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Green synthesis of cobalt nanoxide and study its effect on polymer photo-physical properties (Methylmetha Acrylate)

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***Abstract*---**Because of its applications in the sectors of environmental protection and physical health, biodegradable polymers have gotten a lot of interest. The study included the preparation and study of optical fragmentation of poly (methyl methacrylate) films using the casting method, where weight ratios (0.05, 0.1, 0.2, 0.4 and 0.8) were added of nano-oxide CdO NPs prepared in the laboratory from a plant (grape leaves) and at (55 ± 5).) microns before and after exposure to ultraviolet rays at a wavelength (365) nm the irradiation was carried out at times (0, 15, 40, 80, 120) One hour respectively, and the optical fractionation was followed up by using a UV-visible a UV-Vis spectrophotometer to calculate the dissociation velocity constant (Kd), and infrared spectroscopy was used to calculate The growth coefficient of the carbonyl and hydroxyl groups, to monitor the change in the viscous molecular weight rate, the viscosity technique was used, The results showed that the decomposition velocity constant is directly proportional to the concentration of nanoxide cobalt that was added with the irradiation time. modern techniques (AFM, SEM, and XRD) were used to determine the rate of development of the carbonyl and hydrologic ejectors of the nanomaterial, According to SEM images, cobalt nanoxide CdO NPs was found in the form of irregular spherical particles, The surface topography and minute volume of the cobalt nanoxide were analyzed using atomic force microscope

***Keywords*---**poly (methylmetha acrylate), cobalt nanoxide, grape leaves.

Introduction

Polymers play a significant role in a variety of industries, including packaging, medicinal uses, adhesives, paint, certain clothes, materials, and engineering [1]. Nanomaterials, which vary in size from 1 to 100 nm nanometers, have new and distinct properties that set them apart from the original components from which they were formed. These characteristics make them relevant to a wide range of life and industrial technology fields, including solar energy processes, medical biology, catalysts, and electronics [2]. The use of plant extracts in the green synthesis of nanoparticles is a relatively recent field of study. It is probably preferred to chemical or microbial synthesis because it avoids the difficult process of nanoparticle formation and can achieve large-scale manufacturing at high quality and low cost [3]. Poly (methylmetha acrylate) one of the polymers with good stability owing to its hard polymeric chains, and it is one of the polymers that is resistant to moisture, acids, and bases, and many organic solvents cannot dissolve it. It is transparent and colorless, has optical properties, and is one of the commercially available polymers [4]. Having a distinctive combination of its optical properties such as thermal stability and easy formation as well as good mechanical properties and moisture resistance led to its use in a wide range of applications [5]. Poly (Methylmetha acrylate) PMMA is more environmentally stable than other plastics such as polyethylene and polystyrene, making it a better choice for outdoor applications [6]. It is used in wide areas of use, including in engineering and decoration, as well as in household appliances and medical applications, as it is used in dental dentures and in the areas of manufacturing laser discs [7]. The glass transition temperature (Tg) of inactive PMMA is 105°C (221°F), Tg values for PMMA range from 85 to 165°C (185 to 329°F). So, PMMA is an organic glass at room temperature [8]. The research aims are to make a cobalt oxide nanomaterial and use it as a catalyst for the photodegradation of the polymer (methylmetha acrylate) PMMA, as well as to investigate the dissociation mechanism using infrared and ultraviolet-visible techniques and the rate of change in molecular weight using a viscosity scale.

Materials and Methods

Devices and materials utilized

Aqueous cobalt nitrate ($Co(NO_3)_2.6H_2O$), sodium hydroxide NaOH, poly (methylmetha acrylate) PMMA, chloroform solvent, distilled water, dimethyl sulfoxide solvent, ethanol solvent, and dislocated water Atomic force microscope (AFM), X-ray diffraction(XRD), scanning electron microscope (SEM), UV-Vis and FTIR spectroscopy, infrared spectroscopy, Ostwald viscometer and centrifuge were used.

Preparation of plant extract

We prepare the grape leaves by taking samples of them and then washing them. (Three times) with deionized water, dried in the shade, and crushed with a mortar to make a powder that would be sifted to remove any particles adhering to it and kept in the shade. Weighed 2 gm of powder and placed it in a glass jar. To get the brown-colored extract, add a (100 mL) of ion-free water, swirl stirring

continuously with a magnetic motor stirrer, and heat at 80°C for 30 minutes. Allow to cool to ambient temperature before filtering with filter sheets for a solution to be poured in test tubes and centrifuged at speed (1200) cycle/minute to remove any leftover fibre, and then storing the solution in at low temperature.[9]

Preparation of cobalt nanoxide CoO NPs

CoO NPs were prepared by the green method using the prepared plant extract from grape leaves, where (0.6 g) of aqueous cobalt nitrate ($Co(NO_3)_2.6H_2O$) was dissolved in 40 ml mL of deionized water in beaker with stirring by a glass at room temperature, adding 20ml mL of plant extracts gradually using a stirring burette, and then raising the temperature to (70-60 ^{0}C) for two hours during which 0.1M of NaOH was added in the form of drops until The the color of the solution changed to a dark brown color at pH = 8. The solution was left overnight and then the precipitate was separated by centrifugation for 10 minutes at a speed of 1200, the precipitate was washed three times with deionized water and absolute ethanol to get rid of impurities, the precipitate was dried in an electric oven at a temperature of (60^{0}C). for 8-9 hours, then calcined at 500^{0}C in an earthenware jar for 60 minutes and then left to cool, the precipitate was ground and kept in a sealed container at room temperature.

