

ORIGINAL PAPER

Evaluation of the effect of dried marjoram powder on the technological properties of LDPE

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ABSTRACT

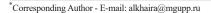
The aim of this study is to encourage a shift in industry towards using natural, biodegradable materials with antimicrobial properties to produce polymeric materials that can be used effectively in areas such as food and pharmaceutical packaging. In this study, polymeric films based on low-density polyethylene (LDPE) were produced using extrusion technology with the addition of marjoram powder at concentrations of 1%, 3% and 5%. The second phase of the study involved determining the technological properties of the obtained films, including their antimicrobial, barrier, physical, and mechanical properties. The results of the research showed that polyethylene samples supplemented with marjoram powder exhibited antimicrobial properties against Bacillus subtilis and Candida albicans at concentrations of 3% and 5%. However, the same samples did not exhibit any antimicrobial properties against Escherichia coli and Aspergillus Niger at any concentration. Polyethylene samples with a 3% concentration demonstrated the greatest tensile strength in both longitudinal and transverse directions. However, as the marjoram concentration increased, the elongation-at-break of the samples declined. Polyethylene films recorded the lowest elongation-at-break values at a 5% concentration in both directions. Water vapor permeability of the films also increased with marjoram powder concentration; it increased by 2.8 times at 3% compared to the control sample. Polyelefins J (2025) 12: 273-279

Keywords: Polymer; marjoram; water vapor permeability; antimicrobial and mechanical properties.

INTRODUCTION

Governments in developed countries are currently focusing on supporting and implementing policies that protect the environment from traditional plastic packaging waste, which decomposes slowly in nature. In light of severe climate change, manifested by severe floods, droughts and declining rainfall rates, environmental conservation has become an urgent necessity [1]. Although scientists' efforts have greatly contributed to the development of biodegradable packaging materials, the main problem with these

materials is their poor physical and mechanical properties, as well as their poor water and barrier resistance [2]. In addition, the global food industry suffers from the use of synthetic preservatives, which have a negative impact on human health and reduce the reliability of the food they are added to [3]. Due to the presence of phenolic compounds such as thymol and terpinen-4-ol, marjoram has antifungal properties against Aspergillus Niger and anti-yeast properties against Candida albicans. It also has





antibacterial properties against Escherichia coli and Staphylococcus aureus [4,5,6,7,8]. Previous research has used marjoram to create pectin-based films. These films were prepared using nanoemulsions and Pickering emulsions loaded with marjoram essential oil. The concentration of the nanocarriers incorporated into the pectin film formulation was 2.5%, 5% or 7.5% by weight. The antioxidant activity of films containing Pickering emulsions was significantly lower (p < 0.05) than that of films incorporating nanoemulsions loaded with marjoram essential oil. Films containing Pickering emulsions exhibited good mechanical properties and a high water barrier due to their dense, impermeable structure. The inhibition zone for S. aureus was 16.00 ± 0.97 mm for samples containing Pickering emulsions, and 14.32 ± 1.02 mm for samples containing nanoemulsions. For E. coli, the inhibition zone was 11.58 ± 1.15 mm for samples containing Pickering emulsions and 12.65 ± 0.94 mm for samples containing nanoemulsions [9]. Anti-hemorrhage bandages with antibacterial and antioxidant properties were developed using marjoram extract and kaolin in polyvinyl alcohol-based sponges. Two sets of samples were created. In the first set, polyvinyl alcohol was used along with marjoram extract at concentrations of 0.1%, 0.25%, and 0.5%. The second set was made using polyvinyl alcohol with marjoram extract at 0.5% and kaolin at 0.1%, 0.25% and 0.5% concentrations. The sample preparation technique involves forming a PVA hydrogel through periodic freezing and thawing to create crystalline aggregates. Adding marjoram extract at ratios of 0.1%, 0.25%, and 0.5% to PVA increased the inhibition rate of B. cereus by 31%, 65%, and 85%, respectively. Adding marjoram extract at the same proportions to PVA increased the inhibition rate of E. coli by 63%, 87% and 90%, respectively. Adding marjoram and kaolin to polyvinyl acetate increased the pore size of the sponge, thereby enhancing its ability to absorb water and control bleeding [10].

