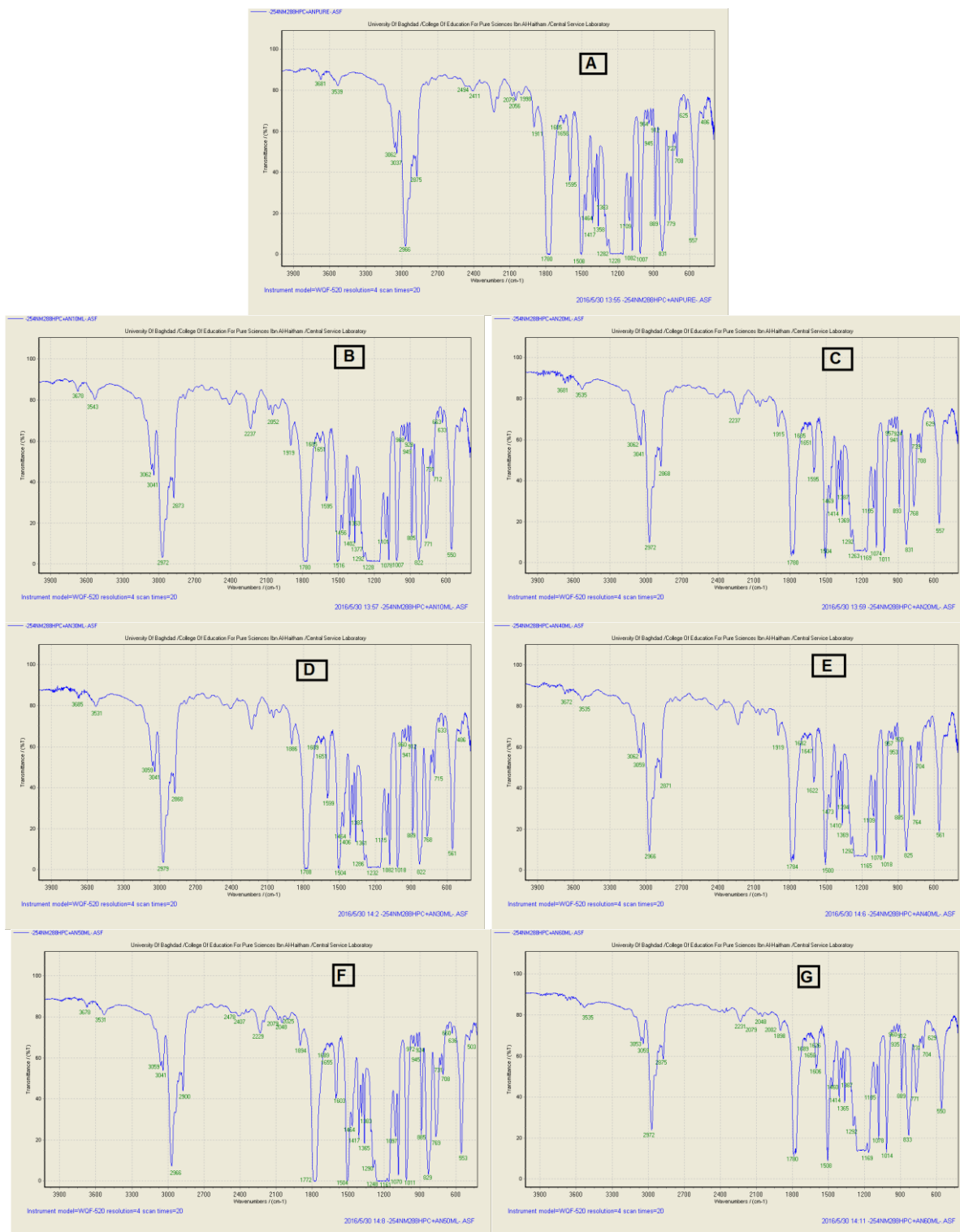


Figure 10. The effect of UV light at wavelength (254nm) for (288 hrs) on FTIR spectra of (A) pure PC film; and PC/anthracene films for different doping ratio (B)10 ml, (C) 20 ml, (D) 30 ml, (E) 40 ml, (F) 50 ml and (G) 60 ml

The effect of UV radiation at wavelength 254nm on FTIR spectra of pure PC film and anthracene doping PC films for different doping

ratios for 360 hrs shows in Fig. 11. The phenylsalicylate appears in pure PC film at 1685 cm^{-1} and in anthracene doping PC films at 1685

cm^{-1} for doping ratio 10, 20, 40, 50, and 60 ml . Furthermore, the FTIR spectra in Fig. 11 show a distinguished peak at 1684 cm^{-1} for the doping ratio 30 ml. The dihydroxylbenzophenone perform in anthracene doping PC films at 1635 cm^{-1} for doping ratio 30 ml , and at 1637 cm^{-1} for doping ratio 40 ml as well as around 1633 cm^{-1} for

doping ratio 60 ml (18). This change in the FTIR peaks position can be attributed as due to the manipulation in the structural properties of the as-synthesized material. Where, at 360 hrs longer UV exposures the polymer becomes further photo-instability hence photo-fries reaction is taking place.