Poly purification (methylmetha acrylate)

Redeposition was achieved by dissolving 20 gm of poly (methylmetha acrylate) PMMA in 100 mL chloroform solvent for one hour while stirring continuously with a magnetic bar to guarantee homogeneity. Then, with steady stirring, the homogenous solution is added to 250 mL of ethanol alcohol until the polymer is deposited.[10]

Preparation of a poly solution (methylmetha acrylate)

9gm poly (methylmetha acrylate) is dissolved in 100 mL chloroform solvent and then mixed continuously for one hour at room temperature using the magnetic motor. [10]

Polymeric Film preparation

The following processes were followed to make polymeric films for PMMA:

1. To a 3 mL PMMA solution, add cobalt nanoxide CoO NPs produced in the following weight ratios (0.00 0.05,0.01, 0. 2, 0.4 and 0.8) percent, then pour the liquid into glass molds constructed from sliced glass.
2. Make sure the solution and oxide are homogeneous, and that there are no bubbles on the polymeric foil, then let the mixture at room temperature (RT) for 24 hours until it dries up without being exposed to sunlight.
3. Blade and forceps are used to remove the polymeric films 24 hours later.
4. At (55±5) micrometers, we utilize the Micrometer to alter the thickness of polymer sheets.

5. For dimensions corresponding to spectral measurement cells (UV-Visible, FT-IR), polymeric films are sliced at the following dimensions (3.5 cm) and viewed (1.5 cm) .

Follow-up the optical fragmentation for poly (methylmetha acrylate)
Infrared spectroscopy

By measuring the infrared spectrum to determine the intensity of the beams absorbed prior to irradiation and after each period of irradiation of pure PMMA chips containing various concentrations of cobalt nanoxide, we use (FT-IR) to track the growth rate of carbonyl group (I_{CO}). where the carbonyl group absorption site (1735 cm^{-1}), through which the growth coefficients of carbonyl (Ico) were found by applying equation (1), adopting the package coefficient, and choosing location (1435 cm-1) as the reference point for PMMA.

$$\mathbf{I}_{(S)} = \frac{\mathbf{A}_{(S)}}{\mathbf{A}_{(R)}} \qquad \mathbf{1}$$

Where, I(S) denotes the study group's growth coefficient and A(S) the study group's absorption. Reference summit absorption, A (R).
Ultraviolet-visual UV-Vis spectroscopy : To determine the absorption value of polymeric films between 200 and 800 nm, we employ UV-VIS light. Using the following equation, we can get the rate of photosynthesis (Kd).

$$2 \qquad ln\ ln\ (A\square\square - At) = ln\ ln\ (A\square\square - A_0) - Kdt$$

Ao represents the pre-irradiation of polymer chips, At the absorption of polymer chips themselves at the time of irradiation (t) and $A\square\square$the absorption of polymeric chips at the end of time. From drawing the relationship between $ln\ (A\square\square - At)$ with irradiation time (t), the slope was found to represent the dissociation constant (-Kd).

Viscosity measurement : Calculating the viscosity rate by using the (Mark-Houwink) equation yields the Mv viscous molecular weight rate of polymer chips.

$$[\boldsymbol{\eta}] = \boldsymbol{K}(\boldsymbol{M_V})^{\boldsymbol{a}} \qquad \mathbf{3}$$

Using measurements of intrinsic viscosity in the chloroform solvent at 200C in the presence and absence of the addition, the molecular weight of a poly (methylmetha acrylate) PMMA was determined by using the equation (4) [12]

$$[\eta] = \mathbf{0.6 \times 10^{-4}\ M_v^{0.79}} \qquad (\boldsymbol{PMMA}) \quad \mathbf{4}$$

The following equations were used to compute the degree of fragmentation (a) and the numerical rate of the series cut (S).[11]

$$\alpha = \frac{\mathbf{1}}{\mathbf{P_t}} - \frac{\mathbf{1}}{\mathbf{P_\circ}} \qquad \mathbf{5}$$

$$\mathbf{S = \alpha\ p_0} \qquad \mathbf{6}$$

P0: the numerical rate of degree of polymerization before to irradiation. Pt: the numerical rate of polymerization at time (t) of irradiation.

Microscopy

We employ a novel microscope to monitor how UV radiation affects the surface characteristics of polymer chips. Before and after irradiation, photographs of pure PMMA and polymeric films containing cobalt nanoxide were obtained.

