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Preparation of Boehmite Nanoparticles and Determination of Their Effect on Photo Stabilization of Polycarbonate

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Summary

In this study, boehmite nanoparticles were prepared using aluminum waste extract and their effect on the photostability of polycarbonate films with a thickness of (5 ± 50) microns after adding different concentrations of the nanoadjuvant (0.0025, 0.005, 0.01, 0.02, 0.04) % to a polycarbonate solution dissolved in chloroform. The boehmite nanoparticles were characterized using various techniques such as EDX, XRD, SEM, FT-IR and TEM. A band at $3200-3500\text{ cm}^{-1}$ belonging to the OH group was identified according to FT-IR analysis, indicating the formation of boehmite nanoparticles. The photodegradation of polycarbonate with and without the additive was monitored using UV-visible analysis at a wavelength of 356 nm during different irradiation times (160,120,80,40,20,10,0) hours and the rate constant of photocatalytic fragmentation of polycarbonate was calculated. The results showed a decrease in the photodegradation of the polymer films with an increase in the nanomaterial concentration compared to the films without the additive, Using FT-IR analysis, the carbonyl absorption coefficient was calculated and found to decrease with the concentration of added nanobohmite, consistent with the K_d values (photodissociation rate constant). In addition, the average viscous molecular weight of the polymer, the photodissociation degree, and the numerical rate of cutting (S) of the polymer chain were calculated using a viscometer. It was observed that the average molecular weight of the polymer decreases with increasing irradiation time to a lesser extent in the presence of the nanomaterial additive

Keywords: polycarbonate, photodegradation, photostability, boehmite, polymers

1- Introduction

Polymers consist of long molecular chains composed of the linking of a large number of small molecules known as monomers. When these monomers are joined together via chemical bonds, polymers are formed that are characterized by the repetition of this small unit⁽¹⁾ Polymers are used in a variety of applications such as adhesives, coatings, some types of clothing, and highly specialized structural and engineering materials. Polymers are increasingly being requested for use in electronic and optical applications due to their unique capabilities in these areas⁽²⁾. Polymers can be rigid or flexible, and are used in construction, automotive, aircraft, electrical appliances, electrical insulation, and thermal barriers due to their ability to provide thermal and electrical insulation⁽³⁾. The effect of UV radiation on polymers can lead to significant degradation of many properties. Photodegradation (oxidation) occurs through UV light, breaking polymer chains, forming free radicals and reducing molecular weight, ultimately leading to degradation of mechanical properties and formation

of undesirable substances. This process of UV-induced degradation of polymers is known as photodegradation.⁽⁴⁻⁸⁾ There are byproducts of the photolysis process that occur naturally during the formation of nanoparticles. Attention must be paid to the effects this reaction can cause when used in the production of polycarbonate.⁽⁹⁾ Nanotechnology has enormous potential in various sectors, including agriculture. This technology involves controlling materials at the level of individual atoms or molecules. In agriculture, nanotechnology can be used to improve crop production, plant protection, and development at the nanometer level⁽¹⁰⁾.

The green synthesis of nanoparticles has seen increasing interest due to its many positive aspects. The method is sustainable and environmentally friendly, involving low energy consumption and little production of harmful waste. It also meets the requirements of the Sustainable Development Goals, reflecting our commitment to protect the environment and support sustainability⁽¹¹⁾.

There are many previous studies conducted by researchers, including

He managed (Muhammad Basil Anwar)⁽¹²⁾ and others Improving the optical stability and UV-blocking ability of polyvinyl chloride by incorporating nanoparticles, PVC is a staple material in various industries due to its low cost and durability, but it deteriorates significantly when exposed to ultraviolet radiation. Protective agents using nanoparticles have been developed to protect PVC and enhance its UV-blocking ability.

The researcher rose ⁽¹³⁾ (Grigoriadou) and others From the analysis of the mechanical properties, the stability of both tensile strength and elongation at break, it can be concluded that the nanoparticles in the HDPE/multi-walled carbon nanotube composites act as UV stabilizers. In addition, the nanoparticles in all the studied samples showed a stabilizing effect on HDPE, especially the nanocomposites containing silicon dioxide (SiO₂) and MMT.

Masoud Mohammadi⁽¹⁴⁾ and others have synthesized boehmite nanoparticles using sol-gel, precipitation, drainage, and hydrothermal methods. Hydrothermal is considered the most suitable method for producing various forms of boehmite. The properties and shapes of boehmite nanoparticles depend largely on process conditions such as pH, temperature, and reaction time. The use of boehmite as a catalyst or catalyst support has been studied.

This study aims to explore the use of chemicals to improve the physical and optical properties of polymer, by adding substances that help prevent degradation of polymeric materials due to exposure to ultraviolet light, to improve durability and resistance to environmental factors for use in outdoor applications such as tanks, pipes and plastic structures, with the aim of minimizing the environmental degradation resulting from the use of polymeric materials

2- Materials and Methods

Aluminum waste was prepared by cutting it into small pieces and washing it with sulfuric acid to remove dyes and impurities. They were exposed to heat to increase the cleaning efficiency, then washed with running water and then distilled water. It was then left to air dry.

3- Preparation of boehmite nanoparticles

(40grams) of aluminum cans were weighed and placed in a sodium hydroxide solution with a concentration of (2) molar, then the mixture was stirred until all aluminum pieces were completely dissolved to form a sodium aluminate solution. The solution was then filtered to remove any impurities such as plastic paint. Hydrogen peroxide (30%) was added slowly and continuously using a magnetic stirrer. The pH value was adjusted to 7 using concentrated hydrochloric acid to form a white precipitate of boehmite. The precipitate was then filtered and washed several times with distilled water to remove sodium chloride salts, and then dried at 100°C for 24 hours

4- Preparation of polycarbonate solution

The polycarbonate solution was prepared by dissolving (10 g) of polycarbonate in (100 ml) of chloroform, with continuous stirring using a magnetic stirrer. This was carried out at room temperature (25°C) for (45) minutes, resulting in a pure polycarbonate solution.

