

PREPARATION AND IDENTIFICATION OF COPPER NANOXIDE USING ZIZIPHUS SPINA-CHRISTI EXTRACT AND STUDY OF ITS EFFECT ON THE PHOTO-PHYSICAL PROPERTIES OF POLY (METHYLMETHA ACRYLATE)

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ABSTRACT : Biodegradable polymers have received considerable attention because of their applications in the fields of environmental protection and physical health maintenance. Where the current research was involved, the optical fragmentation study of poly films (Methylmetha acrylate) was prepared by casting method after adding weight ratios of laboratory-prepared nanoparticle oxide from a plant (*Ziziphus spina-christi*) with weight ratios (0.0025, 0.005, 0.01, 0.02, and 0.04) And at (65 ± 5) micron before and after exposure to the UV source at wavelength. (356) nm and Bazman irradiation (0, 10, 20, 40, 80, and 120) One hour in a row and then follow up on the optical fragmentation process using the UV-visible spectrum device to calculate the dissociation velocity constant (Kd) and an infrared device to calculate the growth coefficient of the carbonyl and hydroxyl groups and viscosity technology to follow the change in the rate of viscous molecular weight. The measured results showed that the decomposition velocity constant was directly proportional to the added copper nanoxide concentration and irradiation time. The growths rate of the carbonyl and hydrologic ejectors of the nanomaterial was diagnosed using modern techniques (AFM, SEM, and XRD). The results showed that copper nanoxide was in the form of irregular spherical particles according to SEM images. The XRD study showed the synthetic properties of the copper nanoxide with a cubic face-centred composition, and the surface topography and minute volume of the nanoxide were studied with atomic force microscope (AFM) technology, which showed that the average minute volume was 20.31 nm.

Key words : Poly (methylmetha acrylate), copper nanoxide, optical fragmentation, viscosity.

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INTRODUCTION

Polymers are of great importance by entering many packaging, packaging, medical applications, adhesives, paint, some clothing, materials and engineering (Subramanian, 2015). The size of nanomaterials ranges from (1-100) nanometers, which exhibit new and unique characteristics that differ from the original components from which they were produced. These qualities make them subjects in a broad field of life and industry technology such as solar energy reactions, medical biology, catalysts and electronics. (Vigneshwaran *et al*, 2018). Copper nanoparticles form an insoluble powder in water (H₂O), dissolve in DMS, and slowly dissolve in ammonia solution (Singh *et al*, 2016). Copper nanoxide is of great importance during the research process for its ability to remove heavy metals from water contaminated with high efficiency and low cost and is of

great significance in advanced techniques and applied fields because it has visual and catalytic properties where its particles are highly thermal and electrical (Devi and Singh, 2014). Green synthesis of nanoparticles through the use of plant extracts is a new area of research. It is likely preferable to chemical or microbial synthesis because it eliminates the complex process of nanoparticle production and is capable of meeting large-scale production at good quality and low cost (Das *et al*, 2013).

A polymer (methylmetha acrylate) has good stability due to solid polymer chains, is resistant to acids and alkali, does not dissolve in most organic solvents, is transparent, colourless, has visual properties and is considered to be commercially available polymers (Nahida, 2012). PMMA dissolves dramatically in organic solvents such as chloroform (CHCl₃) and its resistance to many other chemicals are weak due to the presence of the easily

decomposing ester group. Multiple (Methylmethacrylate) is more stable in the environment than other plastics such as polyethylene and polystyrene, so it is preferable to other polymers in external uses (William, 2006). Poly (methylmethacrylate) are widely used in household appliances, dentures and laser disk manufacturing. PMMA is durable, transparent, corrosion-resistant and UV and has strong weather resistance (Lee and Jang, 1996). Stabilization and fragmentation is a multi-purpose process by which the properties of polymers can be modified or altered. Environmental risks from the degradation of polymers can be overcome (Rivaton and Gardette, 1999). The present study, therefore, aims to prepare a copper oxide nanomaterial and to use the nanomaterial as a catalyst for the photodegradation process of the polymer (methylmethacrylate), the study of the dissociation mechanism through the use of infrared and ultraviolet-visible techniques and study of the rate of change in molecular weight through the use of viscosity scale.

MATERIALS AND METHODS

Materials and devices used

Aqueous copper chloride salt ($\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$), sodium hydroxide NaOH, poly (methylmethacrylate) PMMA, chloroform solvent, distilled water, dimethyl sulfoxide solvent, ethanol solvent, and dislocated water Atomic force microscope, X-ray diffraction, scanning electron microscope, UV spectroscopy, infrared spectroscopy, Ostwald viscometer and centrifuge were used.

Preparation of plant extract

We prepare the *Ziziphus spina-christi* tree by collecting samples of its leaves and washing its leaves. (Three times) with distilled water, dried with shade and crushed with mortar for a powder to be sifted to get rid of objects stuck to it and keep the powder in the shade, weighed 5gm of powder and put it in a glass baker add a mechanism (200mL) of ion-free water with continuous stirring by magnetic motor and heating at temperature 80°C for 30 minutes to obtain the brown-coloured extract. Then leave to cool at room temperature and filter with filter sheets for a solution to be placed in test tubes in the centrifuge at speed (1200) cycle/minute to dispose of the remaining fibre and then store the solution in a glass bottle at low temperature (Sally, 2018).

Preparation of copper nanoxide

It weighs 0.30gm of Aqueous copper chloride salt ($\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$). It is placed in a glass baker and added to a 300 mL mechanism of ion-free water with continuous stirring by the magnetic motor until completely dissolved.

