

## EXPERIMENTAL CREEP TEST SENSIBILITY OF MECHANICAL PROPERTIES OF LDPE FILMS USED AS GREENHOUSE ROOF

### PROCENA MEHANIČKIH OSOBINA LDPE FOLIJA ZA PLASTENIKE EKSPERIMENTALNIM ISPITIVANJEM NA PUZANJE

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#### Keywords

- plasticulture
- multilayer films
- LDPE
- mechanical properties
- greenhouse
- creep-recovery tests

#### Abstract

Plastic films are widely used in agriculture throughout the Mediterranean area and the rest of the world, but their impact on the sustainability of agricultural production and the environment is a major concern. One of the factors limiting the recycling of agricultural plastics is the loss of mechanical properties due to ageing from exposure to external agents. This research analysed the degradation of the mechanical properties of LDPE films used for greenhouse roofs by naturally weathering them for nine months in harsh climatic conditions in the Tiaret region of Algeria. Tensile and creep-recovery tests were conducted to measure changes in the mechanical properties of both monolayer and tri-layer films, and the anisotropic character of the films was preserved during ageing. Cross linking improved the creep resistance for both types of films but affected different deformations. Results show that the degradation performance of the new tri-layer films is significantly better than that of the monolayer film with regard to mechanical properties. Therefore, it can be concluded that the erosion of greenhouse LDPE film by temperature, water, sand particles, wind, and humidity results in an extreme lifetime limit. LDPE film may have a longer period of use in Mediterranean Europe (with lower temperatures and no sand-wind effects) than in North Africa (with high temperature and adverse sand-wind effects).

#### INTRODUCTION

Ultraviolet (UV) radiation, temperature, and humidity are harsh climatic conditions that have been identified as the most damaging factors in the ageing process of LDPE greenhouse covers /1-3/. Given the impact of greenhouses on agriculture, there is a growing demand for their use. The cover film is a critical component of the greenhouse, and

#### Ključne reči

- plastika u poljoprivredi
- višeslojne folije
- LDPE
- mehaničke osobine
- staklena bašta
- ispitivanja pužanjem

#### Izvod

Plastične folije se uveliko koriste u poljoprivredi u čitavoj oblasti Mediterana, kao i drugim delovima sveta, ali je njihov uticaj na održivost u poljoprivrednoj proizvodnji i na okolinu od velikog značaja. Jedan od faktora koji ograničavaju recikliranje poljoprivredne plastike jeste gubitak mehaničkih osobina usled starenja zbog izloženosti spoljnim uticajima. Ovo istraživanje se bavi analizom degradacije mehaničkih osobina LDPE folija za plastenike, prirodnim izlaganjem surovim klimatskim uticajima u trajanju od devet meseci u oblasti Tiaret u Alžiru. Izvedena su ispitivanja zatezanjem i pužanjem radi utvrđivanja promena u mehaničkim osobinama jednoslojne i troslojne folije, dok je anizotropni karakter folija ostao očuvan tokom starenja. Otpornost prema pužanju je poboljšana poprečnim vezama prisutnim kod oba tipa folija, ali se ogledala u različitim deformacijama. Rezultati pokazuju da je svojstvo degradacije novih troslojnih folija znatno poboljšano u odnosu na jednoslojnu foliju, s obzirom na mehaničke osobine. Stoga, zaključujemo da je erozija LDPE folija za staklene bašte, izazvana temperaturom, vodom, česticama peska, vetrom i vlagom, takva da se postiže ekstremni životni vek. LDPE folije mogu dostići i duži radni vek u Mediteranskoj Evropi (sa nižim temperaturama i bez uticaja vetrova sa peskom) u odnosu na Severnu Afriku (sa visokim temperaturama i značajnim uticajima vetra i peska).

LDPE is a widely used material in plasticulture, commonly employed as an agricultural greenhouse cover. Despite its favourable chemical inertness, the LDPE polymer degrades slowly over time due to exposure to heat, solar UV radiation, mechanical stress, and chemical agents, as is typical of all organic substances. Some authors /4/ have demonstrated that the molecular orientation during film blowing affects tensile

