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Development of UV-Resistant Low-Density Polyethylene/ ZnO Nano Composites for Irrigation Pipe Applications

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Abstract

This study investigates the influence of zinc oxide (ZnO) nanoparticles on the mechanical, thermal, and surface properties of low-density polyethylene (LDPE) nanocomposites under accelerated weatheringconditions, including UV exposure, elevated temperature, and humidity. LDPE samples were reinforced with varying ZnO concentrations (0.5%, 1%, and 2%) and compared to neat LDPE (D_0). Shore D hardness and Charpy impact tests showed enhanced mechanical stability in nanocomposites, with D_1 (0.5% ZnO) exhibiting improved hardness and impact resistance before and after exposure. Contact angle measurements indicated increased hydrophobicity in D_1 due to better dispersion of ZnO. At the same time, higher loading in D_2 led to a decrease in contact angle due to surface roughness and nanoparticle agglomeration. SEM analysis supported these findings, revealing smoother, defect-free surfaces in D_1 compared to D_0. Thermal analysis via TGA and DSC demonstrated that D_1 had superior thermal stability, with delayed degradation onset (432.8°C), reduced weight loss (89.18%), and higher enthalpy values (ΔH_2 = 136.7 J/g). In contrast, D_3 (2% ZnO) showed increased degradation and instability due to excessive nanoparticle aggregation. Overall, the incorporation of 0.5% ZnO proved optimal, balancing mechanical reinforcement, thermal durability, and surface performance, making it a promising candidate for outdoor applications such as irrigation piping.

Keywords: LDPE, ZnO ,AFM, SEM, Hardness , Impact strength ,TGA, DSC.

1. Introduction

Climate change and the increasing need for irrigation water have made modern agriculture increasingly reliant on smart irrigation systems such as drip irrigation, a cost-effective method. One of the most significant challenges facing these systems is the vulnerability of plastic irrigation pipes, such as low-density polyethylene (LDPE), to ultraviolet radiation. Especially in Iraq's hot and sunny environment, ultraviolet rays affect the life of the material, causing cracking, loss of elasticity, and mechanical deterioration over time[1-3]. ZnO has a high capacity to absorb ultraviolet rays and acts as a protective shield to protect the polymer from deterioration. Hence, the idea of improving irrigation pipes by adding nano zinc oxide (ZnO) to LDPE came about. In addition, ZnO helps improve the mechanical and thermal properties of the polymer and increases its surface stability. This is important in irrigation pipe applications that are exposed to continuous pressures and thermal changes[4,5]. Nanocomposites are important because of their remarkable properties, which result from the synergy between component characteristics and interfacial qualities[6,7]. Polymer nanocomposites have been manufactured, and some of their properties have been improved by introducing nanoscale inorganic compounds [8,9]. Adding certain metal oxides, such as $Al2O_3$, SiO_2, TiO_2, ZnO, and zirconium oxide (ZrO_2) to the polymers significantly enhances their properties [10].Additionally, van der Waals forces contribute

to the adhesion, facilitating more cohesive interlocking of the nanoparticles within the polymer matrix [11,12]. Enhancing the interfacial contact between the polymer and nanofiller is necessary to create polymer nanocomposites with beneficial chemical, thermal, and mechanical stability [13,14]. One of the most common methods for mixing thermoplastic polymers is the fusion mixing process, using high temperatures suitable for each type of polymer. The high temperature makes the polymer softer, so the reinforcing material is easily distributed[15-17]. In this research, LDPE molds with ZnO added in different proportions (0.5%, 1%, 2%) were prepared using the thermal extrusion technique. The models were evaluated through surface (Contact Angle, SEM), mechanical (hardness and impact), and thermal (TGA and DSC) tests, to determine the material's durability and improve its properties for irrigation water pipes.

2.Materials and Methods

2.1. Materials

Low-density polyethylene(LDPE) are supplied by Saudi Basic Chemical Industries Company (SABIC). The base material is LDPE, to which various percentages of ZnO are added (0.5%, 1%, 2%). Purity of ZnO 99.5% with white color and supplied by Saudi Basic Chemical Industries Company (SABIC).

