ORIGINAL PAPER

A comparative analysis of the effects of ultrasound on the characteristics of TiO_2 -modified LDPE

Ali Yakoub Alkhair*, Irina Anatol'evna Kirsh, Anastasia Alexandrovna Fedorova

Department of Chemistry, Polymer Technology and Packaging Solutions, Federal State Budgetary Educational Institution of Higher Education Russian Biotechnological University, Moscow, Russia Federation

*Corresponding Author Email: alkhaira@mgupp.ru

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ABSTRACT

This article aims to reduce economic losses from milk spoilage and deterioration by developing active, effective packaging materials based on low-density polyethylene with antimicrobial and antioxidant properties. The article examines how ultrasound affects the antimicrobial properties of low-density polyethylene films modified with 0.5%, 1%, or 3% titanium dioxide (TiO_2), including their effectiveness against gram-positive bacteria (Bacillus subtilis), gram-negative bacteria (Escherichia coli), yeast (Candida albicans), and fungi (Aspergillus niger). Additionally, the ability of the films to maintain the acid number of milk during 10 days of storage at 4 ± 2 °C was studied. To evaluate the impact of ultrasonic treatment and TiO_2 addition on forming new chemical bonds and groups within the polymer matrix, samples were examined via Fourier transform infrared spectroscopy (FTIR). The physical and mechanical properties, as well as the water vapor transmission rate (WVTR), of all polyethylene samples were studied. Ultrasound technology has shown great ability to prevent an increase in the acid number of stored milk, as the acid number of milk packaged in ultrasonically treated polyethylene containers decreased by 50% at a concentration of 0.5% TiO_2, as well as improving antimicrobial properties in the Candida albicans and Aspergillus Niger relationship. As for the physical and mechanical properties, the elongation at break was significantly improved using ultrasound. At a concentration of 3%, this resulted in an increase in elongation at break of 39.4% in the longitudinal direction and 43.2% in the transverse direction. Regarding the effect of ultrasound on the barrier properties of polyethylene, ultrasound technology decreased the water vapor transmission rate (WVTR) at all concentrations. WVTR decreased by 16.6% in polyethylene films with 3% TiO_2 that were ultrasonically treated compared to untreated films with the same concentration of TiO_2.

Keywords: low-density polyethylene; titanium dioxide; antimicrobial and antioxidant properties; water vapor transmission rate (WVTR); spectroscopy (FTIR).

INTRODUCTION

Packaging materials play a critical role in ensuring food safety, extending shelf life, and maintaining quality. This has prompted leaders in the global plastics and packaging industry to develop enhanced packaging materials that improve the quality of food products. One innovative approach is introducing additives with antimicrobial and antioxidant activity into packaging materials. This provides additional protection against microbial growth on the product's surface and limits the oxidation process, which significantly reduces the sensory quality of food products [1].

TiO_2 is a chemically inert, heat-resistant compound that is widely used as a filler in polymeric materials. It exists in three polymorphic forms: rutile, anatase, and brookite. Rutile is the most widely used form in polymer engineering due to its high thermal stability and refractive index [2]. TiO_2 protects polymers from degradation and deterioration of their physical and mechanical properties by absorbing ultraviolet radiation (energy <400 nm) due to its high refractive index (n = 2.7 for rutile), which allows it to reflect light. Through these mechanisms, TiO_2 protects the polymer chain structure from deterioration [3]. However, pure TiO_2 must be pretreated with materials such as aluminum oxides or silica before being introduced into the polymer matrix. This is because TiO_2 forms free radicals when exposed to ultraviolet radiation. These free radicals negatively impact the technical properties of polymers [4]. TiO_2 has been used in many studies to produce polymeric materials with improved properties, such as crystallinity, photolysis, and thermal decomposition. In a study by Ndibewu et al. investigating the effect of TiO_2 on the physical and chemical properties of low-density polyethylene (LDPE), four LDPE films were obtained by adding TiO_2 at concentrations of 0%, 5%, 10%, and 15% by weight, respectively, using extrusion technology. Compared to the control sample, adding 5% TiO_2 decreased the tensile strength. However, increasing the concentration to 10% and 15% increased the tensile strength of the membranes, surpassing the control sample. After treating the polyethylene films with ultraviolet radiation, the tensile strength decreased by 0.5 MPa at a concentration of 5% TiO_2. Adding TiO_2 at concentrations of 0%, 5%, 10%, or 15% decreased crystallinity compared to the control sample before exposure to ultraviolet radiation by 13.7%, 13.3%, 10.2%, and 9.6%, respectively [5]. Another study produced polypropylene-based films using extrusion technology and various concentrations of TiO_2 with an average particle size of 0.28 μm, a specific gravity of 4.1, and an oil absorption capacity of 35 g/100 g (TiONA 244): 5%, 10%, 20%, and 30%. The study aimed to investigate the thermal decomposition kinetics of polypropylene compounds using the Horowitz-Metzger method, the Coats-Redfern method, and the Criado model. The results showed that, at a low concentration of 5%wt TiO_2, the thermal stability and activation energy of polypropylene increased due to increased interfacial interactions. At a concentration of 10-20% wt, TiO_2 aggregates within the polymer matrix and the activation energy decreases. However, at a concentration of 30% wt, the activation energy rises again. This increase is attributed to the interconnection of the TiO_2 aggregates forming a continuous network within the polypropylene matrix [6]. The use of TiO_2 is not limited to polyolefin or extrusion technology; it has also been used to produce polyvinyl chloride (PVC)-based films using dimethylformamide (DMF) as a solvent. Two groups of polyvinyl chloride (PVC) films were obtained using an automated film scraping machine. The first group contained unmodified TiO_2, and the second group contained lanthanum ion-modified TiO_2 nanoparticles (La3+/TiO_2) by ball milling with the addition of lanthanum oxide (La2O3) at a rate of 0.6% and hydrochloric acid at a volumetric ratio of 1:1. The modified titanium dioxide increased the weight loss rate of the PVC films eightfold after exposure to ultraviolet radiation compared to the control sample and twofold compared to the unmodified titanium dioxide sample [7]. As explained in the US Food and Drug Administration's regulations (21 CFR 73.575), TiO_2 may be used as a coloring agent in food under specific conditions. The most important condition is that the weight percentage of TiO_2 in the food product should not exceed 1%. Additionally, the concentration of heavy metals, such as lead, arsenic, and antimony, in a solution resulting from boiling 10 grams of TiO_2 in 50 milliliters of 0.5 N hydrochloric acid for 15 minutes should not exceed the following limits: 10 parts per million (ppm) for lead, 1 ppm for arsenic, and 2 ppm for antimony. The method and materials used for packaging greatly affect the quality of milk and the rate at which vitamins, fats, and amino acids break down. The ability of the packaging materials to prevent light from entering the container is particularly important in reducing oxidation. Photooxidation continues in stores while milk is being stored and displayed, resulting in continuous loss of nutritional value, especially vitamins. This problem is exacerbated by stores that leave the lights on overnight, even after closing, to prevent theft. Studies have proven the effectiveness of using TiO_2 as an additive in packaging materials to improve the quality and extend the shelf life of dairy products such as cheese and yogurt. Dairy products were stored in polyethylene (PE)-based packaging with the addition of nano titanium dioxide for 11 days. Two different types of TiO_2 (0.1 g each) were prepared by adding 2 ml of anhydrous isopropyl alcohol. Then, ready-made polyethylene films were coated with the solutions and air-dried for ten minutes. Increasing the rutile content from 2.8% to 6.9% positively affected titanium dioxide's efficiency by enhancing its ability to control the rate at which the acid number rises and lipolysis occurs. The migration of TiO_2 from polyethylene-based packaging materials to which it has been added was also investigated. The results of the FTIR analysis proved that there were no traces of TiO_2 in the packaged milk products [8, 9]. In another study, TiO_2 nanoparticles exhibited antimicrobial properties when introduced into a polyethylene matrix via extrusion technology. These properties were enhanced upon exposure to ultraviolet (UV) radiation. The inhibition rate of polyethylene films containing TiO_2 was 89.3% for Escherichia coli (Gram-negative bacteria) and 95.2% for Staphylococcus aureus (Gram-positive bacteria) [10]. Based on previous studies, it is clear that developing packaging materials with antimicrobial and antioxidant properties is important for ensuring product quality and extending shelf life, particularly when TiO_2 is added. Further research is necessary to understand the unique properties of this additive and its effect on the polymer matrix and packaged food product specifications. This scientific article aims to address the challenges of food storage by developing packaging materials capable of slowing spoilage and preserving sensory properties. The research focuses on the functional properties of low-density polyethylene modified with TiO_2, such as mechanical, barrier, antimicrobial, and antioxidant properties, as well as their effect on stored milk quality.