And then, add 20 ml of the plant extract to the surface in the form of droplets gradually with constant stirring at 20°C , after which the temperature of the solution is raised to 80°C to adjust acid function by adding sodium hydroxide (NaOH) in concentration (1) Molarity where a coloured (green-brown) sediment is formed to cool the sediment to room temperature. Then filter the sediment with filter sheets and wash (three times) with ion-free water and absolute ethanol for impurity disposal and oven-dried at 60°C for 2 hrs (Sally, 2018).

Poly purification (methylmethacrylate) : Re-deposition mediated by dissolving 15gm from poly (methylmethacrylate) in 100 mL chloroform solvent with continuous stirring by magnetic motor to ensure homogeneity for one hour. Then the homogeneous solution is added to 250 mL of ethanol alcohol with constant stirring until the polymer is deposited (Marwa, 2018).

Preparation of a poly solution (methylmethacrylate) : For the preparation of poly (methylmethacrylate), 7gm of it is dissolved in 100 mL of the chloroform solvent and then blended continuously by using the magnetic motor at room temperature until fully homogeneous for one hrs (Marwa, 2018).

Polymeric Film preparation

Polymeric films for PMMA have been prepared according to the following steps:

1. Add copper nanoxide prepared in the following weight ratios (0.00, 0.0025, 0.005, 0.01, 0.02, and 0.04)% to (5 mL) PMMA solution and then place the mixture in glass molds made from sliced glass.
2. Perform homogeneity between solution and oxide and ensure that bubbles are not present on polymeric foil and then leave the mixture for 24 hrs at room temperature (RT) until the mixture dries up without being exposed to sunlight.
3. Polymeric films are removed 24 hrs later by blade and forceps.
4. We use the Micrometer to adjust the thickness of polymer films at (60 ± 5) micrometers.
5. Polymeric films are chopped at the following dimensions at (3.5 cm) and viewed (1.5 cm) for dimensions proportional to spectral measurement cells (UV-Visible, FT-IR).

Follow-up the optical fragmentation for poly (methylmethacrylate)

Infrared spectroscopy : We use (FT-IR) to track the growth rate of carbonyl group (I_{CO}) and hydroxide group (I_{OH}) by measuring the infrared spectrum to

determine the intensity of the beams absorbed within the range (400-4000 cm^{-1}) prior to irradiation and after each period of irradiation of pure PMMA chips containing different concentrations of copper nanoxide, where the carbonyl group absorption site appeared (C=O) at the range (1735) cm^{-1} and the hydroxide group (OH) position appeared at the range (3700-3300) cm^{-1} through which the growth coefficients of carbonyl (I_{CO}) and hydroxide were found (I_{OH}) by applying equation (1) by adopting the package coefficient (Chandra and Handa, 1982) and by selecting location (1435 cm^{-1}) as the reference point for PMMA.

$$I_{(S)} = \frac{A_{(S)}}{A_{(R)}} \quad (1)$$

Where, $I_{(S)}$ represents: Group growth coefficient under study, $A_{(S)}$: Absorption of group under study $A_{(R)}$: Reference summit absorption.

Ultraviolet-visual spectroscopy : We use uv-vis radiation to calculate the absorption value of polymeric films within the range of 200-800 nm. The speed of photosynthesis (K_d) by using the following equation

$$\ln \ln (A_{\infty} - A_t) = \ln \ln (A_{\infty} - A_0) - K_d t \quad (2)$$

A_0 represents the pre-irradiation of polymer chips, At the absorption of polymer chips themselves at the time of irradiation (t) and A_{∞} the absorption of polymeric chips at the end of time. From drawing the relationship between $\ln (A_{\infty} - A_t)$ with irradiation time (t), the slope was found to represent the dissociation constant ($-K_d$).

Viscosity measurement : The M_v viscous molecular weight rate of polymer chips is calculated by calculating the viscosity rate by applying the (Mark- Houwink) equation (Wang *et al*, 2016).

$$[\eta] = K(M_v)^a \quad (3)$$

The molecular weight of a poly (methylmetha acrylate) was calculated by the presence and absence of the additive through intrinsic viscosity measurements in the chloroform solvent at 20 $^{\circ}\text{C}$ using the equation (4) (Hammed, 2019).

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

The degree of fragmentation (α) and the numerical rate of the series cut (S) were calculated according to the following equations (Hammed, 2019).

$$\alpha = \frac{1}{P_t} - \frac{1}{P_0} \quad (5)$$

$$S = \alpha p_0 \quad (6)$$

P_t : Numerical rate of polymerization at time (t) of irradiation, P_0 : numerical rate of degree of polymerization before irradiation

Microscopy

We use a Novel microscope to be able to track the effects of UV irradiation on the surface properties of polymer chips. Pure PMMA and polymeric films containing copper nanoxide were taken before irradiation and after irradiation.

RESULTS AND DISCUSSION

Atomic force microscope (AFM)

It gives information on the topography of the surface of the material with high accuracy up to the atomic level as atomic force microscopy probes scan the topography of the material with very small precision. It gives information about the surface texture and the size of the minutes, showing a Fig. 1 two-dimensional and three-dimensional images of the prepared copper nanoxide surface, where the volume of the prepared minutes is about 20.31 nm in diameter.

Scanning electron microscope (SEM)

It is an imaging technique that shines a beam of electrons into the area to be studied so that electrons interact with surface atoms generating three types of rays: secondary electrons, back-scattered electrons and X-rays. The magnification range reaches 25-250.000 thousand times. To determine the surface morphology and particle shape of the prepared nanoscale (Maruthupandy *et al*, 2017) as shown in (2) form. Scanning electron microscopy images clearly show nanoparticles with irregular spherical particles forming and the average particle size is around 17.31-19.49 nm.