2.2. Preparation of LDPE/ZnO Nanocomposite

Each percentage of nanoparticles is dispersed by ethanol using a magnetic stirrer for four h, after which it is added to the LDPE granules and mixed well until the nanoparticles are fully distributed. Then it is dried using a thermal oven at a temperature of 50°C to completely remove moisture and ensure that the polymer granules are dry. Then it is extruded directly into the thermal extruder. Extrusion was carried out using an England-made twin-screw extruder with six heaters, as shown in **Figure 1-a**. The extruder was heated to (220 C 250°C). The polymer granules were placed in the extruder at a 2 rad/sec rotation speed. The Nanocomposite material was extruded into a die measuring (150mm length x 100mm width x 5mm thickness) and then transferred directly to a locally made thermal-hydraulic press, as shown in **Figure 1-b**. The press was heated to the same temperature as the extruder (220 C-250°C). A pressure of 5 Pa was applied for (10-15) minutes, after which the die was transferred to a water bath to cool to room temperature, as shown in **Figure 2**. Proportions of materials used in this study are mentioned in **Table 1**.

Table 1. Nanocomposite martial

Sample	LDPE(wt%)	Zno(wt%)
D_0	100%	0%
D_1	99.5%	0.5%
D_2	99%	1%
D_3	98%	2%

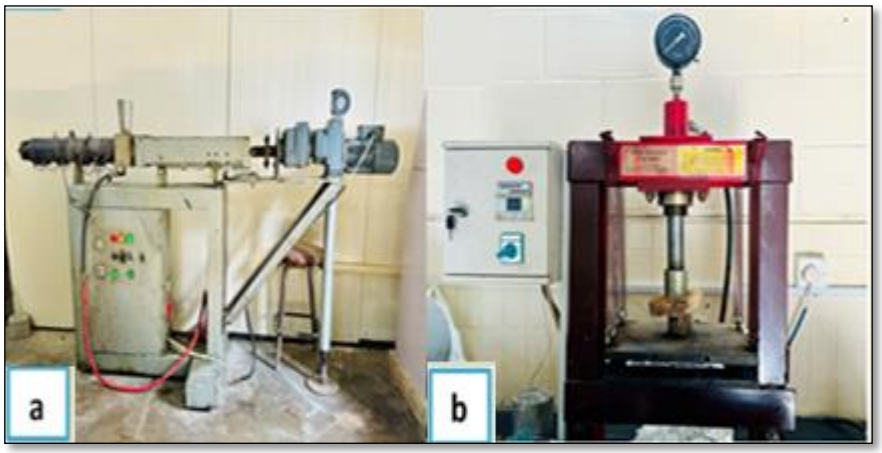

Figure 1. a-Extruder machine, b- Thermal hydraulic press.

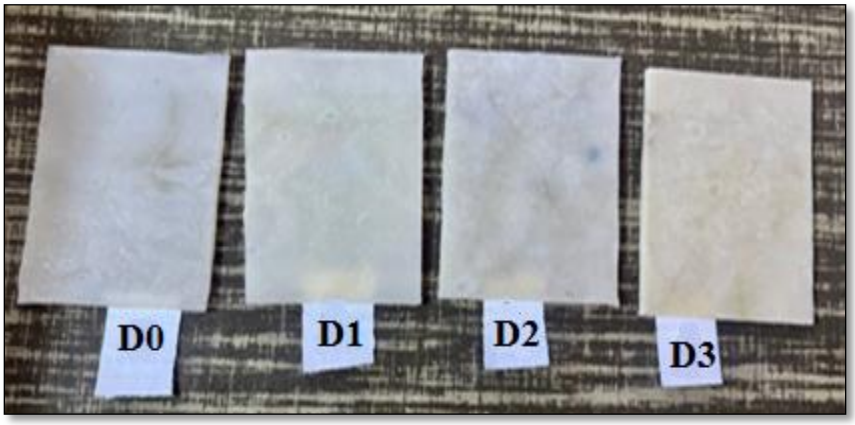

Figure 2. LDPE/ZnO Nanocomposite sample.

2.3. Diagnostics of LDPE /ZnO composition

Atomic Force Microscopy (AFM) is examination used to study the surface topography and smoothness at the nanoscale, through which the details of the surface structure, such as pores and roughness, can be determined and studied. It also gives us the size of the ZnO particles. The scanning electron microscope is a tool used to analyze and examine the surface structure of materials with high accuracy. It provides detailed images showing the shape, distribution, and size of the nanomaterial within the polymer for the purpose of studying the morphological properties of samples. Hardness is the resistance of materials to scratching, denting, or deformation. Udometer (shore D), ASTM D2240. It is one of the important mechanical tests to determine the resistance of materials to deformation or scratching when a certain force is applied (Hassan et al[18]). Charpy impact tests were performed to determine the mechanical performance of impact strength utilizing impact testing according to the ISO-179 standard test technique, with dimensions of 5.5 cm × 1 cm. The samples were cut according to ASTM specifications for the device, as shown in **Figure 3.** Contact angle: Through this test, we can study how a drop of water or any liquid behaves when placed on the surface of a material. If the angle is small, this means that the liquid spreads easily and is hydrophilic. However, if the angle is large, the liquid does not spread, and the surface is hydrophobic. Thermogravimetric analysis (TGA) is a technique that measures the mass of a sample relative to its temperature change and studies the thermal stability of a nano-reinforced polymer material. The samples weighed 3–5 mg and were heated at a rate of 10 °C/min from 0- 700°C. Differential scanning calorimetric (DSC): It is a suitable test for analyzing a material's thermal behavior and phase transformations, as it measures the temperature change and determines the melting point and glass transition point of the material.