EXPERIMENTAL

Materials

Low-density polyethylene (100% LDPE, melt flow index: 2 g/10 min at 2.16kg/190°C, manufactured by "SIBUR", Russia), TiO_2 (rutile type, manufactured by "Rushim", Russia, average particle size is 250 nm), ultra-pasteurized drinking milk (manufactured by "Northern Valley", Russia), phenolphthalein indicator (manufactured by "Moscow City Council of National Economy, Alkaloid Plant", Russia), potassium hydroxide (manufactured by "Oriental Chemical Industries", South Korea).

Sample preparation

Mixtures of polyethylene granules and different amounts of TiO_2 powder (0.5%, 1%, and 3% w/w) were prepared. The mixtures were mixed using a laboratory stirrer (Scientific Industries, USA).

Two types of single-screw extruders were used to obtain the samples with identical speed and temperature modes for extrusion processing (speed: 90 rpm; temperature settings: Zone 1 (T1): 110°C, Zone 2 (T2): 120°C, Zone 3 (T3): 130°C, Zone 4 (T4): 140°C). The first type is an extruder designed for producing strings (model 140/679, Inno Vert, Moscow, Russia) with a 16 mm screw diameter and an L/D ratio of 32. The extruder has an ultrasonic power of one kilowatt at a frequency of 22.4 kHz. The duration of ultrasonic treatment was 45 seconds. The second type is an extruder designed for producing films (model ISD551M21B, Inno Vert, Moscow, Russia). Its screw diameter is 12 mm, and its L/D ratio is 32. The steps for obtaining the samples are explained in Figure 1.

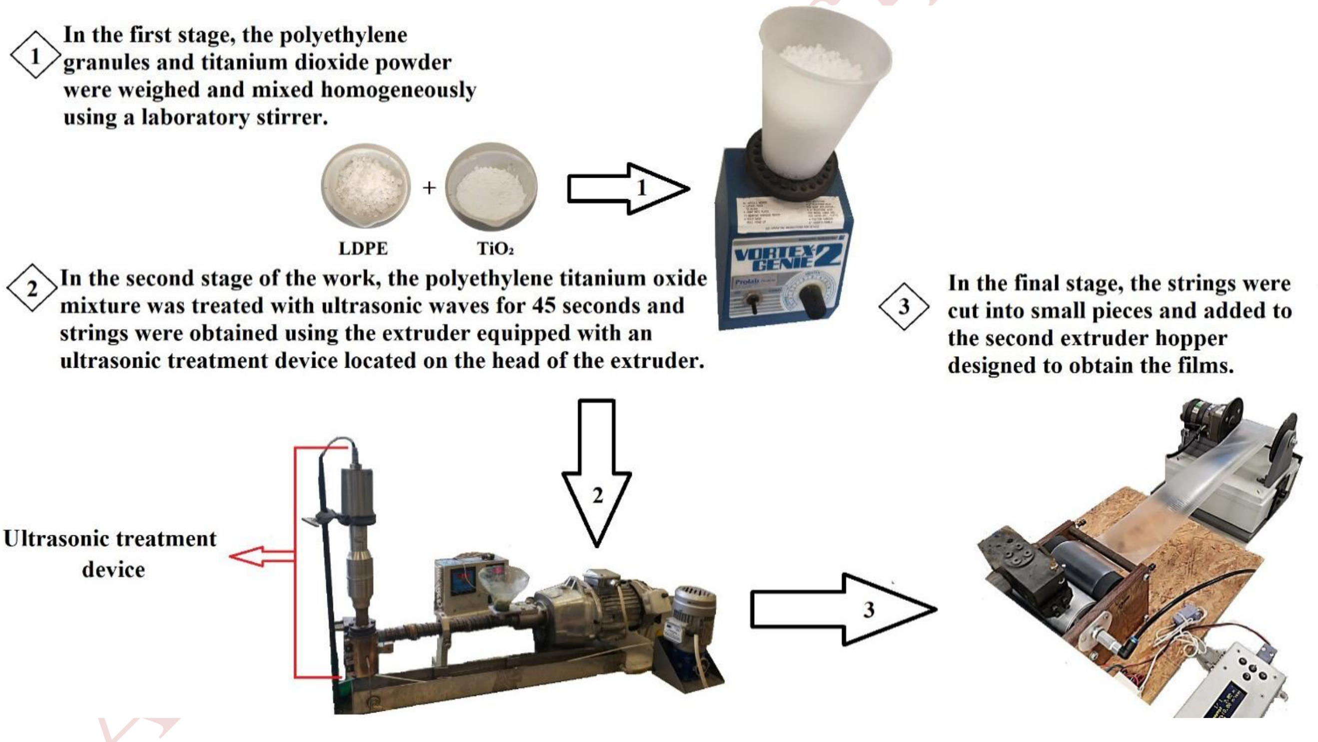

Figure 1. Schematic of the stages of obtaining polymer samples.