X-ray diffraction (XRD)

It's used to get peaks and see where the peaks appear when these rays are shone at different angles and shapes. (3) demonstrates the X-ray diffraction patterns of the

Table 1 : Structural coefficients of prepared copper nanoxide obtained from XRD X-ray diffraction analysis.

2 θ (degree)	
HK1	Observed
110	31.22 $^{\circ}$
002	33.18 $^{\circ}$
111	35.96 $^{\circ}$
202	41.16 $^{\circ}$
020	49.81 $^{\circ}$
202	50.23 $^{\circ}$
113	54.37 $^{\circ}$
311	63.59 $^{\circ}$
0004	76.10 $^{\circ}$

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

Irradiation time (hour) Type of chips	Absorption A_t				
	0.0	20	40	80	120
PMMA	0.145	0.202	0.291	0.363	0.466
PMMA + 0.0025 % CuO	0.181	0.252	0.334	0.444	0.515
PMMA + 0.005 % CuO	0.185	0.308	0.381	0.570	0.697
PMMA + 0.01 % CuO	0.186	0.377	0.493	0.682	0.775
PMMA + 0.02 % CuO	0.187	0.428	0.577	0.783	0.856
PMMA + 0.04 % CuO	0.189	0.520	0.697	0.938	1.090

Table 3 : K_d Breakup Velocity constants values for PMMA films containin CuO nanomaterials.

Disintegration velocity constant K_d (Sec.) ⁻¹ × 10 ⁻⁵	% concentration
0.175	PMMA + 0.0025% CuO
0.222	PMMA + 0.005% CuO
0.251	PMMA + 0.01% CuO
0.272	PMMA + 0.02% CuO
0.313	PMMA + 0.04% CuO

with the ICBD card (JCPDS) found to be identical, and also consistent with literature results (Suleiman *et al*, 2013).

Ultraviolet-visible spectroscopy (UV-VIS)

We note that A peak of 278 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

Table 4 : I_{OH} values with PMMA irradiation time containing differe concentrations of material (CuO) nanoscale.

Irradiation time (hour) Percentage %	I_{OH}				
	0.0	20	40	80	120
PMMA	0.00	0.104	0.141	0.242	0.313
PMMA + 0.0025 % CuO	0.000	0.154	0.224	0.353	0.41
PMMA + 0.005% CuO	0.000	0.176	0.257	0.42	0.51
PMMA + 0.01 % CuO	0.000	0.201	0.308	0.511	0.602
PMMA + 0.02 % CuO	0.000	0.272	0.408	0.601	0.67
PMMA + 0.04% CuO	0.000	0.335	0.468	0.654	0.754

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

Time Irradiation (hrs.)	$(M_v) \times 10^3$	Degree of Polymerization P	$\frac{1}{P} \times 10^{-4}$	Deg. Degree $\alpha \times 10^{-4}$	Ava. Chain Scission (S)
0	225.334	2253.340	4.437	0.0	0.0
10	186.588	1865.880	5.359	0.922	0.207
20	151.778	1517.780	6.588	2.151	0.484
40	124.731	1247.310	8.017	3.580	0.806
80	98.397	983.970	10.162	5.725	1.290

prepared copper nanoxide compared to its standard reference NO.(48-1548) JCPDS The diffraction peaks shall be as follows (110), (002), (111), (202), (202), (113), (311), (004) and shall be attributed at the angles (31.22°, 33.18°, 35.96°, 41.16°, 49.81°, 50.23°, 54.37°, 63.59°, 76.1°), respectively, as shown in Table 1. These indicators and data indicate and confirm that they are prepared copper nanoxide powder with a cubic face-centered composition by standard reference and when matched

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 120 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 CuO nanoscale

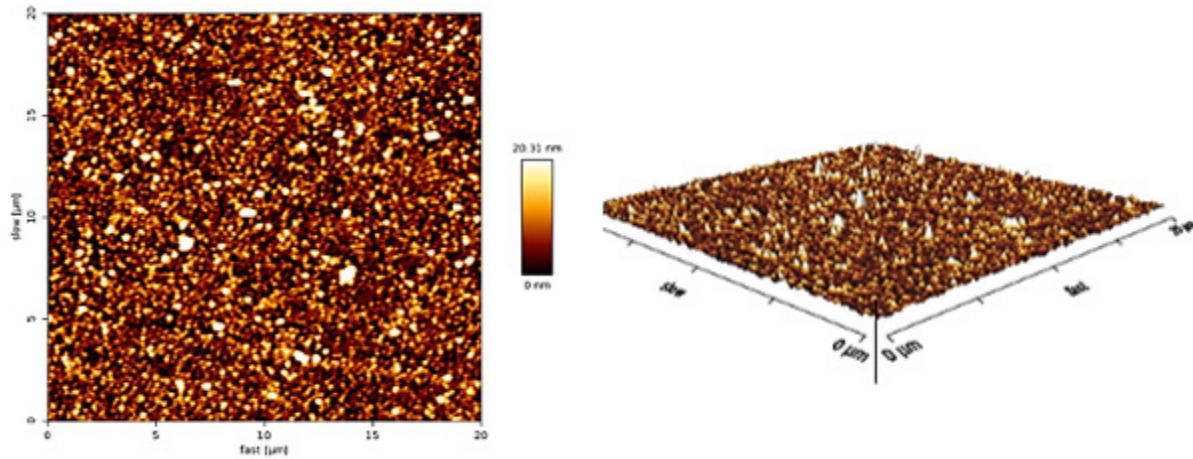


Fig. 1 : 2D and 3D AFM image of nanoparticle oxide tested in green synthesis method.