5- Preparation of the polymeric film

The samples were prepared by converting polycarbonate into flakes by dissolving in chloroform. These samples were catalyzed by adding boehmite nanoparticles. A thickness of $5\pm 50\ \mu\text{m}$ was used to cast them into a glass mold and then allowed to dry and carefully lifted with a blade, after which it was measured using a micrometer (USA Tester.UQ V Q, Inc). In the next step, the samples were cut into slices with dimensions (1.5 x 3.5 cm) to complete the required measurements.

6- Samples

The samples were irradiated using an 18W UV light source with a wavelength of 356 nm. The polymer films were positioned in such a way that they were exposed to the same level of radiation during the specified irradiation period

Monitoring the photostability of polycarbonate

The following methods were used to stabilize the polymer film: UV-Vis method, FT-IR method, and viscometry

7- Results and Discussion

Structural characterization of boehmite nanoparticles (green method)

7.1 FT-IR spectra

The FTIR spectrum of boehmite nanoparticles shows the presence of a new chemical bond on their surface, which means that the application of aluminum waste extract has helped to stabilize and stabilize the boehmite nanoparticles

$3500\text{-}3200\text{cm}^{-1}$ is a prominent peak that appeared in the FT-IR spectrum and clearly shows the effect of aluminum waste extract on boehmite nanoparticles as in Figure 1

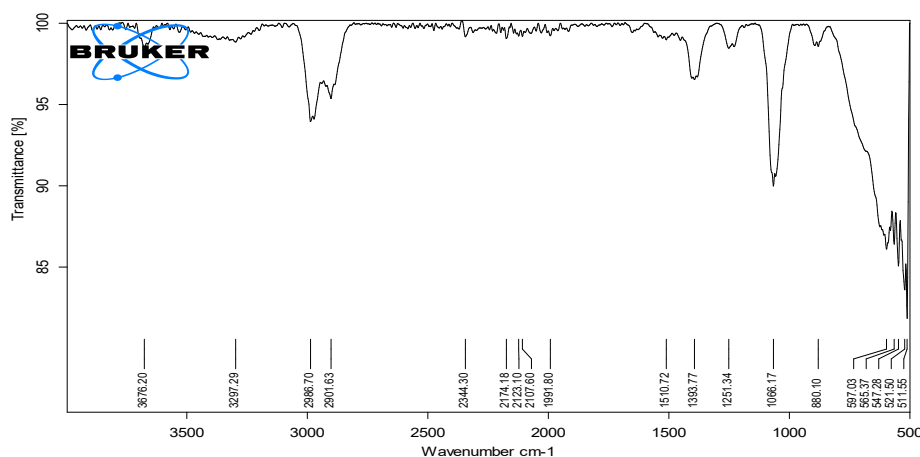


Figure 1: FT-IR analysis of boehmite nanoparticles

7.2 SEM measurements

SEM images of boehmite nanoparticles used in the recycling of aluminum waste show a precise and orderly arrangement. This contributes to a better understanding of the structure and composition of boehmite. The images show the nanoparticles synthesized by the specified method and how the boehmite crystals agglomerate. Clusters of boehmite crystals with a size range of 23.67-38.67 nm can be observed, showing organized crystallization of boehmite within the nanoparticles, as in Figure 2

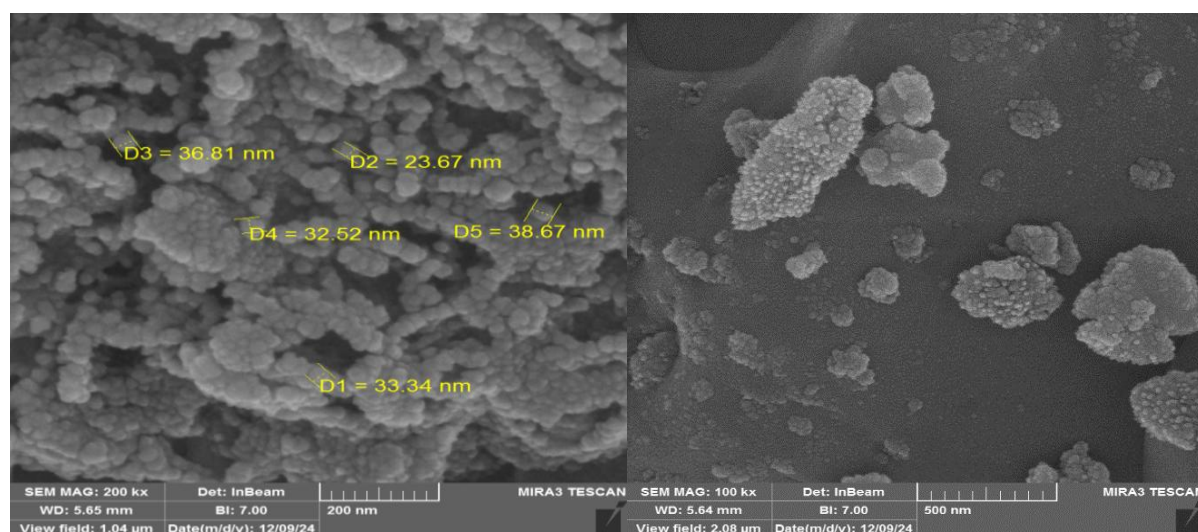
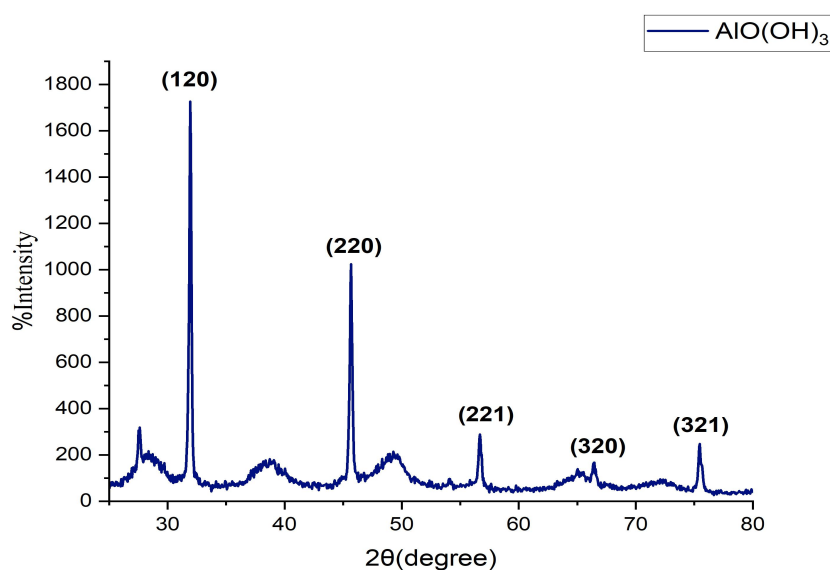