Sample characterizations

Determination of morphological properties

The morphological characteristics of the polyethylene samples were determined using a Bresser LCD 50x–2000x digital microscope (LCD Microscope V5.0, Gutenbergstrasse 2-46414 Rhede, Germany). The microscope lenses were set to 4x, 10x, and 40x.

FTIR spectroscopy analysis

The functional group of the produced composite is used to determine the structural configuration. The FTIR spectrometer (FTIR-990IN, Ins trument SN: ZKRMFIK0 O2, Tianjian Labor Scientific Ins trument Co., Ltd., China) was used to determine the functional groups. The working environment has a humidity level of 15-60% and a temperature of 30.5°C. There are two apodization triangles and the collection time is 20 minutes. The absorption spectra of the FTIR were collaborated between 4000 to 400 cm-1 wavenumber.

Determination of the physical and mechanical properties

This experiment was performed using a C610H automatic tensile testing machine, which was manufactured by Labthink Instruments Co., Ltd. in China. Physical and mechanical properties were assessed in accordance with GOST 14236, and parameters such as tensile strength (MPa) and elongation at break (%) were measured. Specimens measuring 10 x 80 mm in the transverse and longitudinal directions were prepared for the experiment.

Determination of water vapor transmission rate (WVTR)

The water vapor transmission rate of the resulting polymer materials with the addition of TiO_2 was determined using a Labthink water vapor transmission rate test system, model C360. The experiment was conducted according to the GOST 1037, under conditions of a temperature of 25±1 °C and a humidity of 10%. The test dishes were each filled with 25 milliliters of distilled water. Then, the polymer samples were cut into circular shapes to fit the dimensions of the dishes (Sample area 33 cm^2). Each dish was covered with a polyethylene sample and sealed tightly. The dishes were placed in the sealed apparatus chamber. The apparatus weighed the dishes automatically every 1.5 hours for 24 hours and calculated the water vapor permeability of the samples using the following formula:

$$\mathrm{WVTR} = \Delta \mathrm{m}\left(\mathrm{S}/\mathrm{T}\right) \quad (1)$$

where:

WVTR: Water vapor transmission rate (g/m^2.24h).

Δm : The change in mass of the test dish measured by the high-precision internal balance (gram).

S: The exposed test area of the specimen (m^2).

T: The measurement time interval (24 hours).

Determination of antimicrobial properties

The disk method (MUK 4.2.1890–04) was employed to ascertain the antimicrobial properties of the samples. Sabouraud's nutrient medium (for fungi and yeast) and meat-peptone agar (for bacteria) were prepared and subsequently sterilized in an autoclave for 15 minutes at a temperature of 121°C and a pressure of 0.15 MPa. The medium was subsequently transferred to sterile Petri dishes, into which a suspension of gram-positive (*Bacillus subtilis ATCC 6633*) and gram-negative (*Escherichia coli M17*) bacteria, fungi (*Aspergillus Niger F-894*), and yeast (*Candida albicans Y-3108*) in a volume of 0.1 ml was added and distributed evenly across the entire surface with a spatula. Subsequently, the samples were placed in a thermostat at a temperature of 29 ± 1°C for the fungi and 36 ± 1°C for the bacteria. The microbial inhibition zone was measured after 1, 3, and 5 days of incubation.

Determination of acid number

An acid number analysis was conducted prior to packaging. A rectangular milk package was then prepared using 100 x 85 mm film samples. A quantity of 20 milliliters of milk was incorporated into the package and subsequently sealed using a sealing machine manufactured by Hanato in South Korea. The milk package was subsequently stored in a refrigerator maintained at a temperature of 4°C for a period of 10 days. Subsequent to this interval, a re-evaluation of the milk was conducted. The titratable acidity of milk is expressed in Terner degrees (°T). Terner degrees express the volume in cubic centimeters (mL) of 0.1 N potassium hydroxide (KOH) required to neutralize 100 cm^3 of milk. Acidity determination is regulated by GOST 3624-92. The determination of acidity in milk involves the neutralization (titration) of acidic salts, proteins, free acids, and other acidic compounds present in the milk sample with an alkali solution. The presence of phenolphthalein as an indicator is also a crucial component of the titration process. For the experiment, a 0.1 M potassium hydroxide solution and a 1% alcohol solution of phenolphthalein were prepared. The determination procedure is outlined as follows: In a suitable container, 10 cubic centimeters of milk at 20°C was measured using a conical pipette. To this volume was added 20 cubic centimeters of distilled water and three drops of phenolphthalein. The mixture is then thoroughly mixed and titrated with a sodium hydroxide solution until a faint pink color appears, corresponding to the control color standard. This color change must not disappear within one minute.

RESULTS AND DISCUSSION

Appearance of films obtained on the basis of low-density polyethylene.

A comparison of the external appearance of the samples reveals that the transparency of the polyethylene films decreases with increasing TiO_2 concentration. Conversely, all films obtained with the addition of TiO_2 are bright white (Figure 2). Upon closer inspection, the direct effect of ultrasound on the distribution of TiO_2 within the polyethylene matrix becomes apparent. The white color is distributed uniformly and evenly across the entire film surface. In contrast, a more pronounced white line is observed in the center of films not treated with ultrasound. These results are consistent with previous studies [11]. The thicknesses of the unultrasonicated polyethylene samples with added TiO_2 at concentrations of 0%, 0.5%, 1%, and 3% were 125±3 µm, 120±3 µm, 120±3 µm, and 115±4 µm, respectively. The thicknesses of ultrasonicated polyethylene samples with the same concentrations of TiO_2 were 120±3 µm, 120±3 µm, 115±4 µm, and 110±3 µm, respectively.