Results and Discussion

Atomic force microscope (AFM) test

As atomic force microscopy probes scan the topography of the material with extremely tiny precision, it provides information on the topography of the surface of the material with great accuracy up to the atomic level. It provides details on the size of the minutes and the texture of the surface by displaying two- and three-dimensional photographs of the created cobalt nanoxide surface in Fig. 1. The volume of the prepared minutes has a diameter of approximately 8.27 nm.

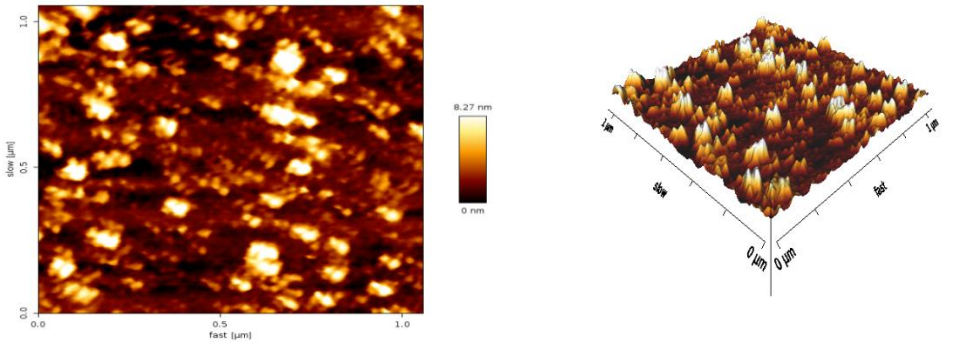


Fig. 1 : 2D and 3D AFM image of nanoparticle oxide tested in green synthesis method

Scanning electron microscope (SEM) Test

A beam of electrons is shone into the region to be seen in this imaging technique, causing the electrons to interact with the surface atoms and produce three different types of rays: secondary electrons, backscattered electrons, and X-rays. The range of magnification is 25–250.000 thousand times. to ascertain the nanoscale's surface morphology and particle form [12] as seen in form (2). Images taken using a scanning electron microscope clearly demonstrate the formation of nanoparticles with irregularly shaped spheres, with an average particle size of around 32.26 nm.

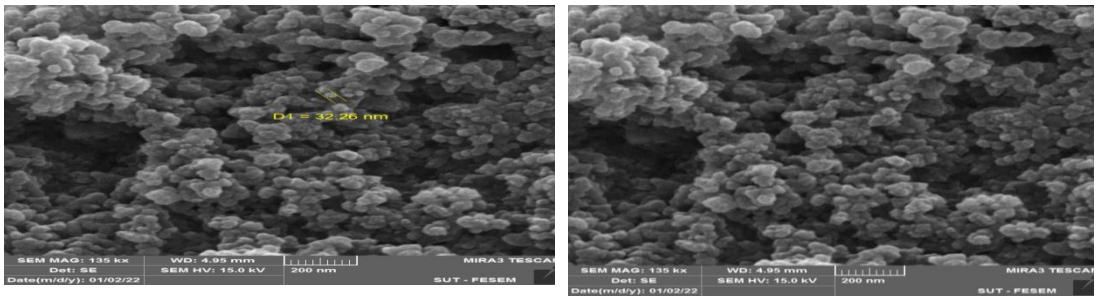


Fig. 2 : Images showing cobalt nanoxide CoO NPs prepared by scanning electron microscope SEM

X-ray diffraction (XRD) Test

When these rays are shone at various angles and forms, it is utilized to get peaks and determine where the peaks occur. (3) shows the X-ray diffraction patterns of the prepared cobalt nanoxide compared to its standard reference NO.(43–1003)) JCPDS shall be attributed at the angles (°21.35 ,°36.02 ,°41.23 ,°52.30 ,°59.51 ,°70.41) respectively, as shown in Table 1. These indicators and data indicate and confirm that they are prepared cobalt nanoxide powder with a cubic face-centered composition by standard reference and when matched with the ICBD card (JCPDS) found to be identical, and also consistent with literature results [13].

Table 1 : Structural coefficients of prepared cobalt nanoxide CoO NPs obtained from XRD X-ray diffraction analysis

2θ(degree)	
HK1	Observed
2.3616	21.35°
0.9840	36.02°
1.1808	41.23°
1.0800	52.30°
1.5744	59.51°
1.5744	70.41°