Figure 2: SEM images of boehmite nanoparticles in different contexts (200 nm, 500 nm)

7.3 XRD measurements

The XRD results of boehmite nanoparticles show that the diffraction peaks match the boehmite reference pattern (JCPDS 01475-072-01), where diffraction peaks such as (120), (220), (221), (203), (321) are associated with corresponding angles (31.93° , 45.66° , 56.67° , 66.42° , and 75.49°), respectively. These indices indicate the quality of the crystal arrangement of the prepared boehmite nanoparticles.



The average crystal size ($D = 27.25$) nm was calculated using the Scherrer equation ⁽¹⁵⁾. From the data shown in Table (1) below:

Table (1): The three strongest peaks of boehmite nanoparticles prepared by XRD measurement

Position (2θ deg.)	FWHM (deg.)	Intensity(count)
31.93259	0.24296	1727
45.66863	0.28285	1024
56.67779	0.28575	289

8- Study of the optical fractionation of polycarbonate

Optical fractionation of 5 ± 50 mm thick polymer films of pure polycarbonate consisting of different concentrations of boehmite nanoparticles. It was the boehmite nanoparticles that determined the effect of increasing the concentration of boehmite nanoparticles on the stability of the polymer by different UV irradiation times, FT-IR, UV-VIS and viscometry were applied as follows

8.1 VIS UV spectroscopy

The photostabilization of polymeric films is that which consists of pure polycarbonate and the polymeric films that comprise it.

Polycarbonates containing different weights of boehmite nanoparticles can be identified by the UV spectrum. While polymer films are exposed to UV light for some time, the films are stable for specific periods of time, indicating photostability during the irradiation process. The absorbance of the polymer films varied with the length of irradiation time and the amount of added boehmite nanoparticles as shown in Table 2

Table (2) Absorbance values of polycarbonate films with a thickness of (50 ± 5) microns pure as well as containing different concentrations of nano-boehmite calculated at a wavelength of 300 nm from UV-visible spectroscopy measurements

Absorbency (A_t)					Irradiation time (hour)	Chip type
160	120	80	40	0.0		
0.468	0.414	0.297	0.197	0.150	PC	
0.410	0.358	0.286	0.192	0.145	PC + 0.0025 % Nano-Boehmite	
0.376	0.329	0.244	0.183	0.142	PC + 0.005 % Nano- Boehmite	
0.326	0.252	0.203	0.178	0.139	PC + 0.01 % Nano- Boehmite	
0.308	0.231	0.192	0.163	0.135	PC + 0.02 % Nano- Boehmite	
0.277	0.216	0.184	0.152	0.115	PC + 0.04 % Nano- Boehmite	

Compared with the films of pure polymer, the absorption spectrum of the prepared polymer films with thickness (5 ± 50) microns, and different additive concentrations, varies with irradiation times. The absorbance of the films decreases directly with increasing additive concentration as in Table (2), the photodegradation of the polymer decreases in the presence of nanomaterial additives can be calculated by plotting the relationship between t In $A - A$ with irradiation time as shown in Figures (6) to (8) where we get a straight line which indicates that the photodegradation reaction of the additive is first order reaction.

From Table (2), we observe that the absorbance values of the pure film are inversely proportional to the irradiation time, as the absorbance value before irradiation was (0 0 0.0) and when irradiated to 160 hours it reaches (1). This is also true for the absorbance values of

films containing different concentrations of nanomaterials used as it decreases with the increase of the nanomaterial added in addition to that at the same time of irradiation at the reading time of a pure polymer is only 1.284 when added 0.005 to reach 0.873 and the decrease in absorbance continues as the concentration of the added material increases to reach a maximum of 0.302 at 0.04. 0 at the same time of irradiation, this applies to the rest of the irradiation stages.

The absorption spectrum of the prepared polymer films with a thickness of $(5 \pm 50) \mu\text{m}$, containing different concentrations with different irradiation times compared to the pure polymer film, the absorbance of the films decreases with increasing the concentration of the additive because the increase of the additive works to block the rays on the polymer films, as in Figure (4) and note that the films containing 0.05% of additive the absorbance decreases as its irradiation time increases, and this applies to the higher concentration used