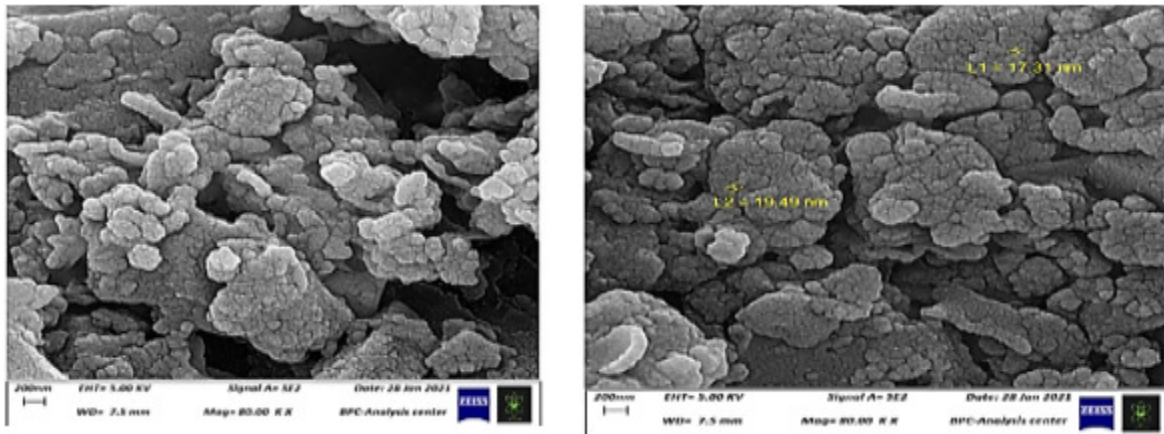


Fig. 2 : Images showing copper nanoxide prepared by scanning electron microscope SEM.

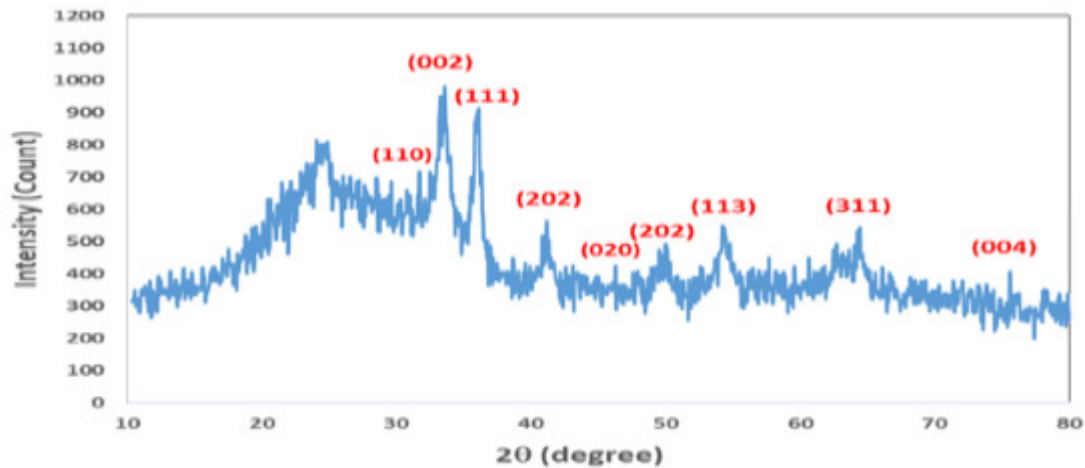


Fig. 3 : CuO nanoxide XRD compared to standard reference NO.(48-1548) JCPDS.

where absorption increases with increased concentration of added nanomaterial and for the same irradiation time. (65 ± 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 120 hours (Yousif *et al*, 2015) as in (4) and (5) forms. And by the relationship between $\ln \ln (A_{\infty} - A_0)$ 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 copper nanoxide is a first-order reaction (Adil *et al*, 2016). By calculating the slop slope, the K_d photosynthetic decomposition velocity constant was measured for multiple added copper nanoxide (methylmetha acrylate) and according to the stated concentrations (Dina *et al*, 2018). 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.

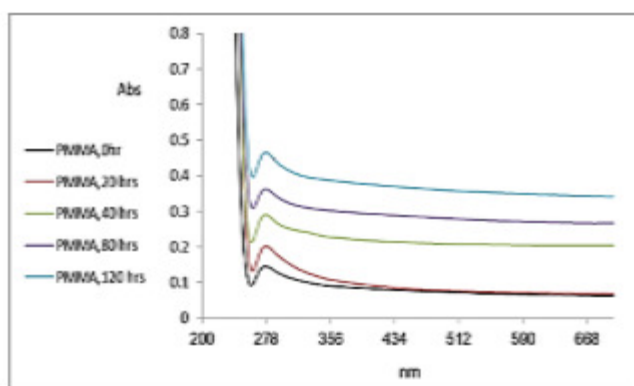


Fig. 4 : Change in radiation spectrum above Purple-visual for PMMA movies free of additives thick (65 ± 5) Micron at different irradiation times.