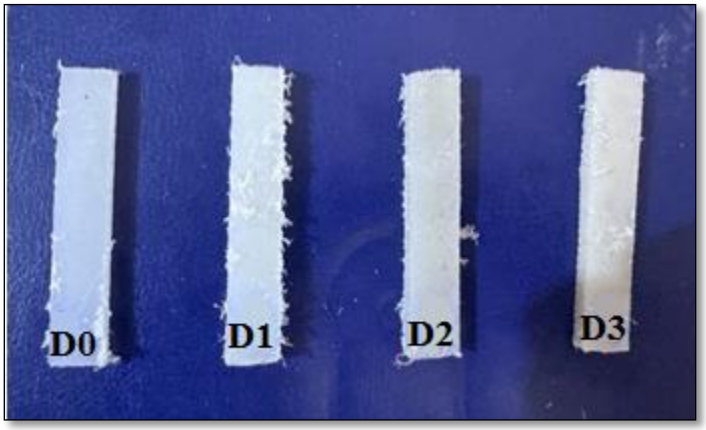

Figure 3. Sample LDPE/ZnO Nanocomposite for impact test

3.Results

3.1. Atomic Force Microscopy Analysis

The surface morphology of Zinc oxide nanoparticles was observed with AFM micrographs. The average diameter for zinc oxide is 56.86 nm. The particles are uniformly distributed and have a clear diagonal alignment. ZnO nanoparticles appear ellipsoidal or elongated and mostly overlap, as shown in **Figure 4.**

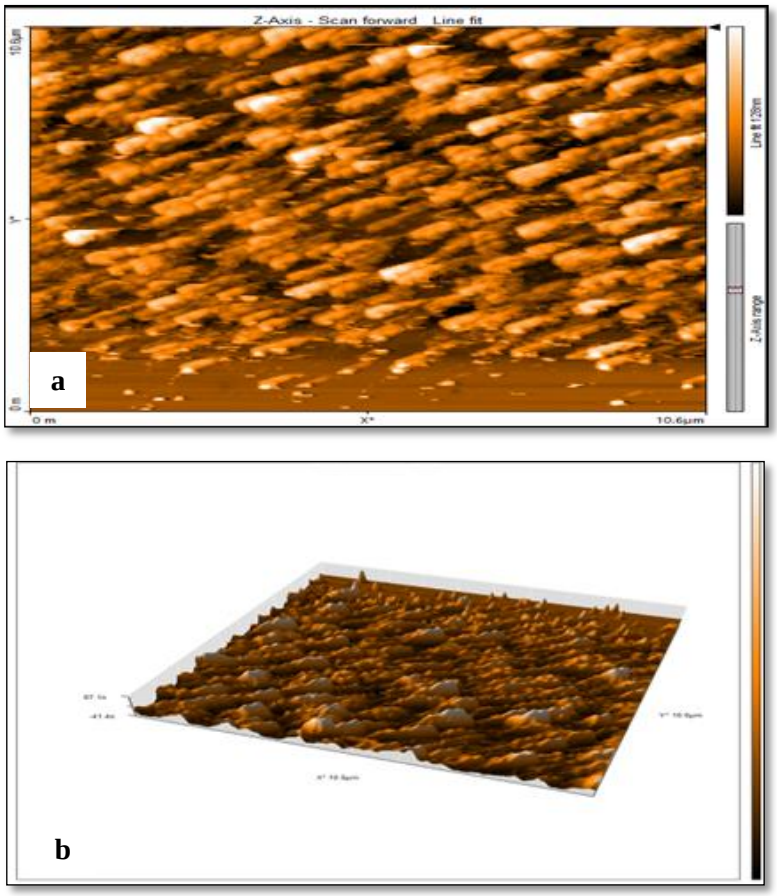

Figure 4. a ,b- FM analysis image of ZnO nanoparticle

3.2. Scanning Electron Microscope (SEM)

SEM examination was conducted for the lowest and highest percentage of added nanoparticles. We notice the image of sample D_1 as shown in **Figure 5-a**. The distribution of ZnO nanoparticles is slightly irregular. It shows areas with light clumping and other areas that are almost empty because the percentage of added nano 0.5%wt is low, which made its effect limited.