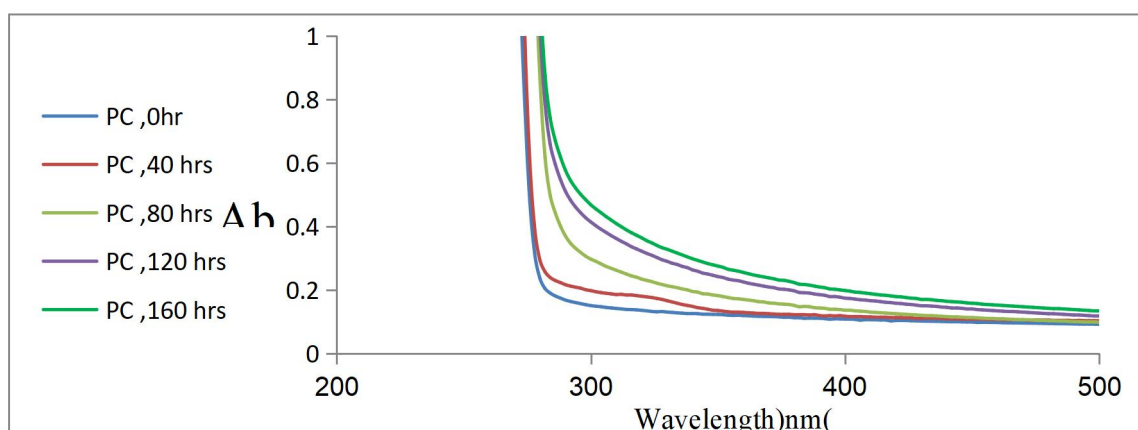


Figure (3): Change in UV-visible spectrum of additive-free polycarbonate films with a thickness of $(5 \pm 5) \mu\text{m}$ at irradiation times

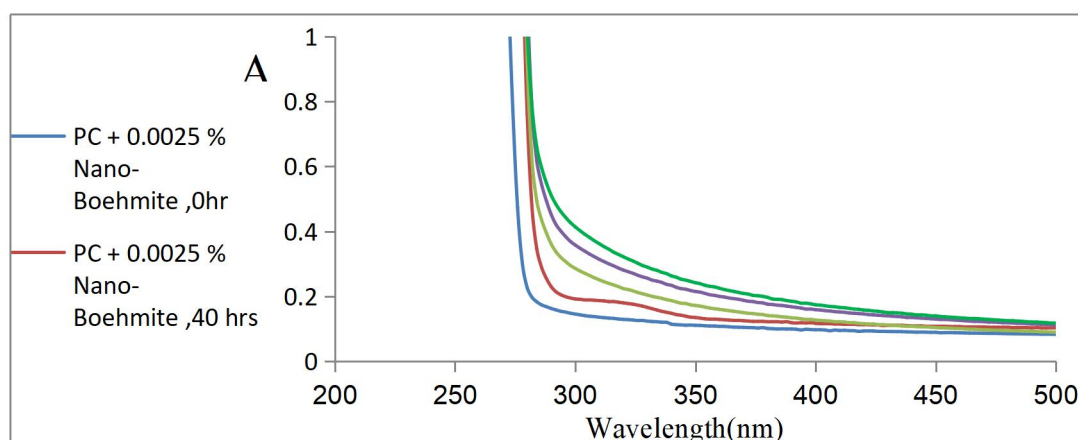


Figure (4) : Change in UV-visible spectrum of polycarbonate films containing 0.0025% concentration of

(Nano-Boehmite) with a thickness of (50 ± 5) microns at irradiation times

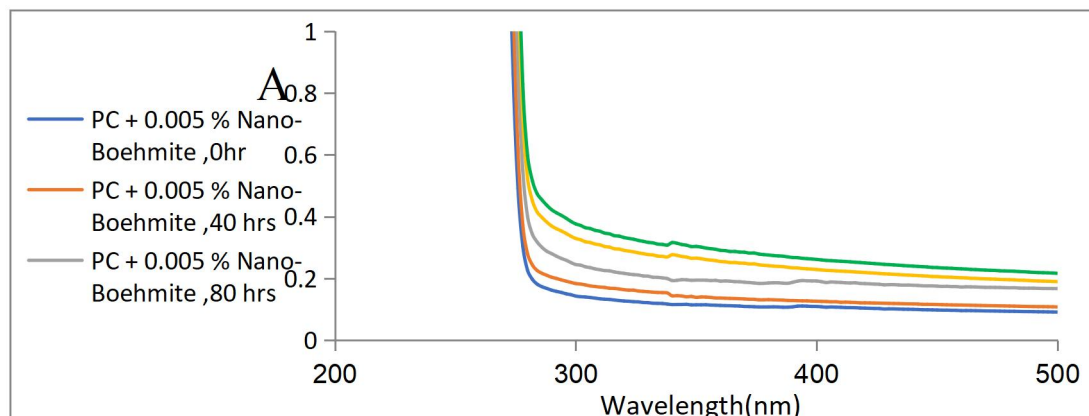


Figure (5): Change in UV-visible spectrum of polycarbonate films containing 0.0005%

(Nano-Boehmite) with a thickness of (50 ± 5) microns at irradiation times

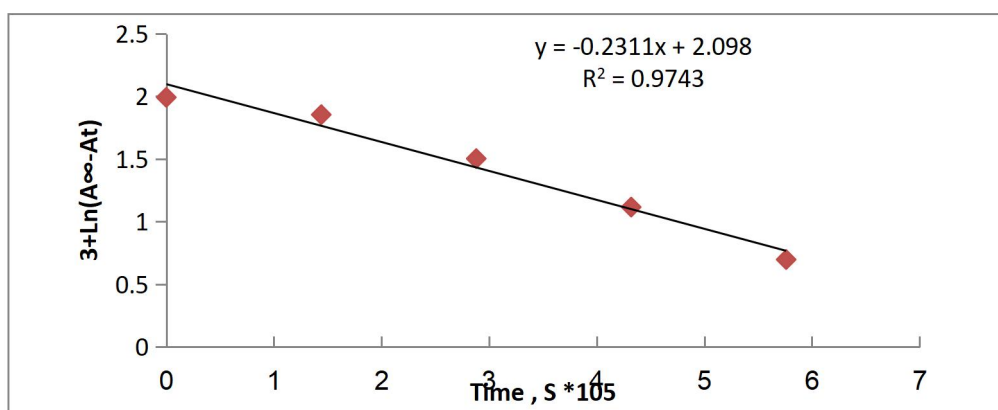


Figure (6): Relationship between the natural logarithm of the absorbance of 0.0025% Nano-Boehmite in polycarbonate films with a thickness of (50 ± 5) microns with irradiation time