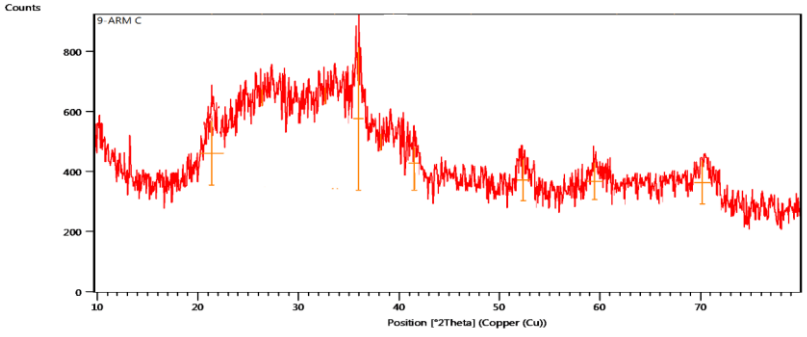


Fig. 3 : CoO nanoxide XRD compared to standard reference NO.(43–1003) JCPDS

Ultraviolet-visible spectroscopy (UV-VIS) test

We note that A peak of 265 nanometers (methylmetha acrylate) will be observed, and it is through this summit that the change is followed by absorption values at different times. It was noted that the pure polymer chips containing the added nanomaterial began with a slight shattering after increased irradiation periods of 80 hours and an apparent increase in fracture at 130 hours as a result of the chips being exposed to polymerization for different irradiation periods as in Table 2. The absorption values of the pure (methylmetha acrylate) have been shown to increase exponentially with irradiation time. So do the absorption values of PMMA containing different concentrations of CoO nanoscale where absorption increases with increased concentration of added nanomaterial and for the same irradiation time. (55±5) micrometer containing different concentrations of nanomaterial at different irradiation times is due to increased light-absorbing functional totals with the highest absorption value at time of 130 hours [14] as in (4) and (5) forms. And by the relationship between ln (A□ – A0) with irradiation time as shown in the shapes (6) and (7) as a straight line has been obtained and this indicates that the optical fragmentation reaction of cobalt nanoxide is a first-order reaction [16]. By calculating the slop slope, the Kd photosynthetic decomposition velocity constant was measured for multiple added cobalt nanoxide CoO NPs (methylmetha acrylate) and according to the stated concentrations. The photodegradable velocity constant was found to be directly proportional to the concentration of the nanomaterial added to the polymer as shown in Table 3.

Table 2 : Pure PMMA absorption values containing different concentrations of CoO. CoO NPs

Absorption At					**Irradiation time (hour)** / **Type of chips**
120	80	40	15	0.0	
0.454	0.350	0.263	0.209	0.156	PMMA
0.548	0.448	0.328	0.251	0.160	PMMA + 0.05 % CoO
0.630	0.508	0.414	0.303	0.163	PMMA + 0.1 % CoO
0.698	0.550	0.454	0.350	0.184	PMMA + 0.2 % CoO
0.757	0.628	0.537	0.421	0.189	PMMA + 0.4 % CoO
0.875	0.780	0.585	0.499	0.194	PMMA + 0.8 % CoO

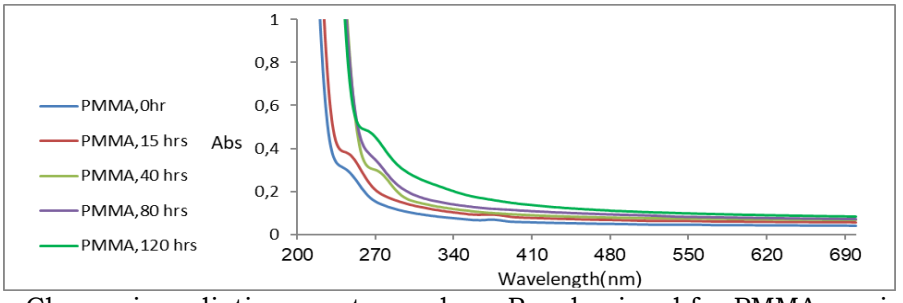


Fig. 4 : Change in radiation spectrum above Purple-visual for PMMA movies free of additives thick (55 ± 5) Micron at different irradiation times.

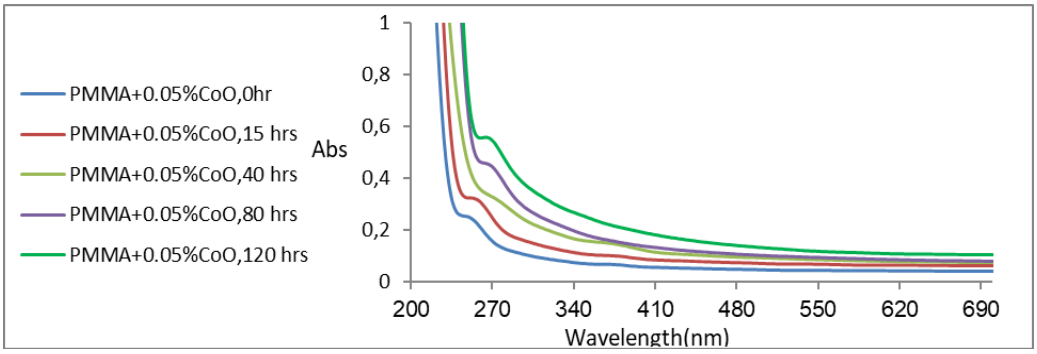


Fig. 5 : Change in UV-Vislafilm spectrum containing concentration of 0.05% of nanomaterials thick (55 ± 5) micron at different irradiation times.