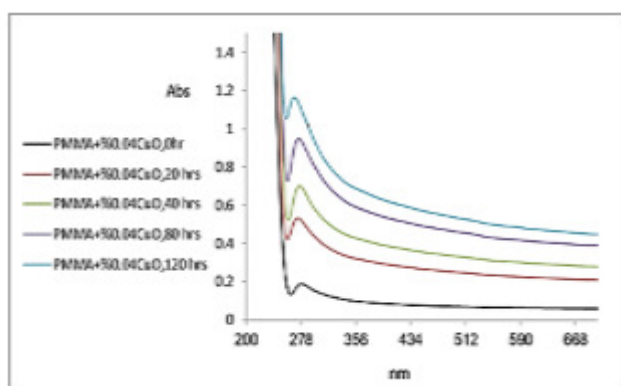


Fig. 5 : Change in UV-Vis spectrum containing concentration of 0.04% of nanomaterials thick (65 ± 5) micron at different irradiation times.

Infrared spectroscopy

The optical fragmentation of a poly methylmethacrylate is followed by the follow-up of carbonyl group absorption packages ($C=O$) and hydroxyl (OH) 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}) (Anju and Narayanankutty, 2017) which showed a spectral change compared to the second

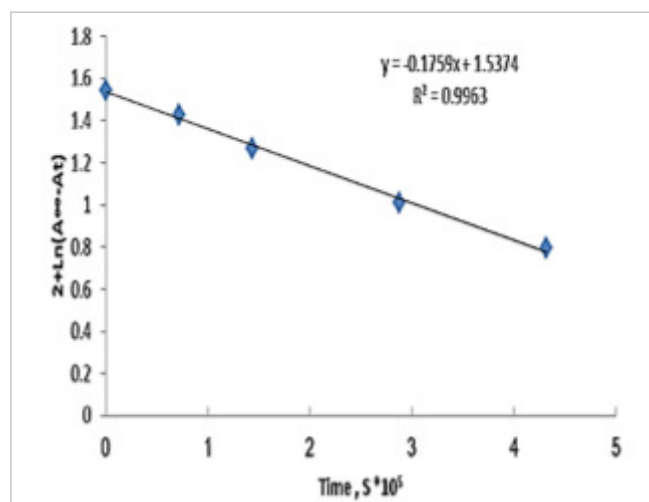


Fig. 6 : Relationship between natural logarithm to absorb copper oxide (CuO) 0.0025% concentration in 0.04% movies thickness (65 ± 5) micron with irradiation time.

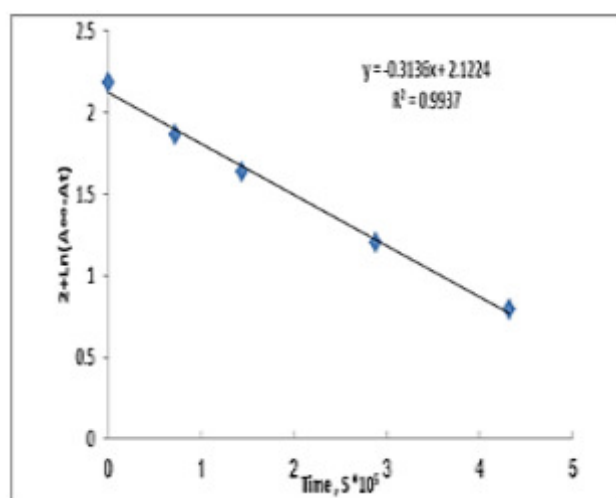


Fig. 7 : Relationship between natural logarithm to absorb copper oxide (CuO) at a concentration of 0.04% thickness (65 ± 5) micron with irradiation time.

Table 6 : Measurements of Visual Molecular weight of PMMA chips containing 0.02% of nanomaterial (CuO).

Time Irradiation (hrs.)	$(M_v) \times 10^3$	Degree of Polymerization P	$\frac{1}{P} \times 10^{-4}$	Deg. Degree $\alpha \times 10^{-4}$	Ava. Chain Scission (S)
0	225.334	2253.340	4.437	0.0	0.0
10	173.757	1737.570	5.755	1.318	0.296
20	129.202	1292.020	7.739	3.302	0.744
40	103.367	1033.670	9.674	5.237	1.180
80	80.505	805.050	12.421	7.984	1.799

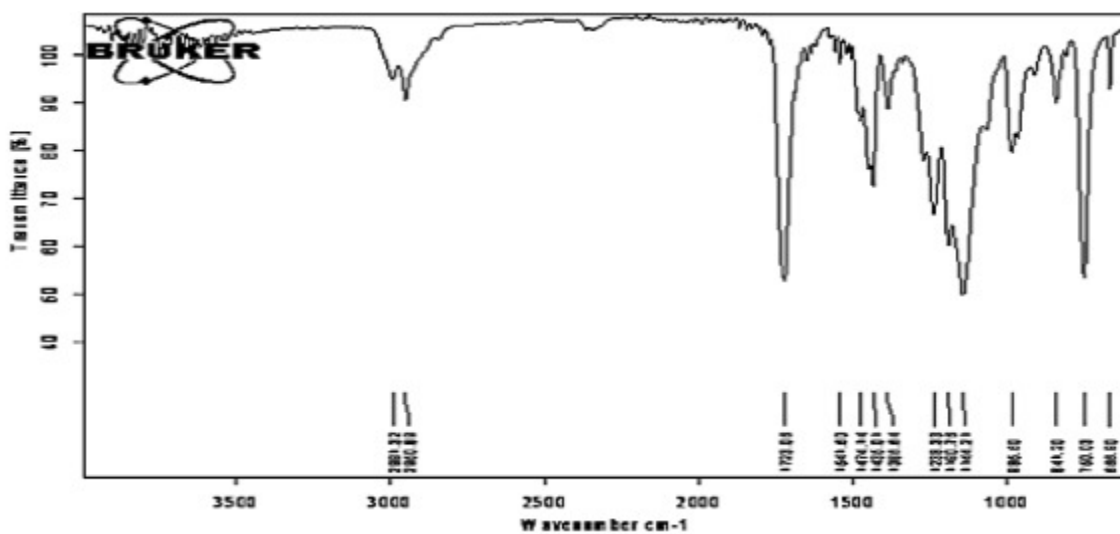


Fig. 8 : Infrared spectrum of additive-free multi-PMMA chips before irradiation.