Figure 5-b for sample D_3, which contains 2% ZnO, is distributed almost homogeneously on the surface of the LDPE polymer. There are some clumps in certain areas. This means that the percentage of ZnO has not been completely integrated into the matrix.

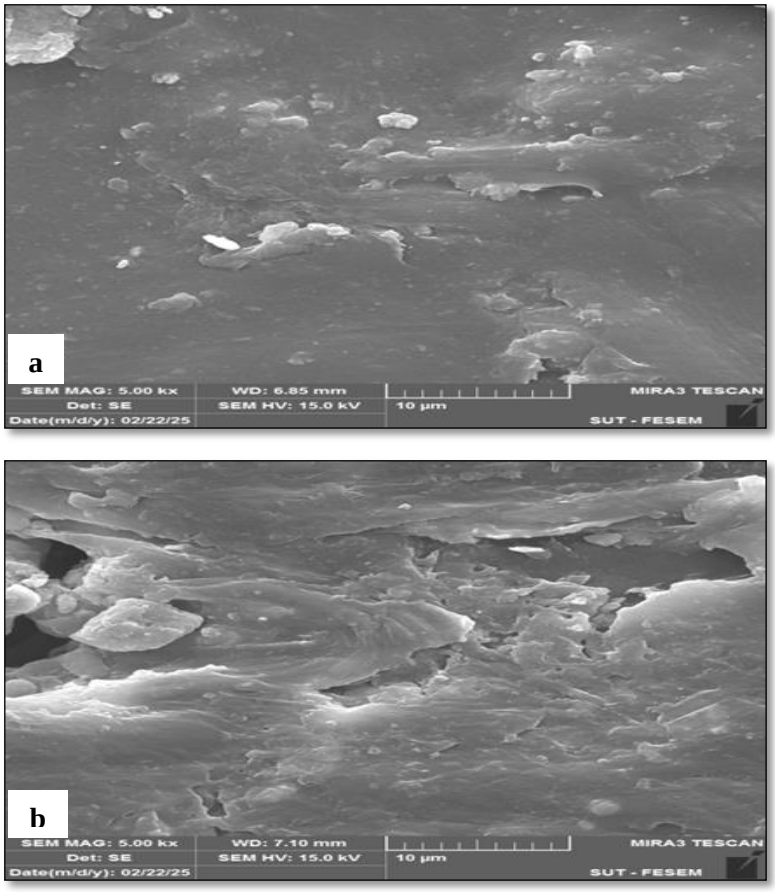

Figure 5. SEM images of the Nanocomposite with a scale of 10µm.(a)D_1 (99.5% LDPE + 0.5% Zno).(b) D_3 (98% LDPE + 2% Zno).

3.3. Mechanical Properties

Samples were cut according to ASTM standards for each test and placed in the Accelerated Weathering Tester device (QUV) for 140 hours under atmospheric conditions at 50°C. After applying moisture, the temperature was reduced to 50°C and exposed to 340nm UV light. Mechanical tests were then conducted before and after exposure to accelerated atmospheric conditions to study the effect on the polymer's mechanical properties

3.3.1. Hardness Evaluation of LDPE and ZnO/LDPE Nanocomposites Before and After Accelerated Weathering Exposure

The hardness test was performed for sample D_0. It was (56.5), which is considered a normal value for LDPE. This means that the material is cohesive and has not been exposed to any deterioration, and the surface has not deteriorated and is relatively smooth. However, after exposure to atmospheric conditions (UV, humidity, and heat), we notice that the hardness value decreased to 55.2, as shown in **Figure 6**. This means that the material began to lose its cohesion and weaken as a result of the effect of ultraviolet rays, which cause cracking in the polymer chains. After adding 0.5% ZnO to the polymer in sample D_1, the hardness became 57.7, higher than the hardness of D_0. This is due to the presence of nanoparticles as a filler, which increased the cohesion and hardness of the polymer. However, after exposure to atmospheric conditions, the hardness decreased slightly to 56.1, as shown in **Figure 6**. This means that deterioration occurred, but at a lower rate than D_0, because ZnO absorbs and scatters ultraviolet rays and acts as a UV stabilizer. Therefore, it reduced chain breakage and prevented rapid polymer decomposition. Sample D_2 contains 1% ZnO.