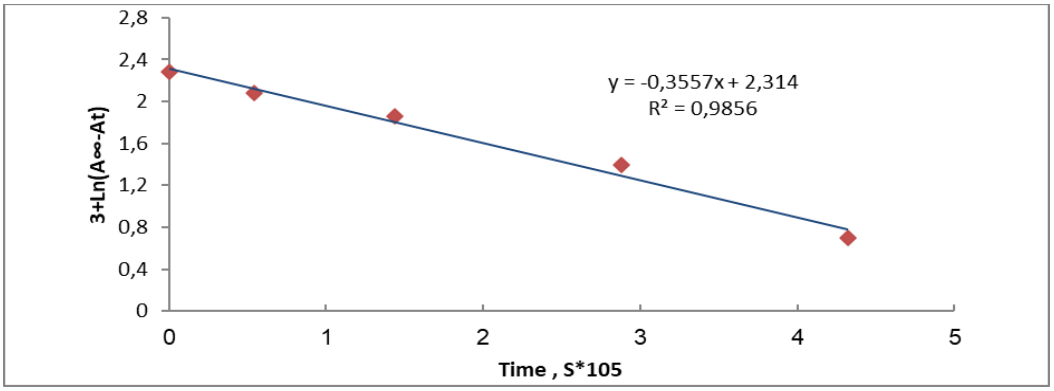


Fig. 6 : Relationship between natural logarithm to absorb copper oxide (CoO) concentration in 0.05% movies thickness (55 ± 5) micron with irradiation time.

Table 3 :Kd Breakup Velocity constants values for PMMA films containin CoO nanomaterials

Disintegration velocity constant Kd (Sec.)$^{-1}$x10^{-5}	% concentration
0.335	PMMA + 0.05 % CoO
0.354	PMMA + 0.1 % CoO
0.366	PMMA + 0.2 % CoO
0.376	PMMA + 0.4 % CoO
0.422	PMMA + 0.8 % CoO

Infrared spectroscopy FTIR test

The optical fragmentation of a poly methylmetha acrylate PMMA is followed by the follow-up of carbonyl group absorption packages (C=O) generated as a result of the polymer being affected by photons by the presence of oxygen. The carbonyl group (C=O) appears at (1735 cm-1) [17-18] which showed a spectral change compared to the second beam, the nanomaterial (CuO) is behaved as an accelerator of the dissociation process. Changes in the infrared spectrum were shown by increased irradiation times compared to the infrared spectrum of the

pure methylmetha acrylite before irradiation as shown in shapes (8) and (9) and observed through FT-IR infrared spectrum readings of PMMA films. The top of the absorption coefficient of the film containing the nanomaterial is higher than the top of the pure film absorption coefficient when compared by (FT-IR) spectrum after irradiation for 120 hrs. As shown in (10) form, coefficient (ICO) of the multiple metha acrylate and the increased irradiation time and when the irradiation time is established. This applies to the rest of the different irradiation periods as shown in the form (11). It can be observed that the speed of disintegration by optical oxidation is directly proportional to both irradiation time and the concentration of cobalt nanoxide added to PMMA films through form (12). This increase is associated with increased UV absorption of polymer films during irradiation as the absorption coefficient values increase. (I_{CO}) This increases the optical oxidation speed of polymer films.

Table 4 : ICO values with PMMA irradiation time containing differe concentrations of material (CoO) nanoscale

I_{CO}					Irradiation time (hour) / Percentage %
160	120	80	40	0.0	
2. 628	2. 256	1. 797	1. 608	1.530	PMMA
2.990	2.554	2.098	1.750	1.606	PMMA + 0.05 % CoO
3. 367	2.894	2.345	1.887	1.653	PMMA + 0.1% CoO
3. 689	3.212	2.609	1.980	1.689	PMMA + 0.2 % CoO
4.003	3.532	3.078	2.435	1.743	PMMA + 0.4 % CoO
4. 301	3.897	3. 454	2.705	1.821	PMMA + 0.8% CoO

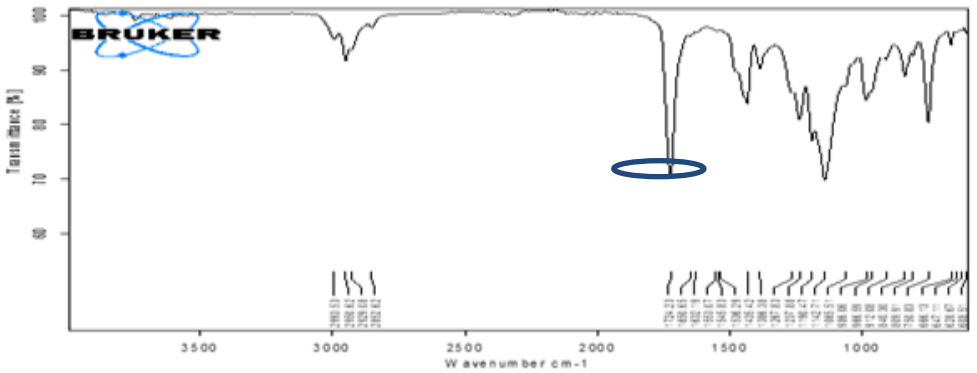


Fig. 8 : Infrared spectrum of additive-free multi-PMMA chips before irradiation.