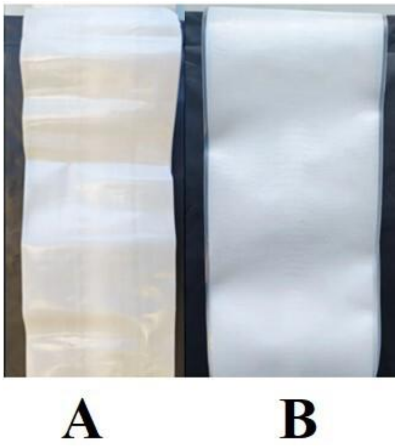

Figure 2. The external appearance of the obtained films: A) LDPE film with TiO_2 3% without ultrasound treatment, B) LDPE film with TiO_2 3% with ultrasound treatment.

Morphological properties

To better understand how TiO_2 is distributed within the polyethylene matrix, the obtained films were studied under a microscope at 4x, 10x, and 40x magnification (Figure 3). In untreated polyethylene samples, the TiO_2 is distributed as white dots that converge to form straight, parallel lines. Conversely, ultrasound disperses these white dots, making them less clumped and more uniformly spaced. This more uniform distribution increases the effective specific surface area, which is expected to improve the functional performance of polyethylene films, including antimicrobial activity and photooxidation resistance.

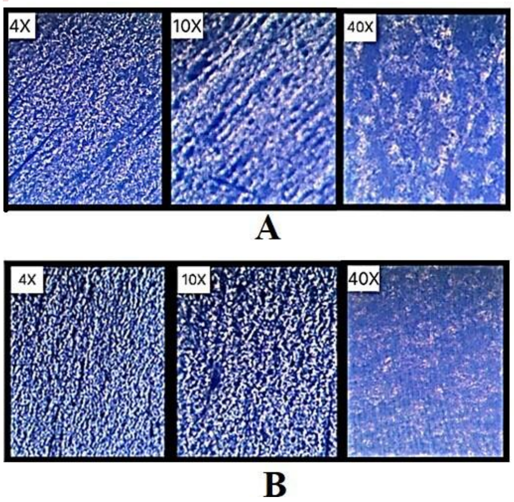

Figure 3. The structure of the obtained materials based on polyethylene under a microscope. Note: A) Sample with 3% TiO_2 without ultrasound, B) Sample with 3% TiO_2 with ultrasound.

FTIR spectroscopy

As shown in Figure 4, the addition of TiO_2 at different concentrations (0.5%, 1%, and 3%) did not affect the infrared absorbance of LDPE. Al-Amin et al. reported that the characteristic peak of infrared absorption of TiO_2 is 690 cm^{-1}[12]. However, the results in Figure 4 did not align with previous reports, as no distinct peaks were identified for titanium dioxide's of infrared radiation. This discrepancy can be explained by the high purity and crystallinity of the TiO_2 used in the study. These properties transform TiO_2 from an infrared absorber to an infrared emitter. Furthermore, sources indicate that rutile and anatase TiO_2 have energy gaps of 3.0–3.2 electron volts. Consequently, some types of radiation with lower energies, such as near-infrared radiation, are unable to induce electron transitions between energy bands [2].

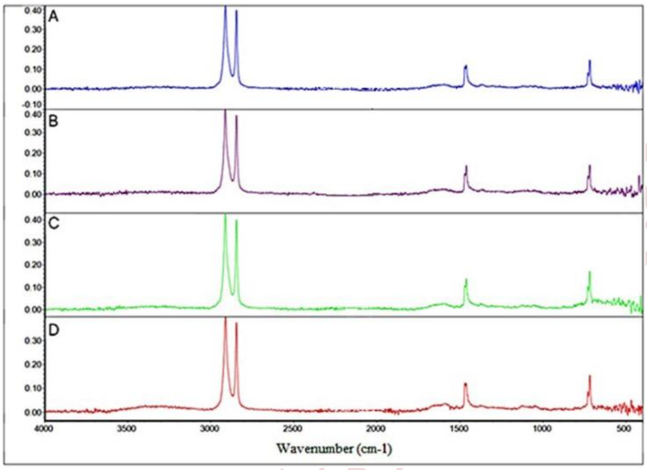

Figure 4. Results of FTIR spectroscopy. Note: A) Sample with 0.5% TiO_2 without ultrasound, B) Sample with 1% TiO_2 without ultrasound, C) Sample with 3% TiO_2 without ultrasound, D) Control sample without ultrasound.

As with TiO_2, ultrasound did not affect the infrared absorbance of polyethylene, as can be seen by comparing all the samples in Figure 5. Previous theoretical and experimental studies have shown that ultrasound only affects the infrared absorption of polyethylene when high-frequency ultrasound is used. This is because new free radicals and functional groups are generated. Furthermore, the duration of ultrasound exposure significantly affects the appearance of new absorption peaks [13]. Therefore, the absence of new absorption regions can be explained by the brief exposure to ultrasound waves, which did not exceed 45 seconds despite an ultrasonic power of 1 kW.

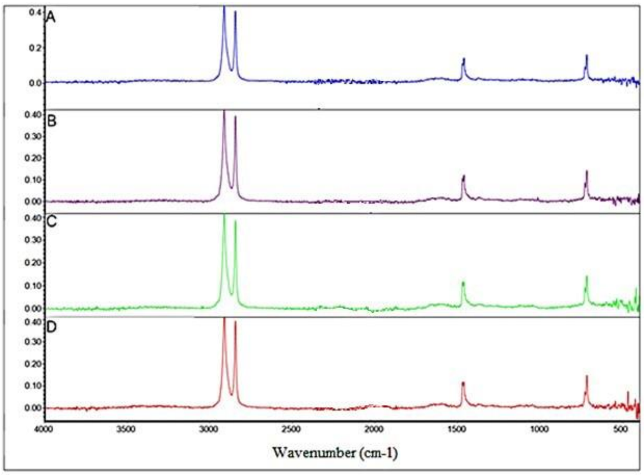

Figure 5. Results of FTIR spectroscopy. Note: A) Sample with 0.5% TiO_2 with ultrasound, B) Sample with 1% TiO_2 with ultrasound, C) Sample with 3% TiO_2 with ultrasound, D) Control sample with ultrasound.