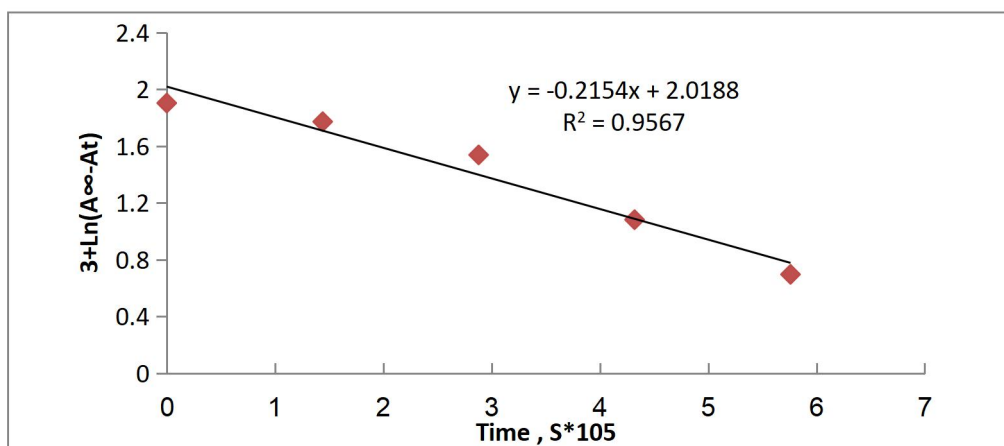


Figure 7: Relationship between the natural logarithm of the absorbance of nano-boehmite at a concentration of 0.005% in polycarbonate films with a thickness of (50 ± 5) microns with irradiation time

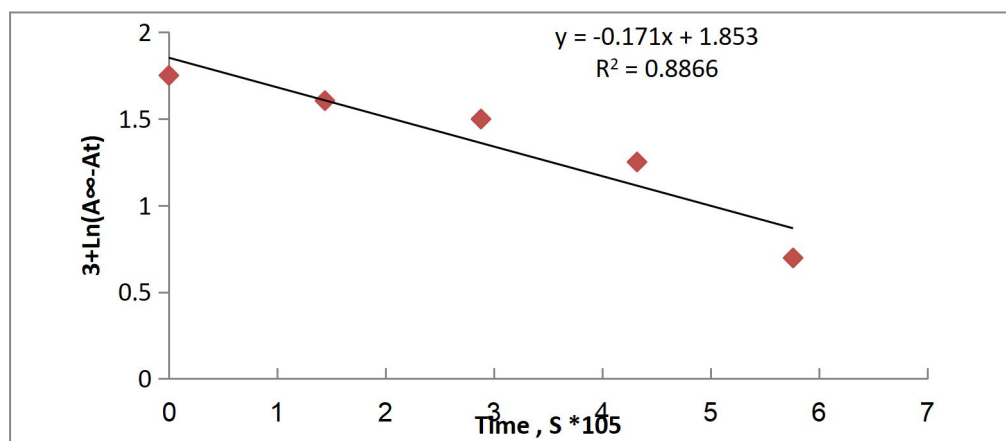


Figure (8): Relationship between the natural logarithm of the absorbance of nano-boehmite at a concentration of 0.01% in polycarbonate films with a thickness of (50 ± 5) microns with irradiation time

When the slope of the straight line (Slop) is calculated from the above graphs, the value of the photodegradation speed constant (K_d) of the nanomaterial added to the PC can be calculated according to the mentioned concentrations. It was found that the photodegradation speed constant (K_d) decreases proportionally with the concentration of nanomaterial added to the polymer (as in Table 3)

Table 3: Disintegration speed constants K_d for nano-boehmite in polycarbonate films

Dissociation rate constant K_d (Sec.) $\times 10^{-5}$	the focus%
0.231	PC + 0.0025 % Nano- Boehmite
0.215	PC + 0.005 % Nano- Boehmite
0.171	PC + 0.01 % Nano- Boehmite
0.162	PC + 0.02 % Nano- Boehmite
0.157	PC + 0.04 % Nano- Boehmite

8-2 Infrared spectroscopy

The disintegration process of pure polycarbonate, containing different concentrations of nanobohmite, was monitored during different periods of irradiation using infrared

spectroscopy (FT-IR) in the frequency range of 400-4000 cm^{-1} . This was done by observing the absorption bands of the carbonyl group ($\text{C}=\text{O}$) that appear as a result of the polymer being affected by photons under the influence of oxygen. The absorption band of the carbonyl group ($\text{C}=\text{O}$) appears at 1774 cm^{-1} ⁽¹⁶⁾. Oxygen acts as a catalyst, while $\text{Al}(\text{OH})_3$ (boehmite) nanomaterial acts as an inhibitor of the dissociation process

Monitoring the growth of the carbonyl group absorption band is important to evaluate the chemical effectiveness of pure polymer films containing different concentrations of nanobohmite, with the highest value at high concentration. The carbonyl growth coefficient (ICO) can be calculated using the baseline method⁽¹⁷⁾.