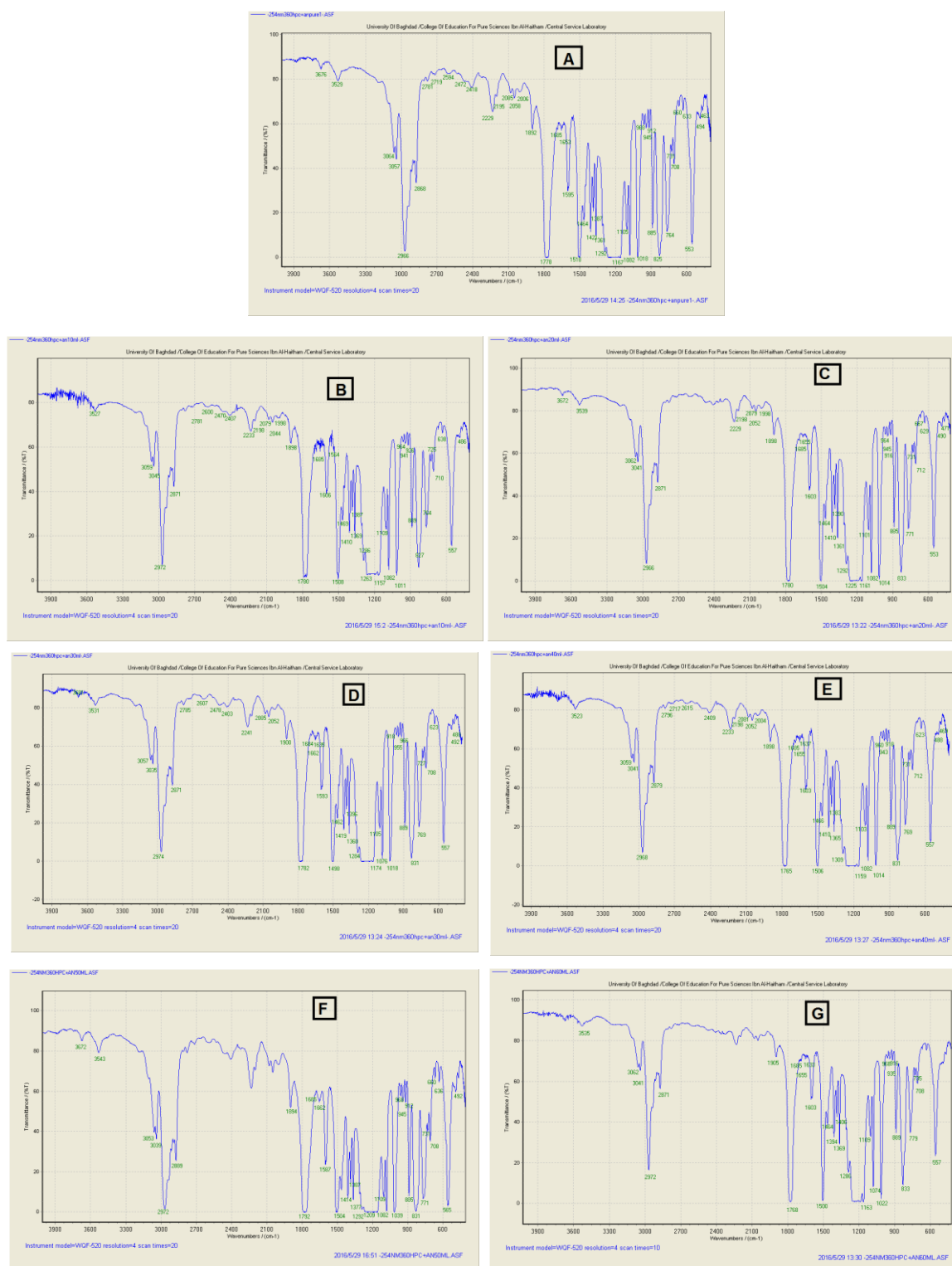


Figure 11. The influence of UV light at wavelength (254 nm) for (360 hrs) on FTIR spectra: (A) pure PC film; and PC/anthracene films for altered doping ratio, (B)10 ml, (C) 20 ml, (D) 30 ml, (E) 40 ml, (F) 50 ml and (G) 60 ml.