Physical and mechanical properties

The experiment showed that increasing the concentration of TiO_2 increases the strength of polyethylene in both the longitudinal and transverse directions, reaching its maximum value at a concentration of 0.5%, while the strength gradually decreases with increasing concentrations of 1% and 3%. Longitudinal strength increased by approximately 11.2% at a 0.5% TiO_2 concentration compared to the control sample. The increase in transverse strength at the same concentration was approximately 42%. The experimental results also demonstrated that using ultrasound positively affects strength. The increase in longitudinal strength was 17.5% at a concentration of 0.5% TiO_2, while the increase in transverse strength was 14.3% at the same concentration (Figure 6). Additionally, it was observed that increasing the concentration of TiO_2 reduces the polyethylene's elongation at break, while using ultrasound increases it. Increasing the TiO_2 concentration to 3% decreased longitudinal elongation at break by 50.1% and transverse elongation at break by 46.7%, compared to the control sample. Ultrasound waves led to increase in elongation at break of 39.4% in the longitudinal direction and 43.2% in the transverse direction at a concentration of 3% (Figure 6). These results are consistent with those of previous studies, although the TiO_2 used in this study is not classified as a nanomaterial [11, 14].

Using ultrasonic waves reduced the elastic modulus by 11% in the longitudinal direction and 10.6% in the transverse direction at a 3% TiO_2 concentration. These results also clearly demonstrate the effect of TiO_2 on the elastic modulus of polymeric materials. Increasing the TiO_2 concentration resulted in increases of 58.2% and 63.9% in the longitudinal and transverse directions, respectively, at a 3% TiO_2 concentration (Figure 6). These results are largely consistent with those of previous research [5].

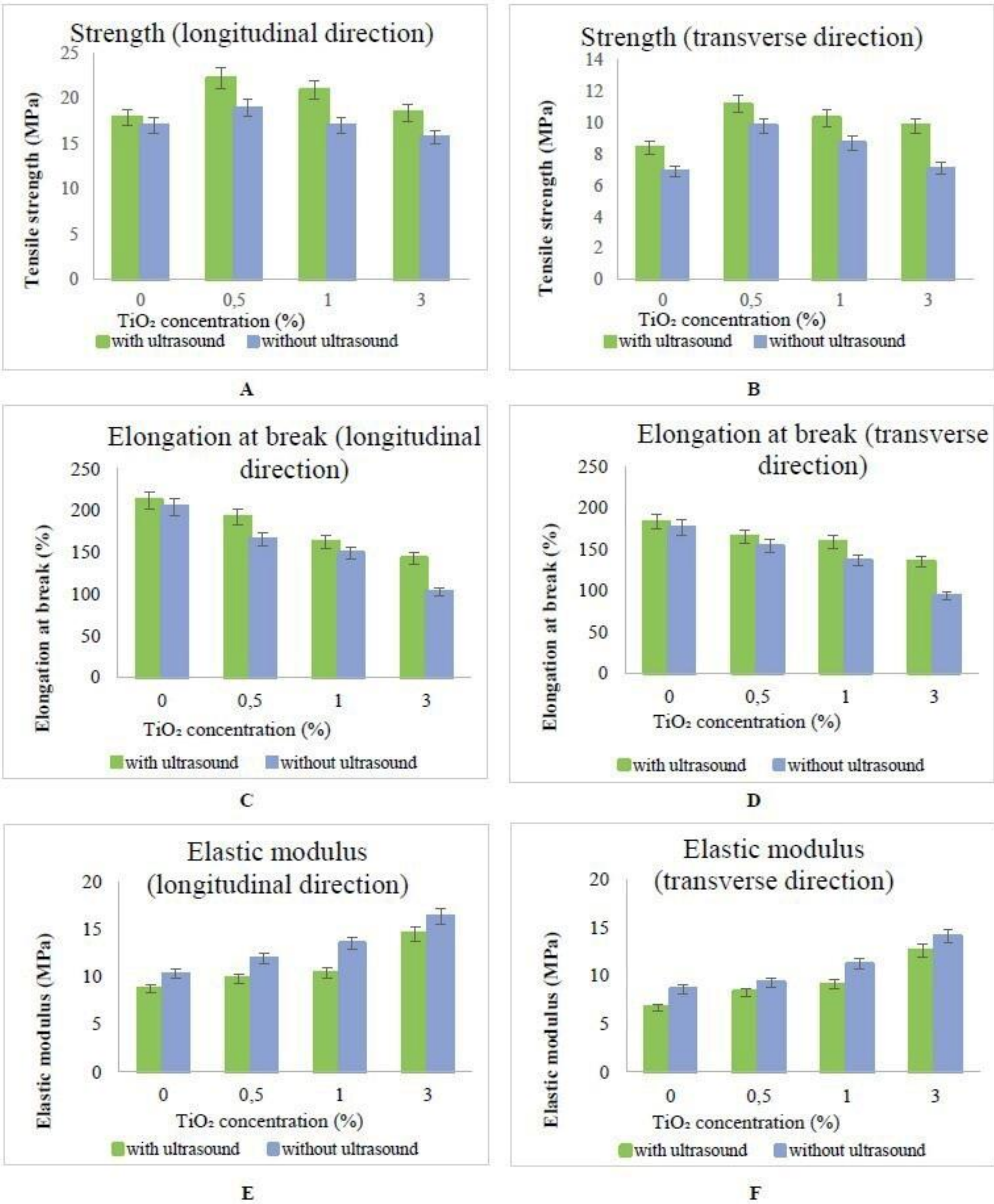

Figure 6. Results of physical and mechanical characteristics: A) Strength of LDPE with TiO_2 (longitudinal direction), B) Strength of LDPE with TiO_2 (transverse direction), C) Elongation at break of LDPE with TiO_2 (longitudinal direction), D) Elongation at break of LDPE with TiO_2 (transverse direction), E) Elastic modulus of LDPE with TiO_2 (longitudinal direction), F) Elastic modulus of LDPE with TiO_2 (transverse direction).

Water vapor transmission rate (WVTR)

Figure 7 shows that increasing the concentration of TiO_2 led to a higher water vapor transmission rate, while using ultrasonic wave technology led to a decrease in it. It is worth mentioning that sound waves did not affect the water vapor transmission rate of pure polyethylene samples without TiO_2. The effect was limited to samples with added TiO_2. The average WVTR values (g/m².24h) for untreated polyethylene samples with added TiO_2 at 0%, 0.5%, 1%, and 3% concentrations were 1.8, 2.9, 3.7, and 6.7, respectively.

For polyethylene samples treated with ultrasound at the same concentrations, the values were 1.8, 2.6, 3.3, and 5.5, respectively.