After exposing pure PC films with a thickness of $5 \pm 50 \mu\text{m}$ to FT-IR measurements, a change in the IR spectrum with different irradiation times was observed compared to the IR spectrum of pure PC before irradiation. Figure 9 shows the IR spectrum of pure polycarbonate before irradiation, while Figure 10 shows the IR spectrum of polycarbonate after 120 hours of irradiation. A change in the carbonyl group band is observed before and after irradiation as a result of UV exposure (as in Table 4)

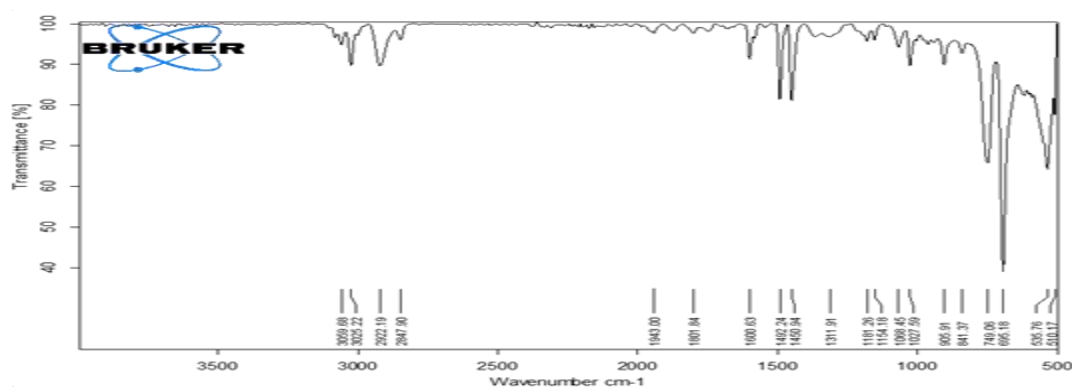


Figure (9): Infrared spectrum of an additive-free polycarbonate thin film before irradiation

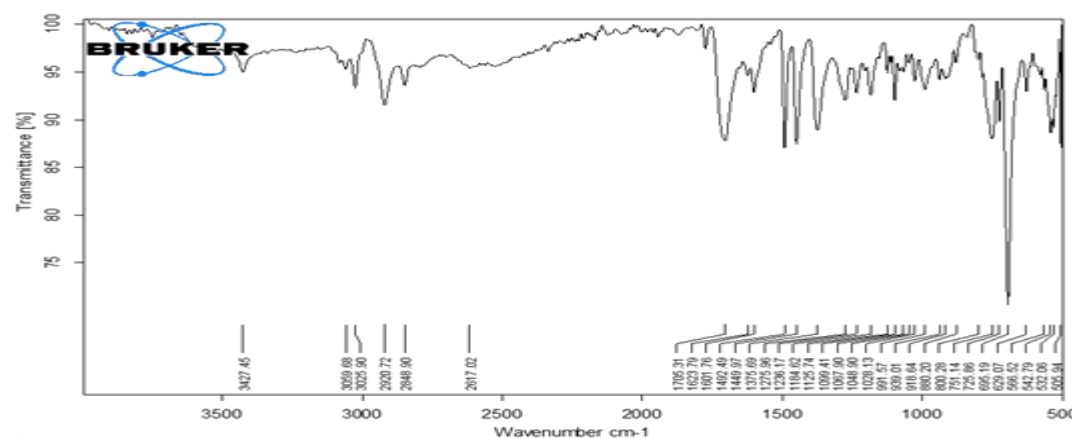


Figure (10): Infrared spectrum of an additive-free polycarbonate thin film with an irradiation time of (120)

hours

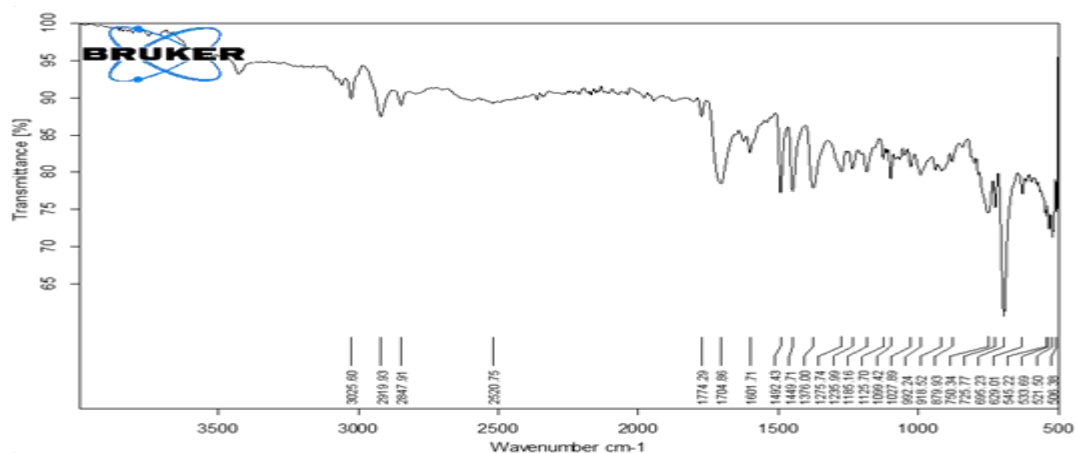


Figure 11: IR spectrum of a polycarbonate film containing nano-boehmite at a concentration of 0.04% with an irradiation time of 160 hours

Table (4): Carbonyl group absorption coefficient (ICO) values with irradiation time for polycarbonates containing different concentrations of Nano-Boehmite

I _{CO}					Irradiation time (hour) / Percentage
160	120	80	40	0.0	
1.489	1.381	1.284	1.089	0.000	PC
1.289	1.199	1.066	0.901	0.000	PC + 0.0025 % Nano-Boehmite
1.115	1.003	0.873	0.701	0.000	PC + 0.005 % Nano-Boehmite
0.908	0.805	0.679	0.538	0.000	PC + 0.01 % Nano-Boehmite
0.777	0.607	0.484	0.333	0.000	PC + 0.02 % Nano-Boehmite
0.567	0.437	0.302	0.156	0.000	PC + 0.04 % Nano-Boehmite