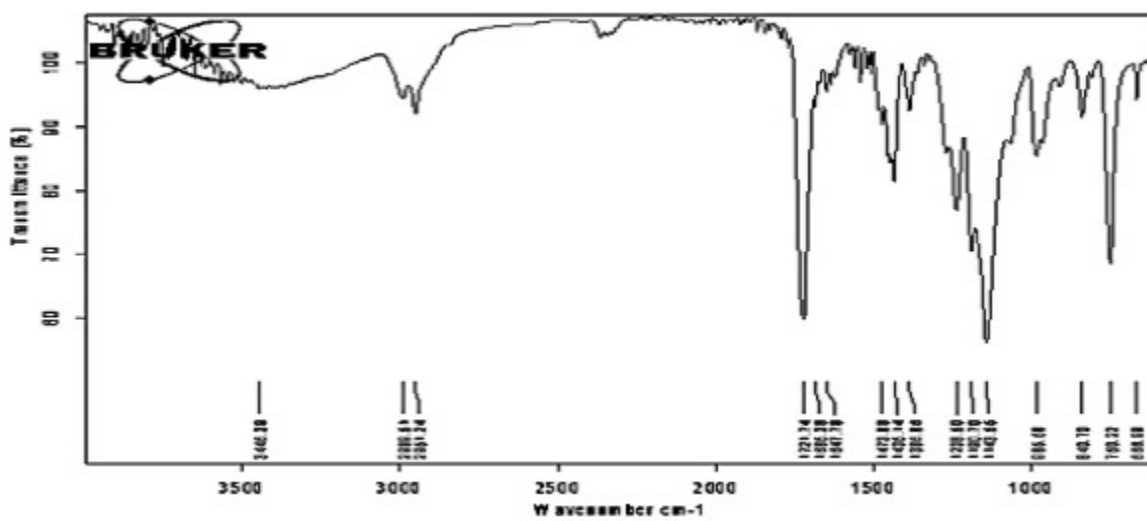


Fig. 9 : Infrared spectrum of additive-free multi-PMMA chips after 120-hour irradiation.

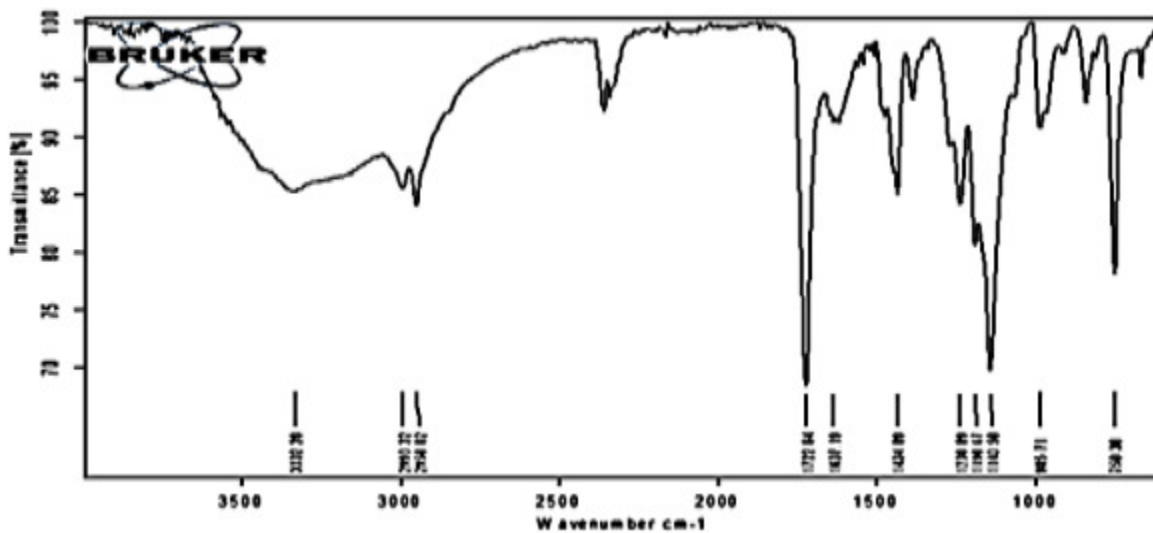


Fig. 10 : Infrared spectrum of multiple PMMA chips containing nanomaterial (CuO) at concentration (0.02%) and irradiation time (120) hours.

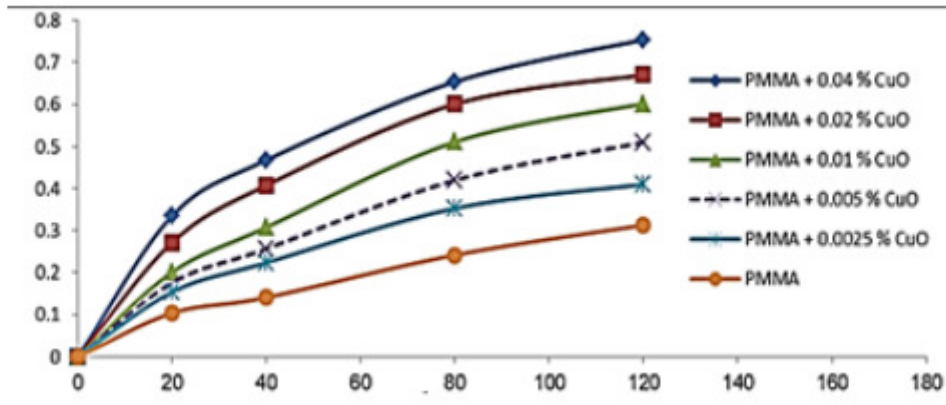


Fig. 11 : Relationship between hydroxyl group absorption coefficient and irradiation time for results listed in Table 4.