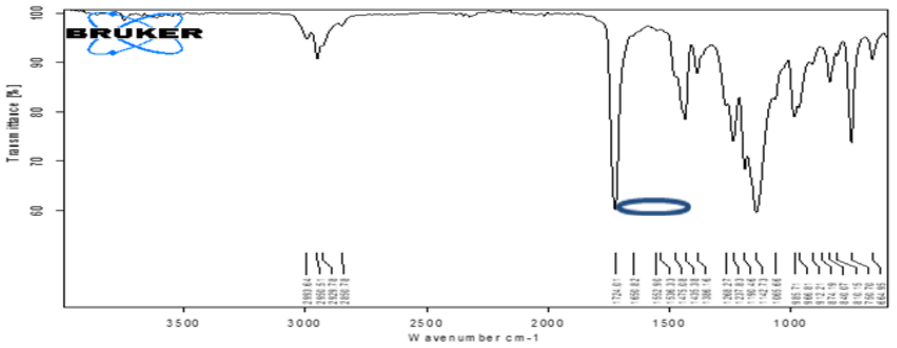


Fig.9 : Infrared spectrum of additive-free multi PMMA chips after 120-hour irradiation

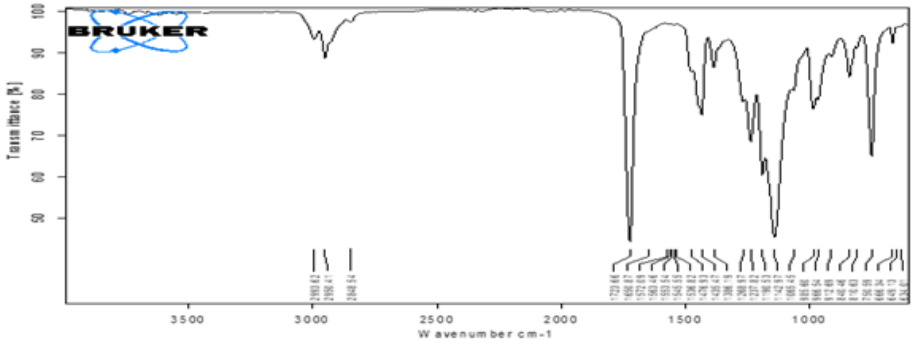


Fig. 10 : Infrared spectrum of multiple PMMA chips containing nanomaterial (CoO) at concentration (0.8%) and irradiation time (120) hours.

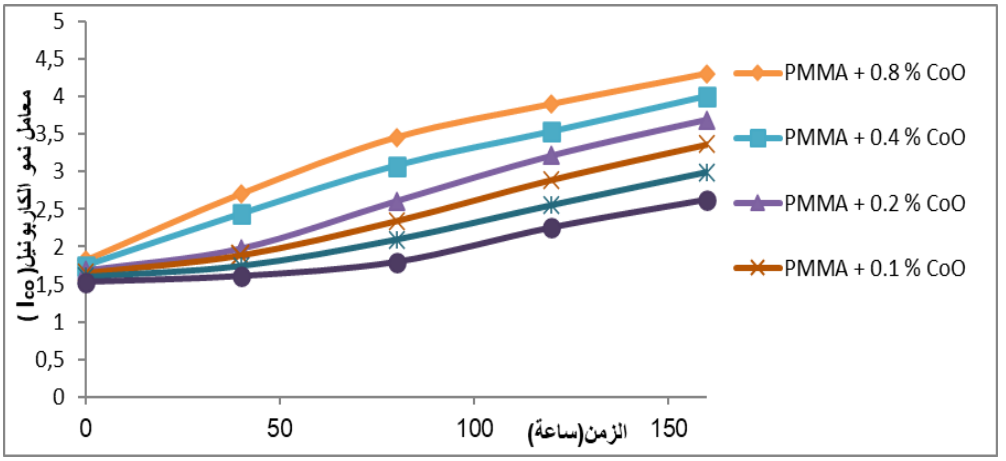


Fig. 11 : Relationship between Carbonyl group absorption coefficient and irradiation time for results listed in Table 4.

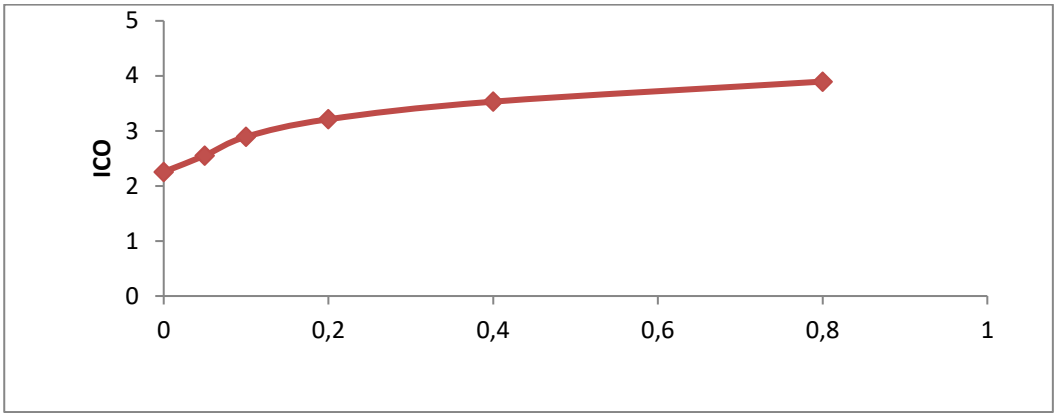


Fig. 12 : Change in CO group absorption coefficient with 120 hour PMMA chip nanomaterial concentration