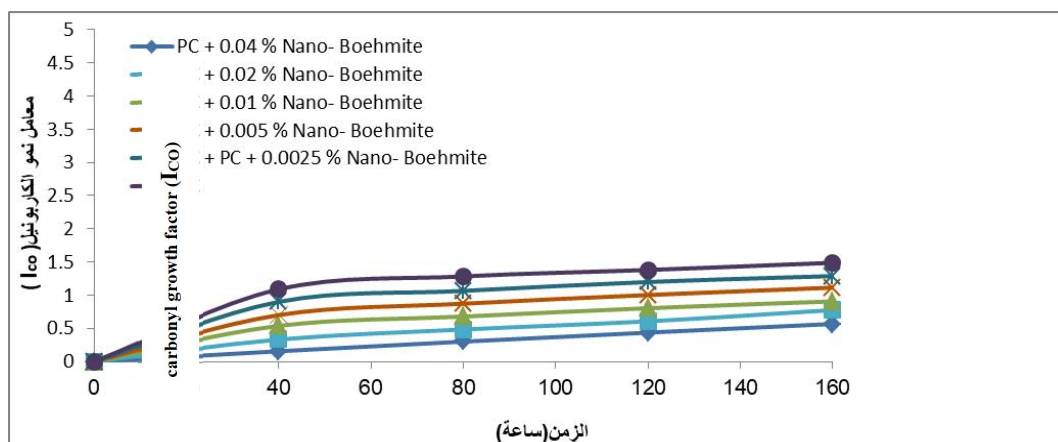


Figure (12): Relationship between carbonyl growth factor (ICO) and irradiation time for the results in Table 4

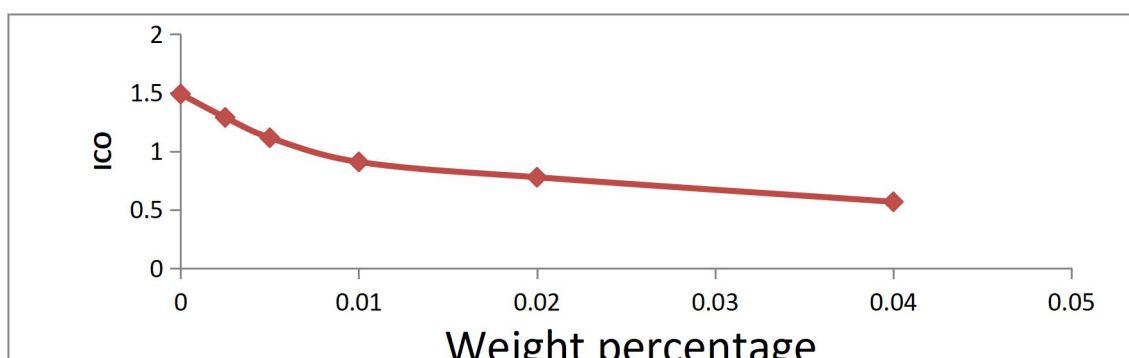


Figure 13: Change in the absorption coefficient of carbonyl group with the concentration of nano-boehmite for polycarbonate flakes at 160 hours

8-3 Viscosity measurement

As the irradiation time increases, the decrease in the average viscous molecular weight increases, as seen in viscosity measurements. Table 5.6 shows the viscosity with respect to molecular weight in the presence of the additive. The decrease in molecular weight increases very rapidly at the onset of irradiation and gradually increases, the reason for the rapid increase is due to the breaking of weak bonds. Figure (21) indicates that the speed of increase of the decrease in molecular weight viscosity is directly proportional to the square of the molecular weight viscosity in a given time interval. The fractionation is shown in Figures (22 ,23) and reflects the efficiency of nanomaterials in reducing optical fractionation. The study shows an increase in the decrease in the average molecular weight of the polymer with increasing number of hours of irradiation, and an increase in the decrease in the average molecular weight of the polycarbonate with increasing addition of nanoparticles on the polymer surface⁽¹⁷⁾.

$$[\eta] = 3.01 \times 10^{-4} M_v^{0.74} \quad (PC) \quad (2)$$

Table (5): Calculated values from viscous molecular weight measurements of pure PC flakes 254.2



Time Irradiation (hrs.)	$\overline{(M_v)} \times 10^3$	$(M_v)^2 \times 10^9$	$\frac{dM_v}{dt} = \frac{M_{v0} - M_v}{t}$	Degree of Polymerization P	$\frac{1}{P} \times 10^{-3}$	Deg. Degree $\alpha \times 10^{-3}$	Ava. Chain Scission (S)
0	210.133	44.155	∞	826.384	1.210	0.0	0.0
40	175.024	30.633	0.243	688.312	1.452	0.242	0.199
80	148.689	22.108	0.213	584.745	1.710	0.500	0.413
120	126.008	15.878	0.194	495.548	2.017	0.807	0.666
160	110.644	12.242	0.172	435.126	2.298	1.088	0.899

Table (5): Calculated values from viscous molecular weight measurements of pure PC flakes 254.28

Time Irradiation (hrs.)	$\overline{(M_v)} \times 10^3$	$(M_v)^2 \times 10^9$	$\frac{dM_v}{dt} = \frac{M_{v0} - M_v}{t}$	Degree of Polymerization P	$\frac{1}{P} \times 10^{-3}$	Deg. Degree $\alpha \times 10^{-3}$	Ava. Chain Scission (S)
0	210.133	44.155	∞	826.384	1.210	0.0	0.0
40	185.667	34.472	0.169	730.167	1.369	0.159	0.131
80	163.070	26.591	0.163	641.300	1.559	0.349	0.288
120	142.565	20.324	0.156	560.661	1.783	0.573	0.473
160	127.041	16.139	0.144	499.610	2.001	0.791	0.653