properties, with greater strength observed in the direction of the covalently bonded carbon-carbon chain than in the transverse direction, dominated by weaker Van der Waals bonds. The mechanical properties of LDPE greenhouse covers are influenced by a range of climatic factors, including solar irradiation, temperature, humidity, rain, wind loads, and environmental pollution, /5/. Such factors cause microstructural changes in the LDPE films, with macromolecules primarily affected by oxidation processes that result in crosslinking or chain scission reactions, as evidenced by Fourier transform infrared spectroscopic analysis /6/. It is a well-established fact that plastic films used in agriculture, especially in greenhouses, undergo a gradual degradation of their mechanical properties when exposed to natural weathering and agrochemicals. According to a study conducted by Vox and Schettini /7/, exposure to climatic agents and agrochemicals did not significantly change the radiometric properties of the films in the solar and PAR wavelength range. However, there was a maximum variation of 10 % in the transmissivity coefficients within six months of experimental field testing. For stabilized films in the LWIR wavelength range, there were higher variations in the radiometric coefficients between the initial and final test values (up to 70 % of the initial value) /8/. The use of agrochemicals by growers also affects the lifespan of the films by generating by-products that cause deterioration of the covering material and changes in its mechanical and physical properties, /9/. The literature contains extensive research on the creep behaviour of LDPE /10-13/, primarily influenced by the deformation of its amorphous phase, itself strongly affected by variations in crystallinity and tie molecule tautness. When using agricultural composites to develop structural building products, improving their mechanical properties, particularly their creep performance, is often necessary. Previous studies have demonstrated that creep of agricultural composites is dependent on the type and content of matrices, coupling treatment, and the relative performance of the fibre-matrix interface in comparison to the viscoelastic properties of the compounds, /12-15/.

Coextrusion is a process that enables the combination of LDPE polymers with specific properties, such as high strength, selected permeation rate, high-barrier properties, high-strength sealing, and abrasion resistance, to achieve significant thickness reduction while maintaining or improving the multilayer films performance. Additionally, reducing the amount of expensive materials in multilayer structures or incorporating layers of recycled materials are additional methods of lowering film costs /16/. The most destructive degradation of plastic properties occurs through photo degradation when exposed to solar radiation, specifically UV-A radiation (315-400 nm), resulting in scission and crosslinking reactions and oxidation /17/. Tarantili and Kiose investigated the effect of accelerated ageing on the structure and properties of single, metalized, and multilayer films used in food packaging by exposing specimens of these films to repeated ageing cycles in a weather-O-meter under the combined action of ultraviolet, humidity, and heat. They found that polyethylene (PE) films exhibited modest decreases in their mechanical properties, which could be attributed to cross-

linking reactions with PE /18/. Gardette et al. studied the photo- and thermal oxidation of two PE samples containing different amounts of unsaturation. Their results showed two main outcomes: the initial number of unsaturated groups' influence on the rate of thermo-oxidation and the significant differences between thermal and photo-oxidation /19/.

Other agricultural plastics have been also taken in consideration by some authors, e.g., plastic containers of agrochemicals, whose ageing was analysed by Picuno et al. These authors concluded that, if properly managed, this category of plastic waste coming from agricultural application could be sufficiently decontaminated, so as to be anyway entrained into ordinary industrial chains for recycling /20, 21/.

The objective of this study is to assess how the harsh environmental conditions impact the mechanical and creep properties of the tri-co-extruded LDPE film. The film samples were exposed to varying temperature and UV radiation for a period of 9 months, and the effects on mechanical properties such as tensile strength, elongation at break, and modulus of elasticity were evaluated. Additionally, creep behaviour of the film was also analysed. The results of this study will provide valuable insights into the performance and durability of the film under harsh environmental conditions, which can be used to optimise its use in various applications.

## MATERIALS AND METHODS

Two distinct types of films manufactured and supplied by Agrofilm SA (Setif, Algeria) were utilized in this investigation as greenhouse covers. The first variant is an extruded monolayer LDPE film (prior to extrusion) that boasts a density of 0.923 g/cm<sup>3</sup> and has a weight-average molecular weight ranging from 90,000 to 120,000 g/mol. The raw LDPE material has a melt flow index (MFI) of 0.33 g/10 min, whereas the MFI with stabilizer is 10 g/10 min, and the thickness of the film is 180 µm. The second variant is a three-co-extruded layer LDPE film having the same thickness as the monolayer film (180 µm) and comprises the following proportions in the layers: 1/4, 1/2, and 1/4. The original hue of the film is a milky yellow.

In this study, tensile tests were conducted using an Instron model 4301 universal testing machine equipped with a 5 kN load cell. The tests were performed at room temperature with a cross head speed of 50 mm/min and under displacement-controlled conditions. To ensure the reproducibility of the results, between five and ten specimens were tested in some cases. At least two valid results were required to calculate the tensile properties, and any unsatisfactory test results were disregarded, with the test repeated if necessary. The tensile tests demonstrated excellent reproducibility. The specimens used in the tests had an initial overall length of 200 mm, an initial length between the jaws of 80 mm, and an initial width between the jaws of 6.5 mm. Each sample was tested five times to ensure accuracy.

## RESULTS AND DISCUSSION

### *Effect on mechanical behaviour*

In order to quantify the effects of natural ageing on the mechanical properties of the monolayer and tri-layer films,

a tensile test was conducted to evaluate the mechanical behaviour of unaged/virgin and naturally aged samples over a period of up to 9 months (6480 hours). The load was applied parallel to the average molecular orientation of the film obtained during processing. Since the tensile curves (Fig. 1, Fig. 2) of both the monolayer and tri-layer films showed an initial linear relationship, the modulus of elasticity (*E*) was determined by calculating the slope of the first portion of the tensile curve.