Viscosity measurement

The polymer goes through a number of sequential events when exposed to ultraviolet UV light, including interlocking, secondary oxidation reactions, and polymer chain cutting, all of which cause cracked bonds, or cutting the polymer chain[17]. Polymer chips' visual molecular weight ratio was calculated prior to and after irradiation from the study of the photosynthesis process of pure (methylmetha acrylate) films containing different concentrations of cobalt nanoxide. Tables 5 and 6 showed poly PMMA films (methylmetha acrylate). The pure is the highest partial mass of films with a concentration of 0.05% of cobalt nanoxide and the reason for this is that the presence of polymer-added cobalt nanoxide CoO NPs accelerated the rates of visual molecular weight decline compared to pure polymer films as shown in the Fig. 13. (a) The numerical rate values for polymeric chain cutting (S) increase in conjunction with irradiation as in Figs. 14 and 15, respectively. This confirms that there are weak bonds distributed randomly along the polymer chain.

Table 5 : Calculated values from Visual molecular weight measurements for Pu PMMA chips

Time Irradiation (hrs.)	$\overline{(M_v)}x10^3$	$(M_v)^2x10^9$	$\frac{\frac{dM_v}{dt}}{\frac{M_{v0}-M_{vt}}{t}}$	Degree of Polymerization P	$\frac{1}{P}x10^{-4}$	Deg. Degree $a \quad x \quad 10^{-4}$	Ava. Chain Scission (S)
0	181.256	32.853	∞	1812.560	5.517	0.0	0.0
40	143.376	20.556	0.263	1433.760	6.974	1.457	0.207
80	110.006	12.101	0.247	1100.060	9.090	3.573	0.484
120	91.907	9.446	0.206	919.070	10.880	5.363	0.806
160	68.118	6.640	0.196	681.180	14.680	9.163	1.290

Table 6 : Measurements of Visual Molecular weight of PMMA chips containing 0.05% of nanomaterial (CoO).

Time Irradiation (hrs.)	$\overline{(M_v)}x10^3$	$(M_v)^2x10^9$	$\frac{\frac{dM_v}{dt}}{\frac{M_{v0}-M_{vt}}{t}}$	Degree of Polymerization P	$\frac{1}{P}x10^{-4}$	Deg. Degree $a \quad x \; 10^{-4}$	Ava. Chain Scission (S)
0	181.256	32.853	∞	1812.560	5.517	0.0	0.0
40	124.082	15.396	0.397	1240.820	8.059	2.542	0.460
80	89.980	8.096	0.316	899.800	11.113	5.596	1.014
120	76.771	5.893	0.241	767.710	13.025	7.508	1.360
160	57.201	3.271	0.215	572.010	17.482	11.965	2.168

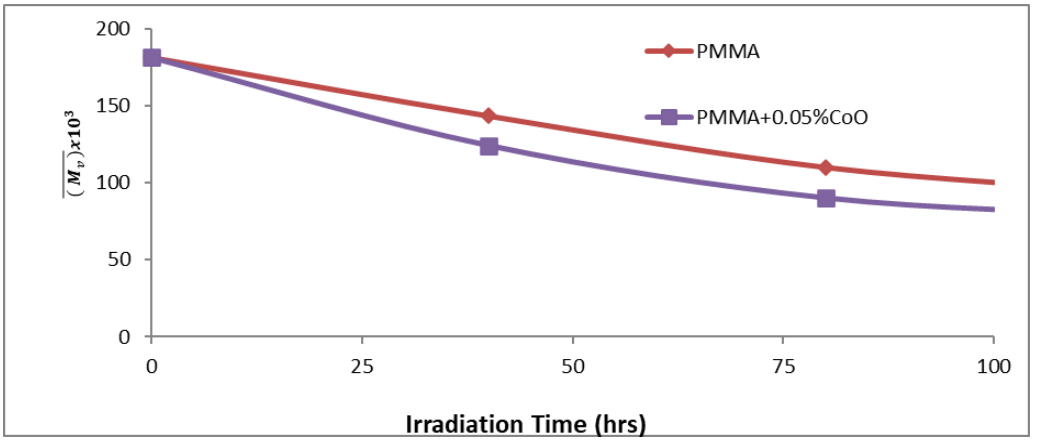


Fig. 13 : Relationship of visual molecular weight rate to irradiation time of PMMA chips with presence and absence of 0.05% concentration of CoO nanomaterials.