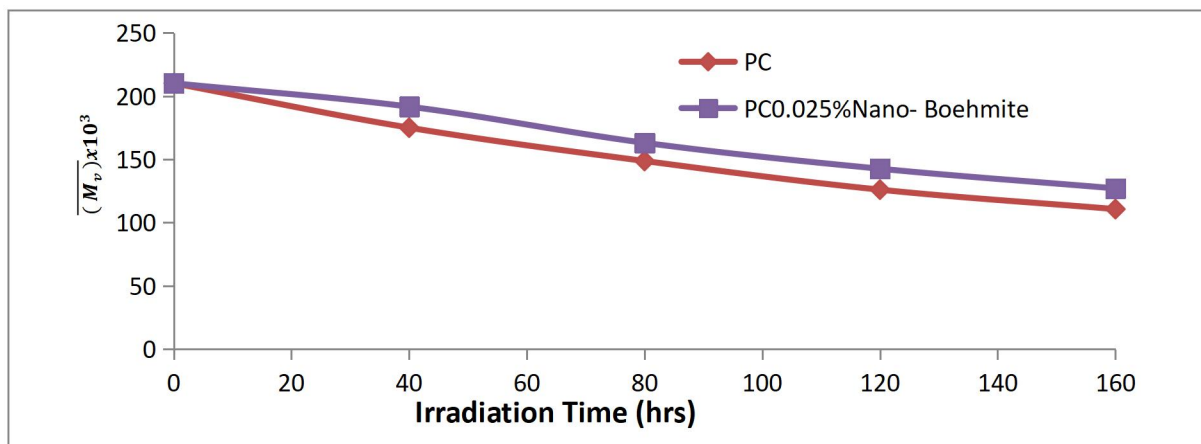


Figure (20): Relationship of average viscous molecular weight with irradiation time for PC wafers with and without 0.025% Nano-Boehmite concentration

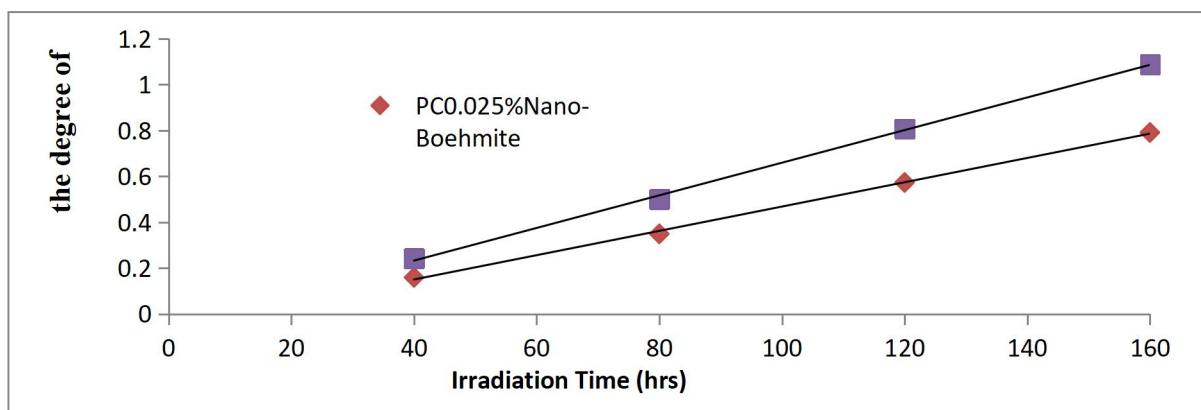


Figure (21): Relationship of the degree of fractionation with irradiation time for PC chips with and without 0.025% concentration of Nano-Boehmite

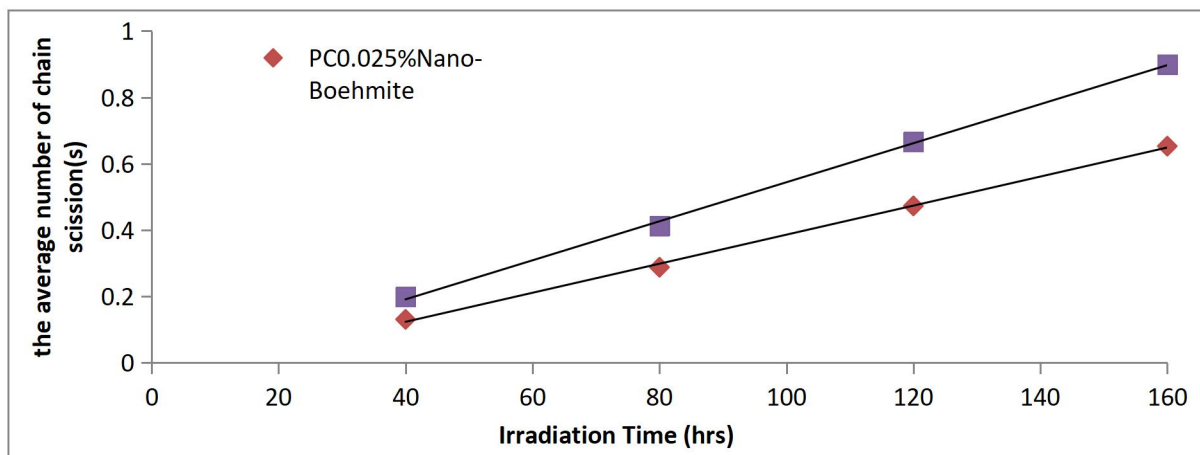


Figure (22): The relationship between the numerical rate of chain cutting and the irradiation time of computer chips in the presence and absence of nanobohmite concentration of 0.025%

9- Conclusions

Using aluminum waste, boehmite nanoparticles were manufactured. Its composition was proven by spectroscopic measurements. Several different concentrations of the added particles were then mixed with the polycarbonate polymer. It was found that the decomposition of the polycarbonate was inversely proportional to the concentration of the added nanoparticles. The results of the analyzes showed that the additive at different percentage concentrations inhibited the optical fragmentation of the polymer chips compared to the chips that did not contain additives. Moreover, polycarbonate films with high concentrations of boehmite nanoparticles lead to a faster inhibition process than at low concentrations. It was found that the decrease in the average viscosity molecular weight of the polymer increased by increasing the concentration of the added nanomaterial compared to the pure polymer. It was found that the molecular weight of the polymer decreases with the increase in the number of hours of irradiation. Which indicates inhibition of polymer degradation. Furthermore, boehmite nanoparticles were found to reduce light absorption.

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