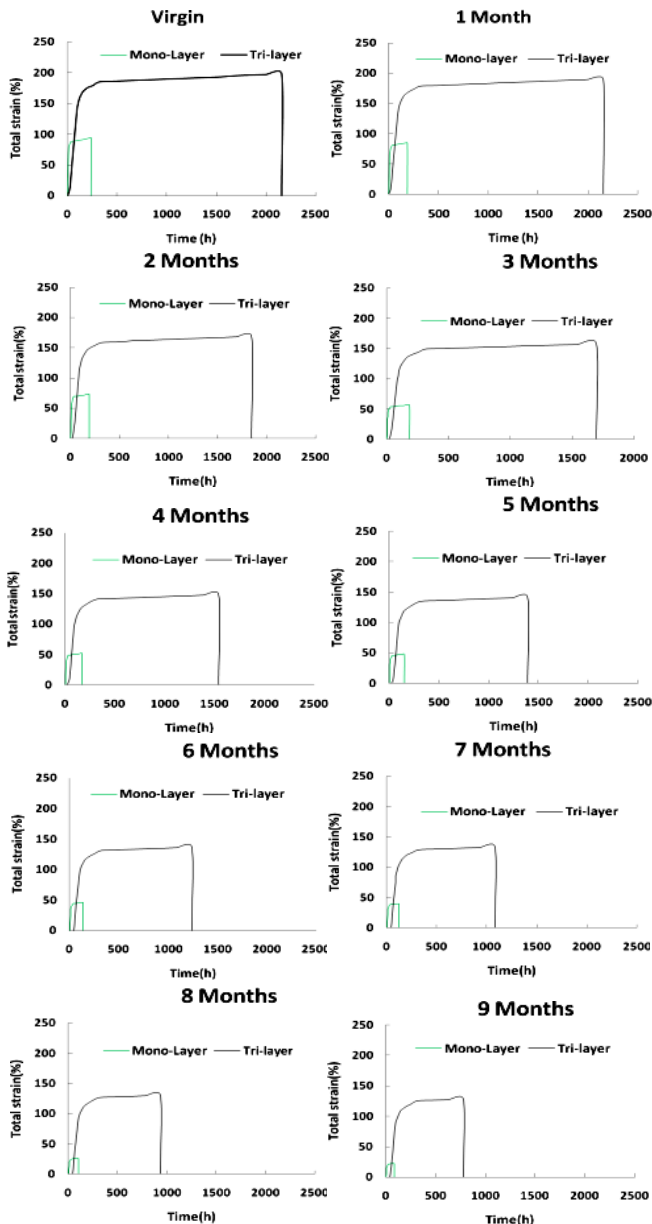


Figure 1. Total-strain curves of monolayer and tri-layer films at different ageing periods.

The variation of *E* over time for both the tri-layer and monolayer films is depicted in Fig. 3.

The modulus of elasticity, often denoted as *E*, is a measure of a material's stiffness or rigidity. When it comes to aging, materials can undergo changes in their mechanical properties, including the modulus of elasticity. Aging effects on the modulus of elasticity are particularly relevant in materials science and engineering.

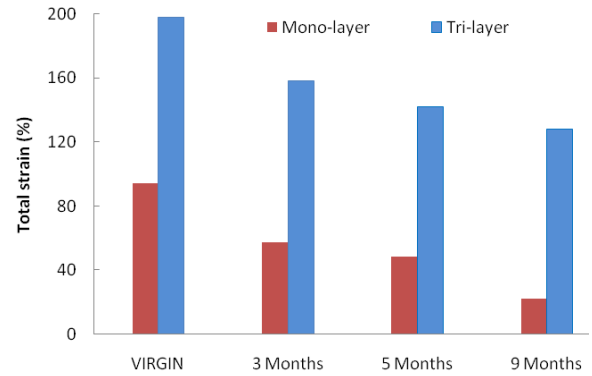


Figure 2. Total strain limit during loading vs. time.

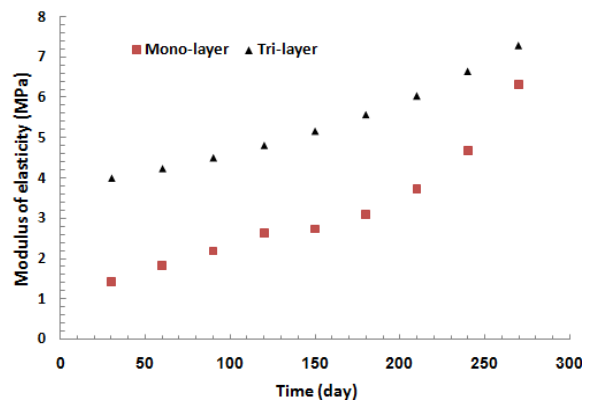


Figure 3. Variation of modulus of elasticity *E* with ageing.