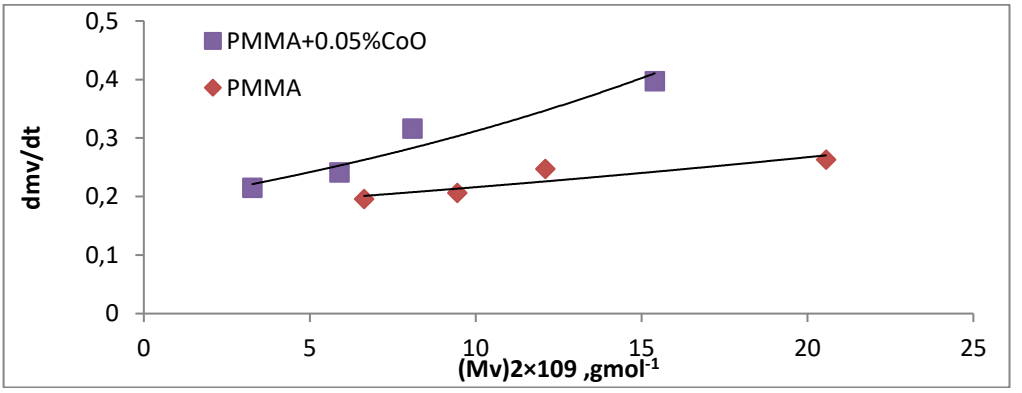


Fig. 14 : Fractional degree relationship with irradiation time of PMMA chips with and no concentration of 0.05% of CoO nanomaterials. NPs

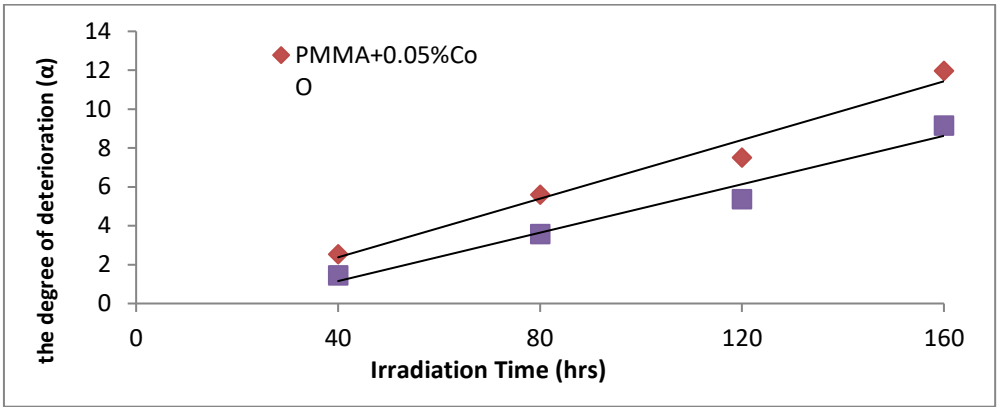


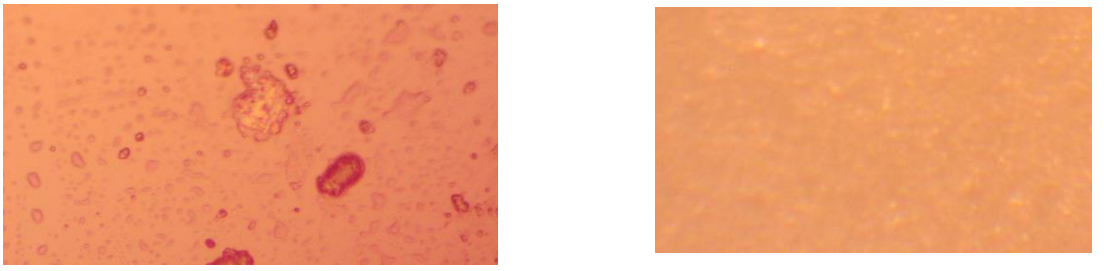
Fig. 15 : Chain cutting numerical rate relationship with irradiation time of PMMA chips with no concentration of 0. 05% of CoO nanomaterials. NPs

PMMA Optical Microscope

After adding cobalt nanoxide CoO NPs at a concentration of 0.05 percent, photographs of the pure methylmetha acrylate and additional photos of the same polymer were obtained. note that the brightness of polymeric chips has changed over time. UV light interacts with polymer films and additional nanoparticles to cause deformation and speed up optical fragmentation, as seen in Figs. 16 and 17.

(B) (A)

Fig. 16 : Pure PMMA (A) images before irradiation and (B) after irradiation for 120 hours.



(B) (A)

Fig. 17 : PMMA images containing 0.05% concentration of nanomaterial (A) prior to irradiation and (B) after irradiation for 120 hours.

Conclusion

Cobalt oxide nanoparticles CoO NPs with a size of (32.26) nm were identified by SEM examinations to have an irregular spherical shape and have a facecentered cubic shape according to the results of XRD tests. Additionally, it was discovered that the dissociation constant and the carbonyl coefficient rise when the viscosity molecular weight, carbonyl coefficient, and dissociation constant are tracked. While the molecular weight of the polymer falls as the irradiation period rises, it dissociates more quickly the more nanoparticle cobalt oxide is added to the polymer.

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