Based on the results of previous research, no studies were found that used dried marjoram leaves and stems mixed with molten low-density polyethylene (LDPE) via an extruder. This research is important because it exploits the antimicrobial properties of dried marjoram without the disadvantages associated with using essential oils. One of the most significant challenges when using essential oils to create polymers with antimicrobial properties is their degradation. They are volatile in air and degrade rapidly in the presence

of light and oxygen, which has a significant impact on their chemical, antioxidant and antimicrobial properties [11]. In many cases, the incompatibility between the polymer and the essential oil leads to the formation of heterogeneous films with holes and irregular distribution of the essential oil in the polymer matrix [12]. In addition, controlling the migration of essential oils and releasing appropriate quantities over regular time periods from the polymer matrix into the packaged product is a difficult challenge due to the potential harm it can cause to consumer health [13, 14]. The physical and mechanical properties of polymer films are also negatively affected by essential oils [15].

This paper presents a study of films based on low-density polyethylene (LDPE) supplemented with marjoram powder at concentrations of 1%, 3% and 5%, obtained using centrifugation technology. The effect of the marjoram powder on the films' antimicrobial, physico-mechanical, and barrier properties was studied.

EXPERIMENTAL

Materials

Low-density polyethylene (100% LDPE, melt flow index: 2 g/10 min at 2.16 kg/190°C) manufactured by Tomskneftekhim LLC, Russia 634067, Tomsk, Kuzovlevsky tarkt 2/202, and marjoram (IP Stolbova AV) were used for the synthesis of the composite.

Sample preparation

Production of polymer films

The films were developed using the single screw extruder model: ISD551M21B, INNO VERT, Moscow, Russia. The single screw speed in the extruder is 90 rpm. Temperature modes for material processing by extrusion: 1 zone (T1) = 160°C, 2 zone (T2) = 170°C, 3 zone (T3) = 180°C, 4 zone (extrusion 5 head) (T4) = 190°C. After all parameters were achieved, LDPE and marjoram were added to the feed section. 100 g of LDPE pellets containing 1%, 3%, and 5% (w/w) of marjoram were examined, respectively. Pure LDPE was used as a control.

Sample characterizations

Determination of antimicrobial properties

The method is regulated by the MUK 4.2.1890–04 document and is used for Bacillus subtilis ATCC 6633,



Escherichia coli M17, Candida albicans Y-3108, and Aspergillus Niger F-894 microorganisms. Samples of the polymer material were cut into rounds with a diameter of 2 cm and cleaned of external contaminants. 0.1 ml of the microorganism suspension was added to Petri dishes containing an agar nutrient medium (Sabouraud for fungi and meat-peptone agar (MPA) for bacteria), distributed over the surface using a Drigalski spatula and then the purified polymer material samples were placed on the inoculated surface. The closed Petri dishes containing the samples were then incubated at temperatures of $37 \pm 1^{\circ}$ C (for B. subtilis and E. coli) and $28 \pm 1^{\circ}$ C (for A. Niger and C. albicans). The zone of microbial inhibition was measured visually after 1 day for bacteria and 3 days for fungi.

Determination of mechanical properties

The tensile strength and elongation-at-break, two of the physical and mechanical properties of low-density polyethylene (LDPE) films, were determined using a PM50 at 100 mm/min. Ten replicate cuts were made in the longitudinal and transverse extrusion directions from the center of the films. The samples were 10 mm width and 50 mm length.

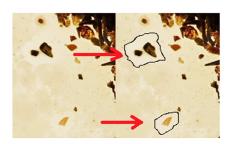
Determination of water vapor transmission rate (WVTR) The determination of the WVTR of polymer materials was carried out using the "C360" device by GOST GB1037. The studies were carried out at a temperature of 38°C and a humidity of 10%.

RESULTS AND DISCUSSION

Antimicrobial properties

In the first stage of antimicrobial properties testing, only the plant parts of the marjoram (excluding the low-density polyethylene) were tested. Powdered plant parts were added to a Petri dish containing a sterile medium and a microbial suspension. The dishes were then incubated at the optimal temperature for growth of each species of microbe. The results showed that the leaf parts of the marjoram plant exhibited antimicrobial properties against Bacillus subtilis and Candida albicans, with a clear microbial inhibition zone observed (see Figure 1). The arrows in the images (Bacillus subtilis) and (Candida albicans) indicate the inhibition zones (zone of microbial inhibition is the clear, sterile area surrounding an antimicrobial agent on an agar plate where bacterial or fungal growth is prevented). However, the plant parts did not exhibit antimicrobial properties against Escherichia coli and Aspergillus Niger, as the zone of microbial inhibition was not identified. On the contrary, the fungus was able to grow on the powdered surface of the marjoram plant (Figure 1). The study's results are inconsistent with previous studies on marjoram's antibacterial properties against Escherichia coli and Aspergillus Niger. This may be due to improper storage of marjoram, exposure to high temperatures and humidity, and oxidation processes.