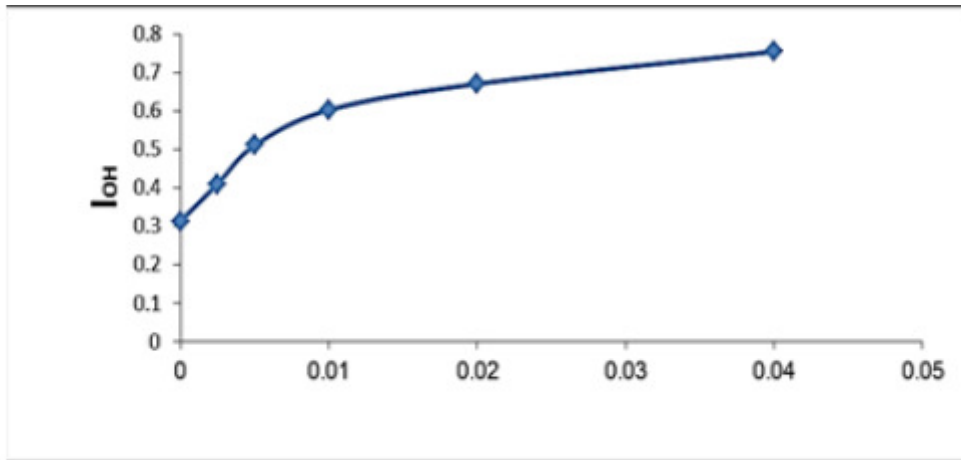


Fig. 12 : Change in OH group absorption coefficient with 120 hour PMMA chip nanomaterial concentration.

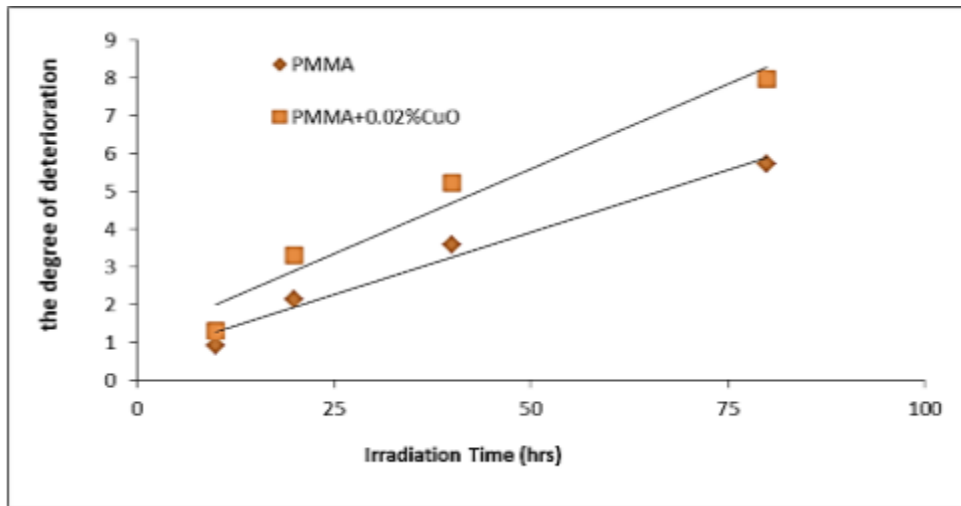


Fig. 13 : Relationship of visual molecular weight rate to irradiation time of PMMA chips with presence and absence of 0.02% concentration of CuO nanomaterials.

beam, the hydroxyl group (OH), which appears at the (3300-3700) cm^{-1} (Pipatpanukul *et al*, 2018) range where 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 PMMI 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. Table 4 notes that the proportionality is direct between the values of the hydroxyl group absorption

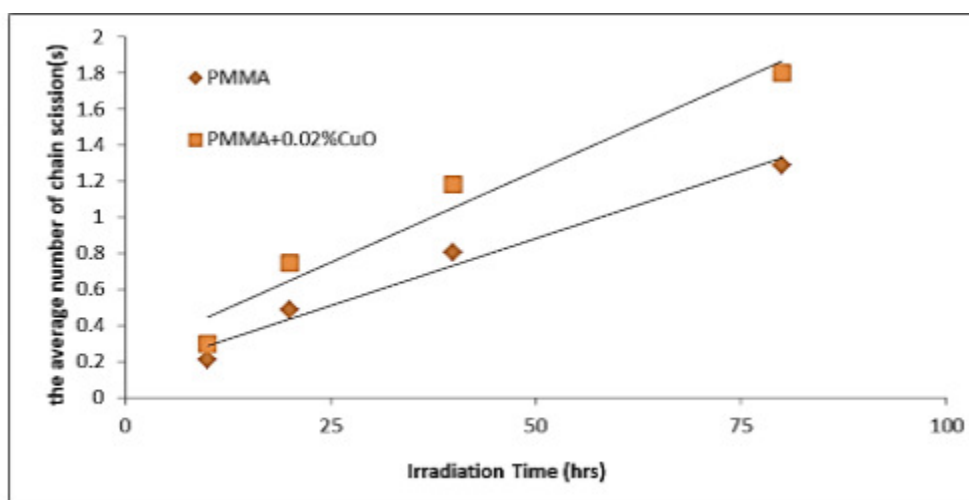


Fig. 14 : Fractional degree relationship with irradiation time of PMMA chips with and no concentration of 0.02% of CuO nanomaterials.