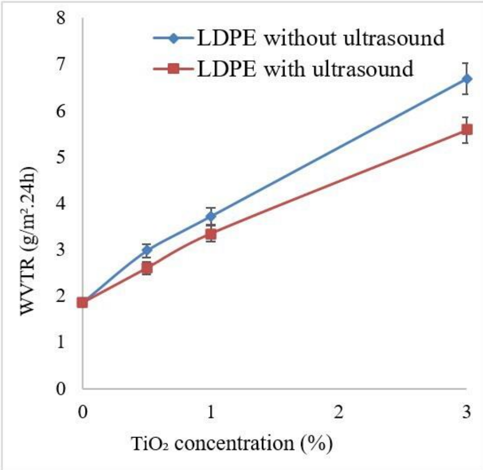

Figure 7. Results of determining the water vapor transmission rate (WVTR) of polyethylene samples.

These results are consistent with previous studies that reported an increase in the water vapor transmission rate of polyethylene films as the TiO_2 concentration increased. One possible explanation for this phenomenon is agglomeration of the TiO_2 when it is added at high concentrations, creating gaps in the polymer matrix. These gaps increase the water vapor transmission rate through the polymer matrix. Conversely, films treated with ultrasonic waves exhibit a decreased water vapor transmission rate because the ultrasonic energy reduces the degree of TiO_2 agglomeration and distributes it uniformly. This fills the gaps and reduces the water vapor transmission rate [15].

Antimicrobial properties

The results showed that TiO_2 could inhibit the activity of microorganisms, though to varying degrees (see Figures 8, 9, 10, and 11). The inhibition zone was 0.8 ± 0.05 mm for untreated polyethylene samples with ultrasound and TiO_2 concentrations of 1% and 3% in the *Bacillus subtilis* relationship. However, the inhibition zone was smaller for the untreated polyethylene samples using ultrasound in the *Escherichia coli* relationship than for the same samples at the same concentrations in the *Bacillus subtilis* relationship. The inhibition zone reached 0.3 ± 0.05 mm at 3% and 1% TiO_2 concentrations. Using ultrasonic wave technology or increasing the TiO_2 concentration from 1% to 3% did not increase the diameter of the inhibition zone for Bacillus subtilis and *Escherichia coli* bacteria. The microbial inhibition zone does not increase despite the increased TiO_2 concentration because TiO_2 is distributed more in the center of the film and does not migrate to the surface, which is in direct contact with living organisms. This leads to the formation of the inhibition zone. Additionally, the rigidity of polyethylene prevents TiO_2 from migrating from the center of the film to the outer surface. Therefore, increasing the concentration of TiO_2 will not lead to an increase in the inhibition zone [16]. Therefore, it can be concluded that the 45-second ultrasonic treatment time was insufficient to achieve a more homogeneous distribution of TiO_2 within the resulting polyethylene films to inhibit the growth of these microorganisms.

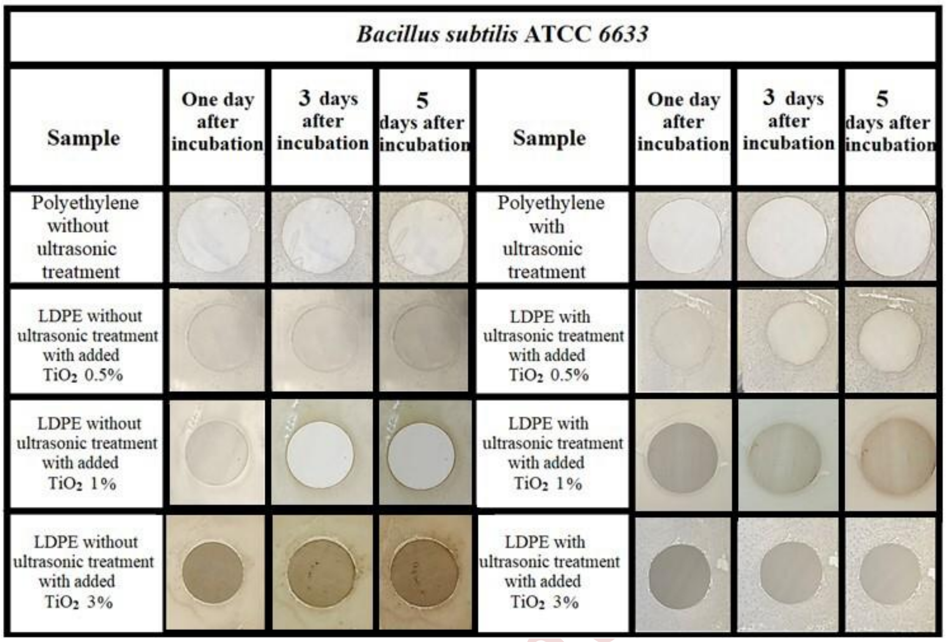

Figure 8. Results of determining antimicrobial properties in the *Bacillus subtilis* relationship.