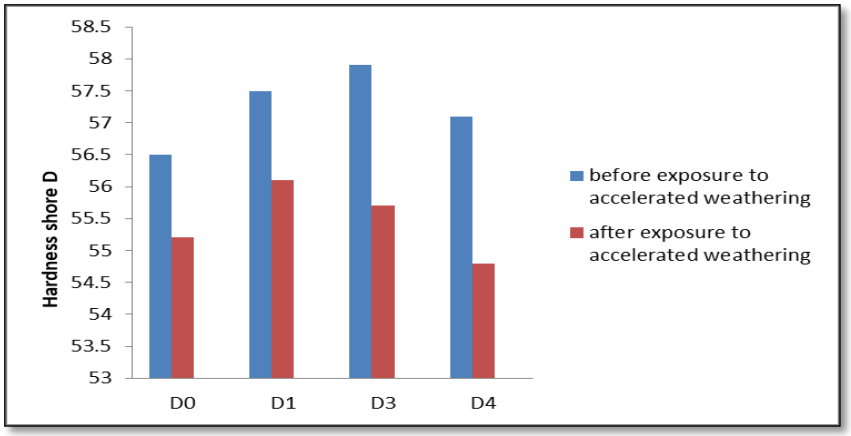

Figure 6. Hardness shore D before and after exposure to accelerated weathering.

3.3.2. Impact Evaluation test for LDPE and ZnO/LDPE Nanocomposites Before and After Accelerated Weathering Exposure

Charpy impact tests were performed to determine the mechanical performance of impact strength. The results showed that sample D_0, which consisted of LDPE only, before exposure to atmospheric conditions, recorded an impact energy of 43.506 KJ. In comparison, sample D_1 recorded a higher energy level of 49.504 KJ as shown in **Figure 7**. The reason for this improvement is the presence of 1% ZnO nanoparticles that act as a filler, enhancing the polymer's cohesion and contributing to stress distribution when exposed to impacts, leading to an increase in the material's resistance to fracture.After exposing the samples to harsh weather conditions (temperature 60 °C, humidity, and ultraviolet rays with a wavelength of 340nm), we noticed a decrease in the shock resistance of both samples, as D_0 decreased to 38.68 KJ, and D_1 decreased to 39.285 KJ, as shown in **Figure 7**.

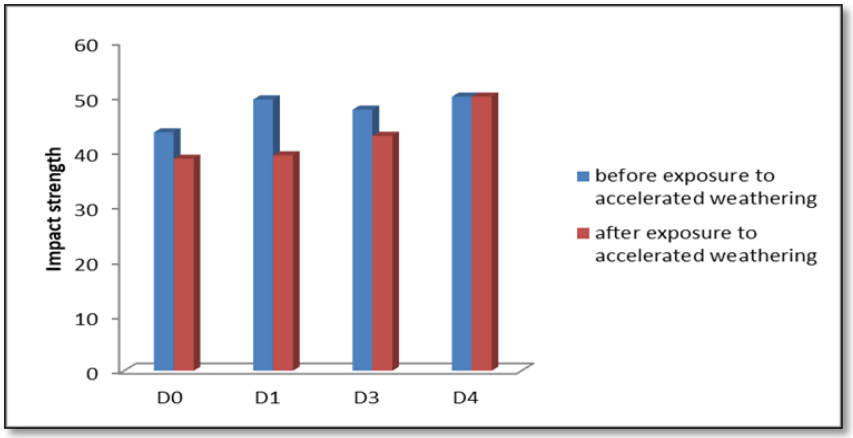

Figure 7. Impact strength before and after exposure to accelerated weathering.

This decrease is explained by the occurrence of decomposition in the polymer chains as a result of exposure to ultraviolet rays, which led to the deterioration of mechanical properties. However, the decrease in D_1 was relatively less due to the effective role of ZnO absorbs and disperses ultraviolet rays, thus significantly reducing the rate of deterioration. This supports the hardness test result, as D_1 maintained a higher hardness value than D_0 after exposure to UV, i.e., it has good structural cohesion. This result was confirmed through SEM examination. In sample D_2, containing 1% ZnO added to LDPE, the impact strength before exposure to weathering was 47.646 KJ, which is slightly lower than that of sample D_1 but higher than D_0. This indicates that adding 1% ZnO helped increase the cohesion of the material and its resistance to fracture. However, this higher concentration of the nanomaterial caused an increase in the hardness of the

material and a decrease in its ductility. For this reason, D_2 did not outperform D_1 despite the presence of a higher percentage of ZnO. After exposure to ultraviolet rays, heat, and humidity, we notice that the shock resistance value decreased, as shown in **Figure 7**, to 42.857 KJ.