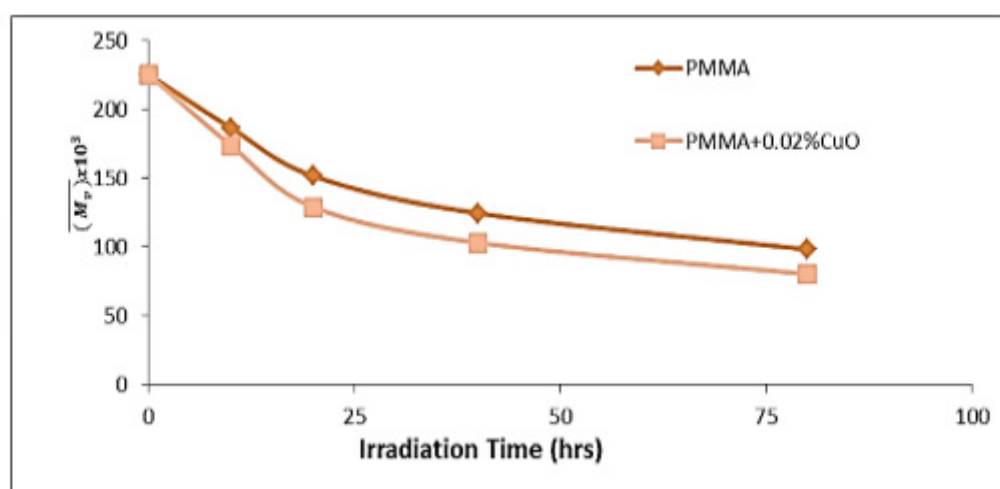


Fig. 15 : Chain cutting numerical rate relationship with irradiation time of PMMA chips with no concentration of 0.02% of CuO nanomaterials.

coefficient (I_{OH}) 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 copper 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_{OH}) This increases the optical oxidation speed of polymer films.

Viscosity measurement

The polymer, when exposed to uv radiation, has several sequential processes, including interlocking, secondary oxidation reactions and polymer chain cutting, which result in fractured bonds, *i.e.* cutting the polymer chain (Banik *et al*, 2018). 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 copper nanoxide. Tables 5 and 6 showed poly films (methylmetha acrylate). The pure is the highest partial mass of films with a concentration of 0.02% of copper nanoxide and the reason for this is that the presence of polymer-added copper nanoxide accelerated the rates of visual molecular weight decline compared to pure polymer films as shown in the Fig. 13. (α) 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.

PMMA Optical Microscope

Photographs of the pure methylmetha acrylate and other images of the same polymer were taken after the addition of copper nanoxide at a concentration of 0.02%. Note that over time there has been a distortion in the brightness of polymeric chips. Deformation occurs as a

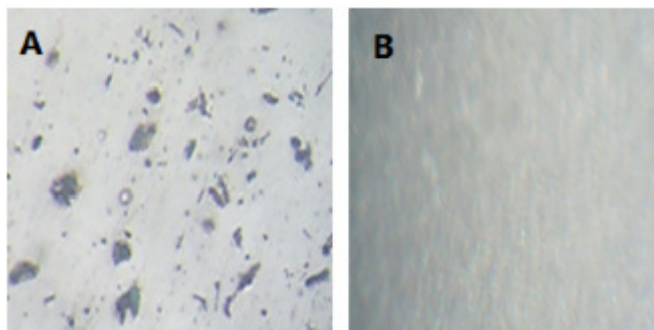


Fig. 16 : Pure PMMA (A) images before irradiation and (B) after irradiation for 120 hours.

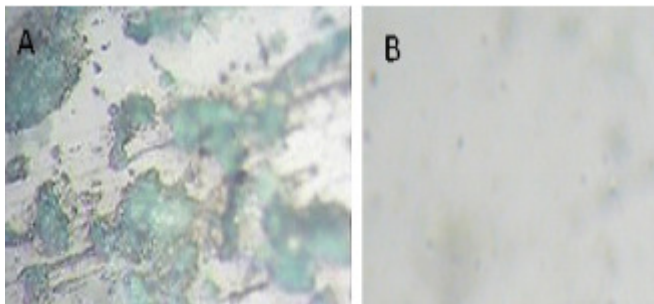


Fig. 17 : PMMA images containing 0.02% concentration of nanomaterial (A) prior to irradiation and (B) after irradiation for 120 hours.

result of the interaction of uv light with polymer films and added nanomaterials, which accelerate the process of optical fragmentation as in Figs. 16 and 17.

CONCLUSION

The results obtained from XRD measurements showed that the copper oxide nanoparticles have a face-centered cubic shape and are in the form of irregular spherical particles through SEM measurements that refer to the CuO nanoparticles with a size of (19.49) nm. And by following the rate of change in the viscosity molecular weight, the carbonyl coefficient and the dissociation constant, it was found that the dissociation constant and the carbonyl coefficient increase. While the molecular weight decreases as the irradiation time increases and the higher the concentration of nanoparticle copper oxide added to the polymer, the greater the dissociation speed of the polymer.

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