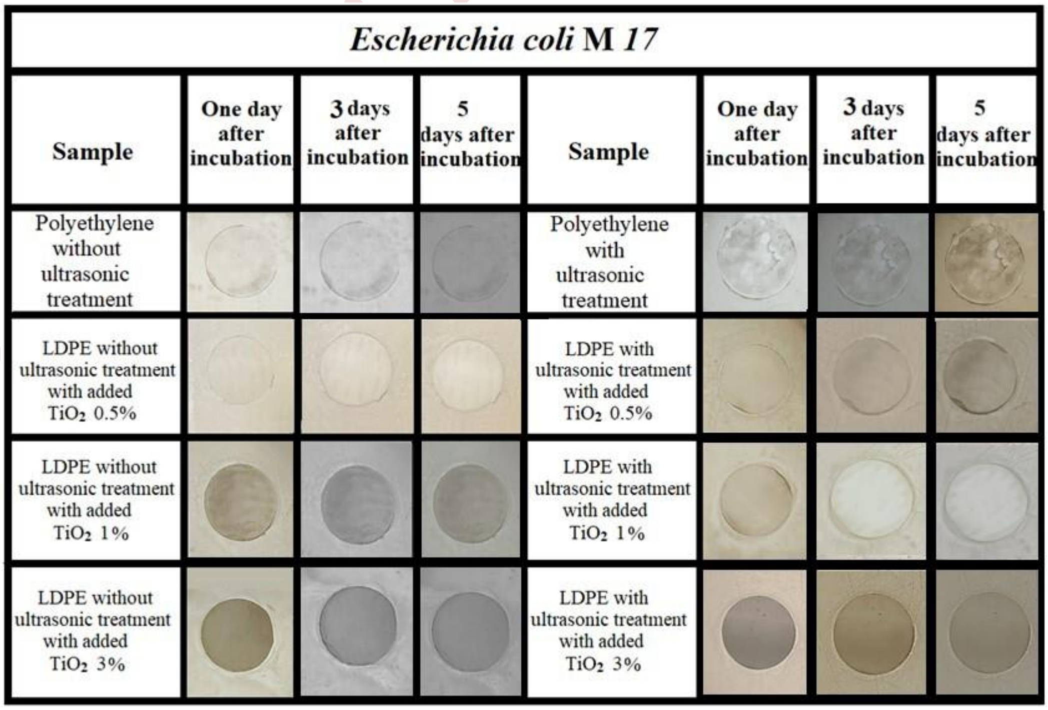

Figure 9. Results of determining antimicrobial properties in the *Escherichia coli* relationship.

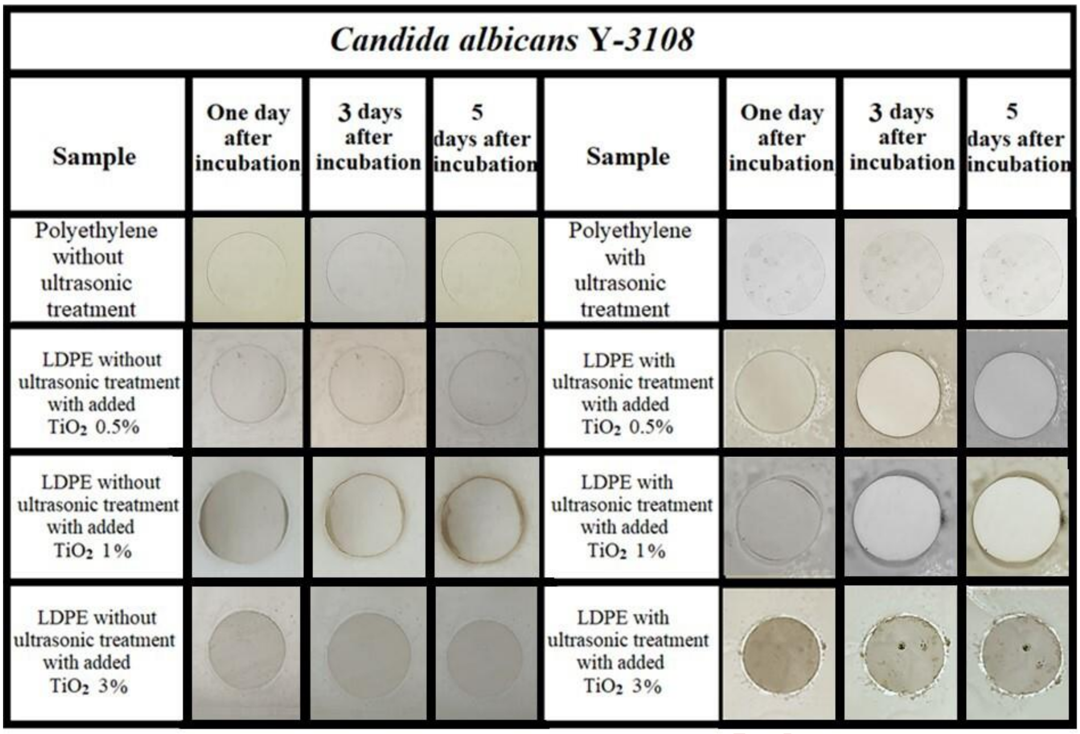

Figure 10. Results of determining antimicrobial properties in the *Candida albicans* relationship.

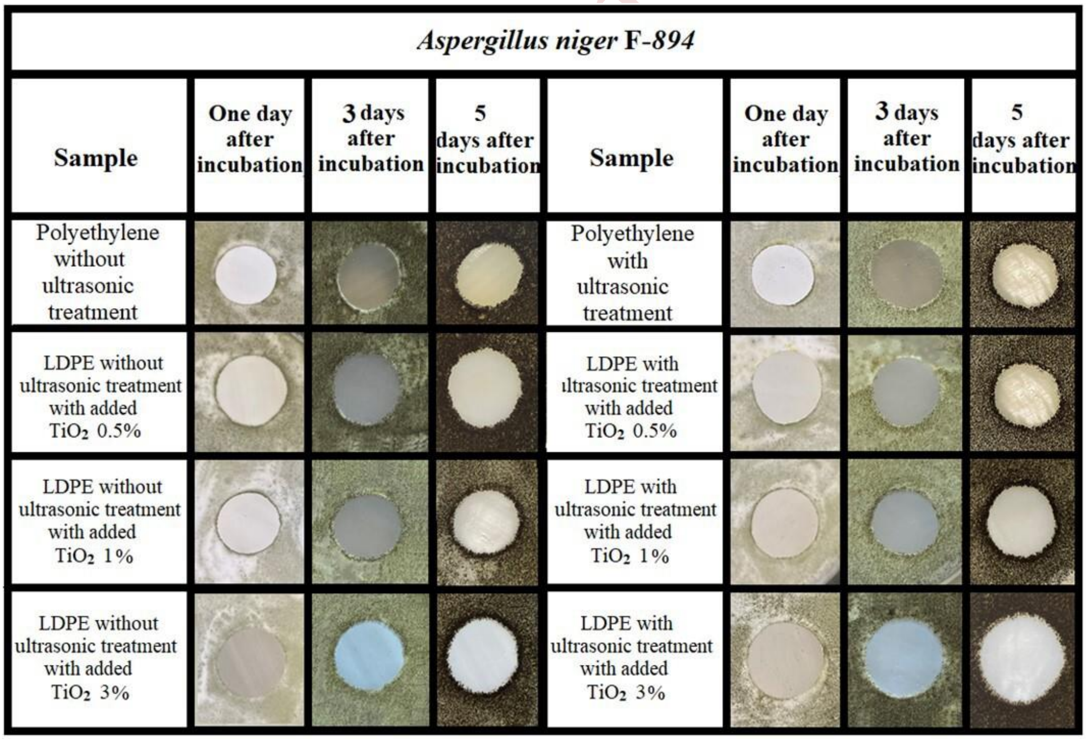

Figure 11. Results of determining antimicrobial properties in the *Aspergillus Niger* relationship.