3.4. Contact angle

After conducting a contact angle test, the results showed a clear change in the surface behavior of the samples. Sample D_0 represents pure LDPE and has an average contact angle of 92.12, shown in **Figure 8-a**, indicating that the surface possesses balanced hydrophilic and hydrophobic properties. When 0.5% ZnO was added to sample D_1, we noticed an increase in the contact angle value at a rate of 99.60, as shown in **Figure 8-b.** This indicates that the surface has become more water-repellent. This behavior, due to the good distribution of nanoparticles on the polymer surface, led to the blocking of pores and the reduction of surface roughness, as shown by the SEM examination result. When the percentage of nanoparticles increased to 1%, as in sample D_2, a significant decrease in the contact angle was observed at a rate of 74.28 ,as shown in **Figure8-c**), this indicates that the surface behaves in a hydrophilic manner, and the reason for this decrease is the presence of irregular clusters of ZnO. As shown in **Figure 8-d**, there is a significant decrease in the contact angle value for sample D_3 compared to the other samples, indicating that it is hydrophilic. This is related to the increase in the percentage of ZnO within the polymer. The decrease in the contact angle is because the ZnO nanoparticles carry hydroxyl groups (-OH) on their surface that can form hydrogen bonds with water, which makes the surface hydrophilic. From **Figure b-5**, the SEM examination of sample D_3 shows that the distribution of the nanoparticles was not completely homogeneous, with the presence of some clumps that create a contrast in the surface roughness, increasing the interaction between the water droplet and the sample surface, thus reducing the contact angle.

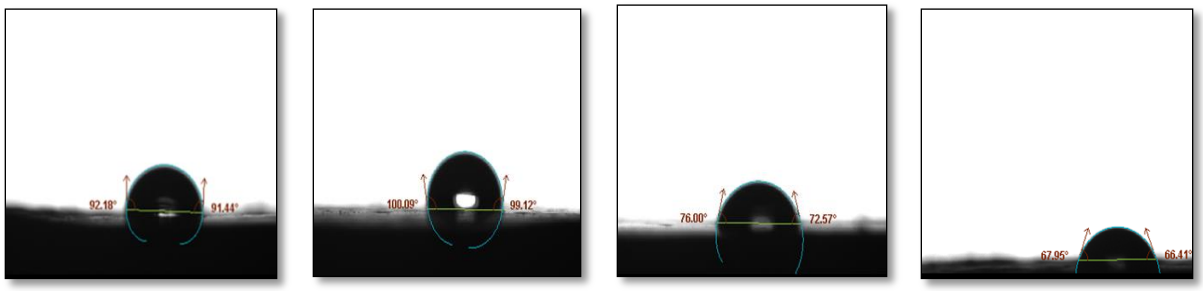

Figure 8. Water contact angle of a. D_0 b. D_1 c.D_2 d.D_3

3.5. Thermal properties (TGA&DSC)

Thermogravimetric analysis (TGA) was applied to evaluate the thermal stability of the nanocomposite material. From **Figure 7**, sample D_0, which contains 100% LDPE, shows the expected thermal behavior consistent with the properties of this polymer. Actual thermal decomposition began at approximately 437°C and continued until 506°C as shown in **Figure 9-a**. The sample lost approximately 97.3% of its original weight, which is evidence of its high purity and the absence of inorganic fillers. DSC analysis reveals multiple decomposition and melting stages. The first phase transition occurs at 28C-55°C due to partial crystallization, followed by a thermal decomposition stage at 406°C and final decomposition peaks at 506°C-492°C, indicating complete structural breakdown of the polymer. This result supports the ability of LDPE to decompose at high temperatures and emphasizes the importance of monitoring thermal stability in industrial applications [28].TGA and DSC analysis of sample D_1 showed good thermal stability upon the addition of 0.5% ZnO. The thermal decomposition started at 432°C and reached its peak at 504.37°C, resulting in a weight loss of 89.18% of the original weight, as shown in **Figure 9-b**. The DSC curve showed two distinct thermal peaks at 406.45°C and 489.55°C, indicating multiple decomposition stages resulting from the interaction of ZnO with the polymer matrix. Compared to D_0, the addition of 0.5% ZnO contributed to enhanced thermal stability and increased absorbed energy, confirming the role of nanoparticles in improving

thermal performance. The thermal analysis results, as shown in **Figure 9-C**, for sample D_3 showed a distinct thermal behavior compared to the previous samples. The TGA result showed the onset of thermal decomposition at 444.84°C, and the peak of thermal decomposition reached 489.88°C with a high weight loss of 98.92% of the original weight, indicating almost complete decomposition. The DSC results, as shown in Figure 7-C, showed the presence of multiple thermal peaks at 270°C, 374.05°C, 485.53°C, and 485.53°C with a high thermal energy of 389.2J/g. **Table 2** shows the values of the TGA & DSC test results.