In the second stage, the antimicrobial properties of low-density polyethylene (LDPE) films supplemented



Bacillus subtilis



Escherichia coli



Candida albicans



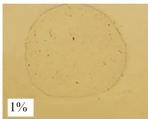
Aspergillus niger Figure 1. Antimicrobial properties of dried marjoram leaves and stems.

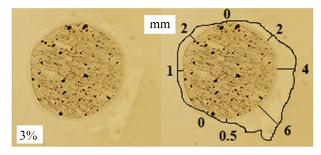
Table 1. Antimicrobial properties of low-density polyethylene samples with the addition of marjoram powder.

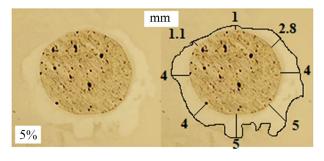
Polymer films based on LDPE	Average values of the microorganism suppression zone (mm)							
	Bacillus subtil		Escherichia coli		Candida albicans		Aspergillus Niger	
	24 hours of	10 days of	24 hours of	10 days of	24 hours of	10 days of	24 hours of	10 days of
	incubation	incubation	incubation	incubation	incubation	incubation	incubation	incubation
LDPE	0	0	0	0	0	0	0	0
LDPE + 1% M	0	0	0	0	0	0	0	0
LDPE + 3% M	1.94	0.75	0	0	0.75	0.75	0	0
LDPE + 5% M	3.36	1.7	0	0	0.86	0.86	0	0

with marjoram leaf powder were tested against bacteria and fungi using MUK 4.2.1890-04. The experiment results for LDPE films containing 1%, 3% and 5% marjoram leaf and stem powder were consistent with the initial test results for the additive. A microbial inhibition zone was only observed for Bacillus subtilis and Candida albicans at concentrations of 3% and 5%. As the additive concentration increased, so did the microbial inhibition zone. After 10 days of incubation, the zone of microbial inhibition against









Bacillus subtitis

Figure 2. Antimicrobial properties of low-density polyethylene samples with the addition of marjoram powder at concentrations of 1, 3 and 5% in the Bacillus subtilis relationship.

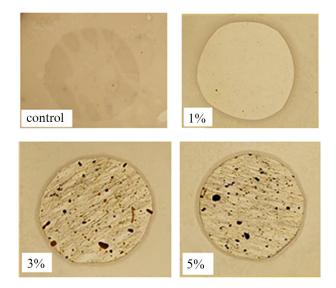
Bacillus subtilis decreased compared to the results of incubation after 24 hours. In contrast, the zone of microbial inhibition against Candida albicans did not change after 10 days (Table 1 and Figures 2,3,4 and 5).

Physical and mechanical characteristics

The 3% concentration film showed the best tensile strength results in both longitudinal and transverse directions, while the tensile strength at the 5% concentration decreased by 35.06% in the longitudinal direction and by 39.79% in the transverse direction compared to the control sample (Figure 6).

The addition of dried marjoram powder significantly deteriorated the elongation-at-break, which decreased with increasing additive concentration in both longitudinal and transverse directions compared to the control sample (Figure 7).

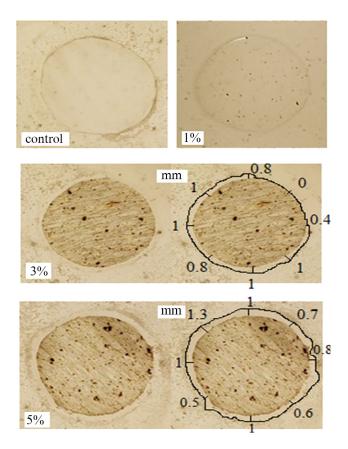
The results of our study are consistent with the results of previous studies that used plant extracts as



Escherichia coli

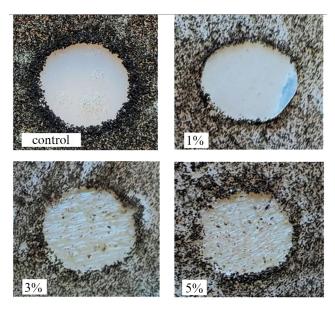
Figure 3. Antimicrobial properties of low-density polyethylene samples with the addition of marjoram powder at concentrations of 1, 3 and 5% in the Escherichia coli relationship.