Conclusions:

Successfully synthesis of pure polycarbonate (PC) and anthracene doping PC films for different doping ratios 10, 20, 30, 40, 50 and 60 ml was achieved by using cast technique. This study proved that there was a strong influence of UV radiation 254 nm at various irradiation times 5, 240, 288, and 360 hrs on the absorption spectra of as-grown samples. The results reveal that the formation of phenylsalicylate and dihydroxybenzophenone compounds. Thus, the photo-fries reaction is take place. Furthermore, anthracene doping of PC film with appropriate ratio leads to accelerate the photo-fries rearrangement of polycarbonate in as-grown films. Apportion peaks of as-prepared films of PC and anthracene doping PC films revealed appear and disappeared different absorption peaks depend on UV irradiation time and anthracene doping ratio 10, 20, 30, 40, 50 and 60 ml of films.

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Authors' declaration:

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are mine ours. Besides, the Figures and images, which are not mine ours, have been given the permission for re-publication attached with the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee in University of Baghdad.

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تأثير نسبة التطعيم بالأنثراسين وزمن التشعيع بالأشعة فوق البنفسجية على إعادة ترتيب تفاعلات الفوتو- فريز للبولي كاربونيت

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² قسم الفيزياء، كلية التربية للعلوم الصرفة ابن الهيثم، جامعة بغداد، بغداد، العراق.

الخلاصة:

تم في هذه الدراسة تحضير افلام نقيه من البولي كاربونيت (PC) وافلام اخرى من البولي كاربونيت مطعمة بنسب مختلفة من مادة الانتراسين (10 ، 20 ، 30 ، 40 ، 50 و 60 مل) بواسطة تقنية الصب لاجل بحث مدى تأثير التطعيم بمادة الانتراسين على اعادة تفاعلات الفوتو-فريز للبولي كاربونيت. كذلك تم دراسة تأثير التشعيع بواسطة الاشعة فوق بنفسجية بطول موجي مقداره (254 نانومتر) لفترات زمنية مختلفة للافلام النقيه من البولي كاربونيت والمطعمة بمادة الانتراسين. تمت متابعة تفاعلات الفوتو-فريز التي تحدث في افلام البولي كاربونيت النقيه وافلام البولي كاربونيت المطعمة بالانتراسين باستخدام التحليل الطيفي للاشعة فوق البنفسجية و تحليل فورير للاشعة تحت الحمراء. حدوث تفاعلات الفوتو-فريز يؤدي إلى تفكك ترابط الكاربونيت و تشكيل الفينول سلاسلات و ثنائي هيدروكسي بنزوفينون. نتائج فحص طيف الاشعة فوق البنفسجية (UV) اكدت اختفاء قمم البولي كاربونيت، في حين أن قمم الفينول سلاسلات و ثنائي هيدروكسي بنزوفينون قد ظهرت عند (320 نانومتر) و (360 نانومتر)، على التوالي. يمكن تمييز المركب ثنائي هيدروكسي ثنائي الفينيل من خلال طيف الاشعة فوق البنفسجية عند الطول الموجي (340 نانومتر). طيف الاشعة تحت الحمراء (FTIR) يدعم تكوين فينول سلاسلات ومركبات ثنائي هيدروكسي بنزوفينون التي تظهر في منطقة الكاربونيل في (1689 سم⁻¹) و (1630 سم⁻¹) ، على التوالي. اثبتت الدراسة ان تطعيم الافلام بمادة الانتراسين يسرع من تفاعلات الفوتو-فريز في افلام البولي كاربونيت بسبب ان مادة الانتراسين تؤدي إلى تكوين أكسجين احادي (¹O₂). ان تكون الاوكسجين الاحادي (¹O₂) يؤدي الى تكوين هيدرو بيروكسيد ، والذي يمكن أن يتحلل ويسبب حدوث سلسلة من الانقسام يؤدي الى تشكيل محطة لمجموعة الكاربونيل. اثبتت هذه الدراسة ان وجود مجموعات الكاربونيل في البوليمر يجعله عرضة للتدهور.

الكلمات المفتاحية: الانتراسين، تأثير نسبة التطعيم، طيف الاشعة تحت الحمراء، بولي كاربونيت، تفاعلات الفوتو-فريز.