Table 2. thermal degradation and enthalpy data of LDPE/ZnO nanocomposite based on TGA and DSC analysis.

Sample	Zno(wt%)	Beginning of decomposition (C°)	end of decomposition (C°)	Weight loss (wt)	ΔH_1(J/g)	ΔH_2(J/g)
D_0	0%	420.1	485.6	97.30%	282.6	105.3
D_1	0.5%	432.8	504.4	89.18%	263.3	136.7
D_3	2%	444.8	489.9	98.92%	248.0	153.9

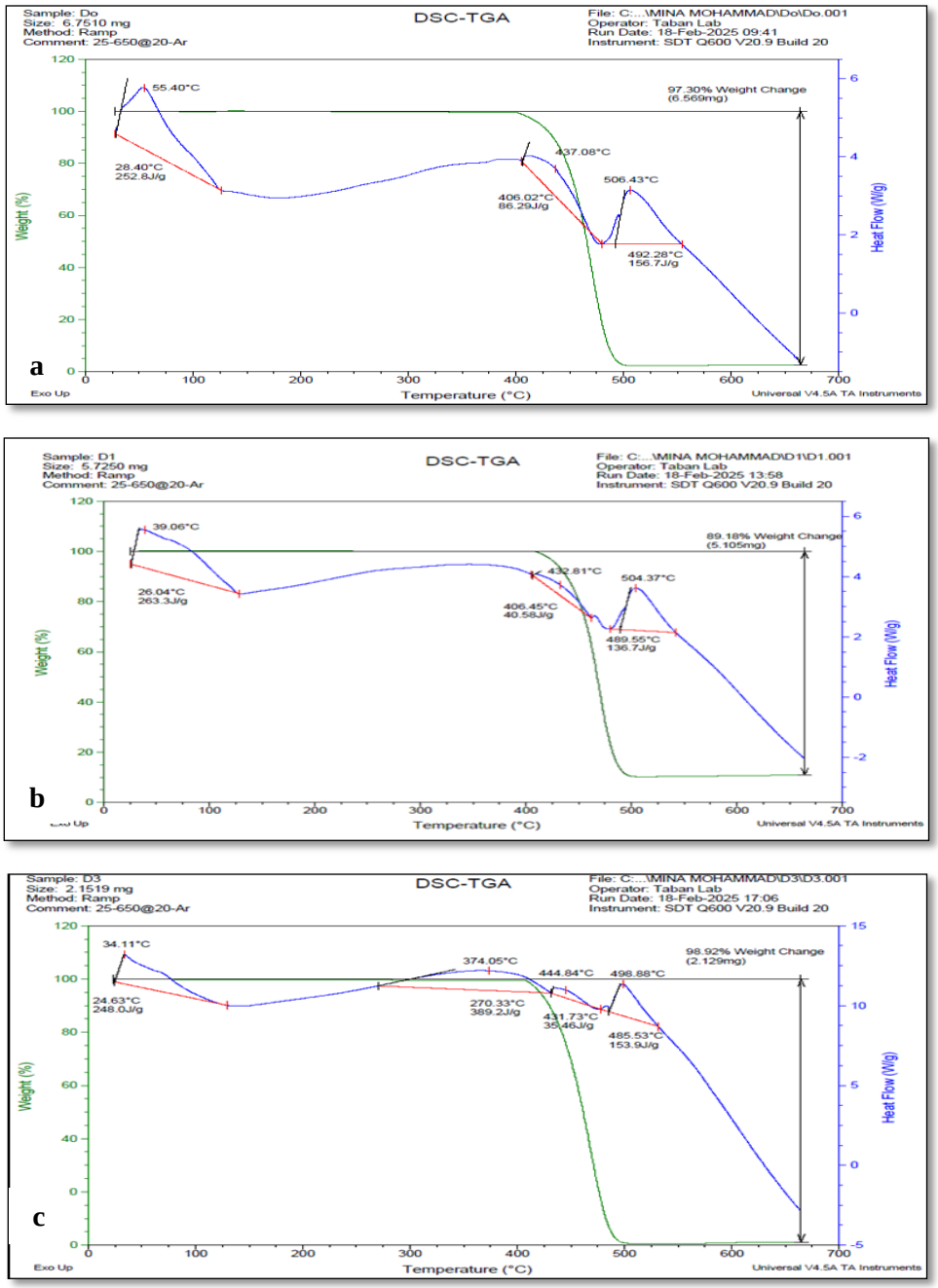

Figure 9. TGA and DSC of a. D_0 b. D_1 c. D.