Candida albicans

Figure 4. Antimicrobial properties of low-density polyethylene samples with the addition of marjoram powder at concentrations of 1, 3 and 5% in the Candida albicans relationship.



Aspergillus niger

Figure 5. Antimicrobial properties of low-density polyethylene samples with the addition of marjoram powder at concentrations of 1, 3 and 5% in the Aspergillus Niger relationship.

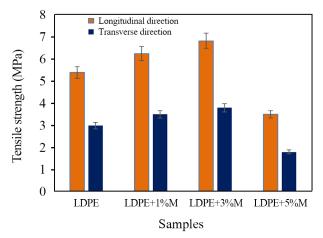


Figure 6. Tensile strength of LDPE with marjoram powder (longitudinal and transverse directions).

additives to polymers, including biopolymers, as most of these extracts led to a deterioration in the physical and mechanical properties, and the thickness of the biopolymer increased with increasing concentration of the added plant extracts [16].

Vapor permeability of low-density polyethylenebased films

Dried marjoram powder at a concentration of 5% deteriorated the water vapor permeability of low-density polyethylene and increased the permeability approximately 15 times compared to the control sample. At a concentration of 3%, permeability increased 2.9 times compared to the control sample, and the effect of the additive at a concentration of 1% was negligible (Figure 8).

The results of our study were inconsistent with those of previous studies, which reported that most plant extracts decreased water vapor permeability [16]. This

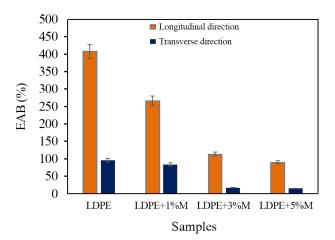


Figure 7. Elongation-at-break of LDPE with marjoram powder (longitudinal and transverse directions).

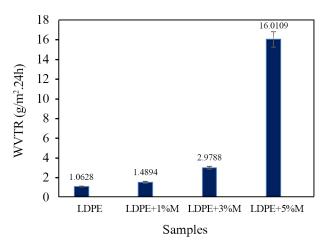


Figure 8. Results on the vapor permeability of the marjoram powder-based LDPE films.

is because the effect of dried plant powders or extracts on the polymer's barrier properties depends largely on the type of both the plant powder and polymer. The fiber content and hydrophilic chemical composition of the plant powder affect barrier properties, including absorption and permeability to gases, in addition to affecting the physical and mechanical properties of the polymer [17]. The method of drying the powder also has a decisive impact. The use of spray-dried powders increases the polymer's thickness, which significantly affects the barrier properties. The use of freeze-dried powders ensures the preservation of the effectiveness of heat-sensitive (which are destroyed by heat) and bioactive compounds, which in turn preserves their antimicrobial and antioxidant properties [18].

CONCLUSION

In the research results, low-density polyethylene samples supplemented with dry marjoram powder at a concentration of 3% showed the best technological properties. Increasing the concentration to 5% resulted in a significant increase in water vapor permeability and a deterioration in the physical-mechanical properties. Conversely, at 3%, the tensile strength of the samples increased in both longitudinal and transverse directions. At 1% concentration, water vapor permeability and physical and mechanical properties were not significantly affected, but LDPE samples did not show any antimicrobial activity at this concentration.

Based on the research results, a 3% concentration of marjoram powder is recommended as the ideal

manufacturing concentration for low-density (LDPE) films. Given the polvethylene antimicrobial properties of the used marjoram powder against fungi and Gram-negative bacteria, it is suggested that additional additives be used in future research alongside marjoram powder to enhance these properties. These additives, such as enzymes (glucose oxidase and lysozyme) and essential oils (clove essential oil), are able to maintain their antimicrobial properties when blended with polyethylene at a melting point of 190°C inside an extruder, and they are homogeneously mixed and evenly distributed throughout the polymer matrix.

CONFLICTS OF INTEREST

The authors declare that there are no conflicts of interest to disclose.

ABBREVIATIONS

LDPE Low-density polyethylene M Marjoram

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