No inhibition zones were observed around untreated polyethylene samples, except at a 3% concentration of TiO_2 in relation to *Candida albicans*, where the inhibition zone reached 0.3 ± 0.05 mm. Furthermore, an inhibition zone of 1±0.05 mm was determined for a polyethylene sample treated with ultrasound at a 3% TiO_2 concentration. This is attributed to ultrasonic waves' ability to remove clumps and achieve a more homogeneous distribution of TiO_2 within the polyethylene matrix. This increases the effective surface area in contact with microbes. Regarding the assessment of the antimicrobial properties of the *Aspergillus niger* relationship, an inhibition zone was determined for the untreated polyethylene sample treated with ultrasound and a 3% concentration of TiO_2 (1.3 ± 0.05 mm). Notably, ultrasound treatment increased the inhibition zone by 0.5 mm, raising it to 1.8 ± 0.05 mm at a 3% concentration.

Overall, these results align with previous research indicating that TiO_2 exhibits antimicrobial properties against Gram-positive bacteria (*Bacillus subtilis*), Gram-negative bacteria (*Escherichia coli*), yeasts (*Candida albicans*), and fungi (*Aspergillus niger*) [17- 20].

Acid number

As Figure 12 shows, the addition of TiO_2 directly affects the acid number of milk stored for 10 days. As the concentration of TiO_2 increases, the acid number decreases significantly. The acid number of milk samples stored at 4 °C in untreated polyethylene packaging, to which TiO_2 was added at concentrations of 0%, 0.5%, 1%, and 3% by ultrasound, reached the following values: 3°T, 2°T, 1.8°T, and 0.8°T, respectively. The acid number of milk samples packaged in ultrasonically treated polyethylene containers decreased by 50% at a 0.5% concentration of TiO_2, by about 55.5% at a 1% concentration, and by about 37.5% at a 3% concentration, compared to samples not treated with ultrasound at the same concentrations of TiO_2.

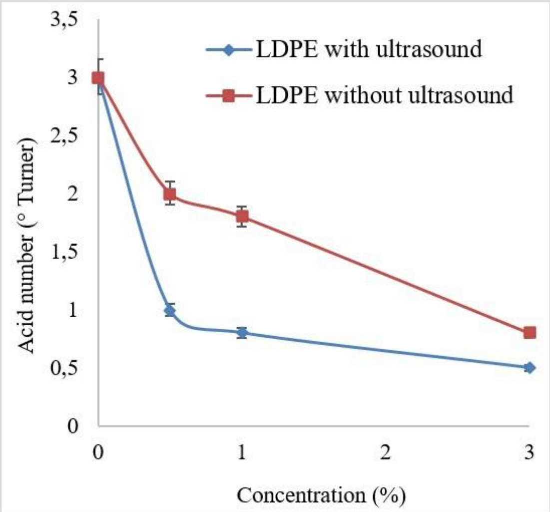

Figure 12. Results of determining the acid number of packaged milk in polyethylene samples.

The potential of ultrasound to reduce the acid number can be attributed to the impact of these waves on the dispersion and homogeneous distribution of TiO_2 within the polymer matrix. This effect increases the specific surface area of TiO_2, thereby enhancing its capacity to inhibit photo-oxidation. Furthermore, previous reference studies have indicated that TiO_2, due to its ability to inhibit certain types of bacteria that convert sugars into lactic acid (or other organic acids), curbs the rise in the acid number of the food product [9].

CONCLUSION

In the initial phase of the study, two types of low-density polyethylene films were obtained by extrusion technology with the addition of TiO_2 at concentrations of 0, 0.5, 1, and 3%. The utilization of ultrasonic wave technology was instrumental in achieving enhanced homogeneity in the distribution of TiO_2 within the polyethylene matrix. In the subsequent phase of the research, the morphological, physical, mechanical, and chemical properties, as well as the antimicrobial properties, of the prepared films were studied to determine the effect of two primary factors: first, the addition of TiO_2 to the polyethylene matrix, and second, the effect of ultrasonic treatment on these properties. To ascertain the efficacy of the fabricated membranes, an independent experiment was conducted to determine their effect on the acid number of milk stored for ten days.

The study yielded the following significant findings:

The findings of the FTIR spectroscopy analysis demonstrated that the incorporation of TiO_2 and the utilization of ultrasound waves did not alter the absorption of infrared radiation by polyethylene. Furthermore, these processes did not result in the formation of functional groups or the emergence of new bonds. This phenomenon can be attributed to the exceptional purity of the TiO_2 utilized in the study. The purity of the TiO_2 is such that it transforms into a scattering of radiation rather than absorbing it, a property that can be attributed to its crystalline structure. The results showed that the optimal concentration of TiO_2 in the polymer matrix to achieve the best tensile strength is 0.5%. Additionally, using ultrasound was shown to increase tensile strength and elongation at break but decrease the elastic modulus. The water vapor transmission rate (WVTR) of polyethylene membranes was found to be adversely impacted by elevated TiO_2 concentrations, exhibiting a 3.6-fold increase at a TiO_2 concentration of 3%. The utilization of ultrasonic waves has been demonstrated to result in a reduction in water vapor transmission rate (WVTR). This indicator has decreased by 16.6% for polyethylene films with 3% TiO_2 added and ultrasonically treated compared to those that were not treated at the same concentration of TiO_2. However, the application of ultrasound did not result in an augmentation of the inhibition zone in TiO_2-modified polyethylene samples when evaluated in the context of the Bacillus subtilis and Escherichia coli relationship. Additionally, the TiO_2 concentration was increased from 1% to 3%, yet the inhibition zone remained unaltered. In light of these findings, it is advised that future research extend the ultrasound exposure time beyond 45 seconds to ensure optimal efficacy in the studied membranes. In contrast, the utilization of ultrasound in the treatment of polyethylene films resulted in the emergence of inhibition zones surrounding the polymer samples in Candida albicans and

Aspergillus Niger. These zones were either absent or exhibited minimal visibility in polyethylene samples that were not subjected to ultrasound treatment. The application of ultrasound technology resulted in a significant reduction in the acid number of stored milk, as evidenced by the obtained results. The acid number exhibited a 50% decrease at a concentration of 0.5% TiO_2 compared to untreated samples at the same TiO_2 concentration.

ABBRIVIATIONS

Abbreviation	Full name
LDPE	Low-density polyethylene
TiO_2	Titanium dioxide

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