4. Discussion

The ZnO particles are spaced apart, so there is no strong bond between them and the polymer. The dispersion is considered imperfect because the nanoparticles did not disperse well in the LDPE, resulting in some small gaps. The overall surface appearance appears relatively smooth, but the ZnO and LDPE have not yet reached the stage of perfect integration. We conclude that the small percentage of nanomaterials was unable to change the microstructure significantly, so the potential for improving mechanical properties remains limited[19,20]. The presence of ZnO particles gives a rough surface that may affect the mechanical properties. There appear to be some small gaps and voids that may be a result of weak bonding between the polymer and ZnO, which negatively affect the mechanical properties[21,22].

Its hardness is higher than d0 and d1 before exposure to weathering conditions because ZnO acts as a filler that strengthens the polymer and makes the material more cohesive, harder, and resistant to deformation under load. However, after exposure to weather conditions, the hardness may decrease, but only slightly, because the nanomaterial absorbs and scatters a large portion of the ultraviolet rays, thus reducing the degradation of the material [23]. The presence of 2% ZnO, as in sample D_3, relatively increased the hardness of the polymers, which was 57.1 before weathering. According to the SEM examination, the distribution was not 100% uniform, with clumped areas forming weak points once weathered, especially since humidity, temperature, and UV affect the bond between ZnO and LDPE. After weathering, the hardness value decreased slightly to 54.8. This indicates that the ZnO partially supported the sample in resisting weathering [24].

Despite the decrease, D_2 remains better than D_0 and D_1 after exposure. This proves that increasing the concentration of the developing material ZnO to 1% After exposure, proves that increasing the concentration of the developing material ZnO to 1% enhances protection against photodegradation and prevents significant deterioration in mechanical properties under the influence of atmospheric conditions. This supports the interpretation of the hardness test, as sample D_2 showed relatively stable hardness after exposure to UV, meaning that ZnO acts as an effective protector against UV and stabilizes the polymer chains [25]. Sample D_3 showed remarkable stability in the impact test, as the impact strength value reached 50 KJ before and after exposure to weather conditions, meaning that the material maintained its strength and fracture energy absorption after adding 2% ZNO. Even after exposure to weathering conditions, the nanoparticles worked to straighten the polymer matrix and reduce chain movement. Although the hardness test showed a slight decrease after exposure to weathering conditions, it did not affect the internal structure in a way that would weaken the impact resistance. This reflects the effectiveness of ZnO incorporation in enhancing the mechanical stability of the composite under different environmental conditions [26] .

At this higher concentration, the surface roughness increased, which led to an improvement in the ability to absorb water instead of repelling it. This change in surface behavior when the concentration of the nanomaterial varies reflects the sensitivity of the nanopolymer structure to the concentration and distribution of the nanomaterial. It is a key factor in modifying the functional properties of materials [27].

Thermogravimetric analysis (TGA) was applied to evaluate the thermal stability of the nanocomposite material. This reflects complex thermal reactions resulting from the high concentration of ZnO compared to the low percentage in D_1, which showed better thermal stability. The increase in the percentage of ZnO in sample D_3 led to the agglomeration of nanoparticles, as shown by the SEM results, which reduces their efficiency and negatively affects their distribution within the polymer matrix. Therefore, the high concentration of ZnO did not show a clear enhancement in thermal performance, but was associated with increased decomposition and mass loss[29,30.]

5. Conclusion

The results of this study showed that the addition of nanoparticles to LDPE improved the surface, thermal, and mechanical properties of the polymer under harsh environmental conditions such as UV radiation, humidity, and temperature. The hardness of samples D_1 and D_2 was higher than that of D_0, with a smaller decrease after exposure to atmospheric conditions. This confirms the behavior of nanoparticles in strengthening the polymer and its resistance to damage. As for the contact angle, results showed that D_1 has a more hydrophobic surface, as the contact angle was 99.6 As a result of the regular distribution of nanomaterials, the angle for D_2 was less at 74.28 due to the increased concentration of ZnO, which led to a rougher surface and made it more hydrophilic.While the thermal analysis showed that sample D_1 had the best thermal stability, as it recorded the lowest weight loss of 89.18% and a significant increase in the second latent heat Δ, H_2 = 136.7 J/g. This indicates that it has better thermal resistance, in contrast to sample D_3, which contained 2% ZnO. Although it had a higher latent heat, it recorded a significant weight loss of 98.92%. This indicates a decrease in structural stability with increasing nanomaterial content. Based on these results, sample D_1 (0.5% ZnO) offers the best balance between mechanical strength, thermal stability, and weather resistance, making it most suitable for irrigation pipe applications that require high tolerance to solar radiation and harsh environmental conditions.

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Conflict of Interest

The authors declare that they have no conflicts of interest.

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