- With ageing, the material may undergo structural changes at the microscopic level. For instance, in polymers, there might be cross-linking reactions or chain scission events that alter the overall material properties.
- In metals, aging might involve processes like precipitation hardening, where certain elements form precipitates that affect the material's elasticity.
- The evolution of the modulus of elasticity with aging can often be described by mathematical models. These models may take into account factors such as time, temperature, and the nature of the aging process.
- A generalised form of the evolution equation for the modulus of elasticity (*E*) with aging might be represented as:

$$E(t) = E_0(f(t))$$

where: *E*(*t*) is modulus of elasticity at time *t*; *E*<sub>0</sub> is the initial modulus of elasticity; and *f*(*t*) is a function describing the evolution over time.

The specific form of *f*(*t*) would depend on the nature of the ageing process and would typically be determined through experimental data and theoretical considerations. It is important to present equations or models for the modulus of elasticity in both the initial (untreated or unaged) state and the aged state. This allows for a direct comparison and understanding of how ageing affects the material's stiffness.

To determine the maximum allowable usage time of the film based on a 50 % reduction in its initial property criterion (i.e., strain at break), one can plot the normalised property variation against ageing time, depending on the total solar energy produced, [22]. In our tests, the single-layer film can withstand only 12.5 light-years (Ly), while for the triple-layer films; it can go up to 25 Ly - which is nearly double.

Creep experiments

The creep testing apparatus was developed in the laboratory of Engineering Physics. The tests were conducted by applying a constant load to the specimens, delivering an initial stress of 1500 g for a period of 24 hours each. Recovery immediately followed the removal of the stress and lasted for a further 24-hour period /23/. The creep-recovery response was evaluated for various exposure times (0, 1, 2, 3, 4, 5, 6, 7, 8, and 9 months).

To evaluate the creep behaviour of unaged/virgin and naturally aged films (up to 9 months), creep-recovery tests were conducted. The specimens were loaded parallel to the average molecular orientation obtained during film processing. The films' instantaneous elastic strain as a function of time was recorded, and after stress application, much of the deformation was almost instantaneously recovered. A quantitative assessment was performed to determine the impact of natural ageing on the films' mechanical properties based on measured properties. Stress levels at the same ageing period were significantly higher for tri-layer films compared to monolayer films, /24-25/.

The total strain curves for monolayer and tri-layer films at various ageing periods are shown in Figs. 2 and 3 for unaged and aged films up to 9 months. The strain-time diagrams for all monolayer and tri-layer samples indicated an initial linear increase in the first region of the curve, followed by a nonlinear increase in the second region. Regardless of the ageing conditions and type, all samples exhibited the same trend in behaviour. Figures depicting typical creep-recovery curves for monolayer and tri-layer LDPE films stretched along the machine direction were presented for various exposure time periods. The analysis of the samples' mechanical properties showed the value of instantaneous elastic strain, /26-29/.

Figure 4 illustrates the changes in tensile strength during loading for the two films at different ageing stages. The behaviour of the ultimate properties confirms that cross-linking is the dominant mechanism during the first four months of ageing. The variations in strain, stress, and elasticity modulus for monolayer and tri-layer films can be grouped together based on the diagrams of stress-strain and the elasticity modulus graph, as shown in Table 1.

Table 1. Stress and average strain during ageing.

Time of ageing (months)	Virgin	1 (May)	2 (Jun)	3 (Jul)	4 (Aug)	5 (Sep)	6 (Oct)	7 (Nov)	8 (Dec)	09 (Jan)
<i>e</i> (%)										
Monolayer	89, 56	86, 27	74, 32	56, 76	52, 21	45, 55	43, 05	38, 28	22, 32	20, 59
Tri-layer	192, 54	192, 5	192, 32	179, 2	176, 2	171, 78	169, 65	158, 21	153, 7	128, 44
<i>a</i> (MPa)										
Monolayer	16, 25	11, 5	14, 18	9, 53	8, 38	10, 73	14, 58	7, 28	11, 58	11, 8
Tri-layer	27, 45	27, 88	27, 98	30, 95	22, 02	28, 68	22, 83	25, 9	27, 18	19, 15
<i>E</i> (MPa)										
Monolayer	0, 82	1, 435	1, 83	2, 2	2, 64	2, 75	3, 1	3, 73	4, 7	6, 33
Tri-layer	3, 99	3, 99	4, 24	4, 51	4, 82	5, 17	5, 58	6, 5	6, 66	7, 31

*E*: modulus of elasticity; *a*: fracture stress; *e*: elongation at break.

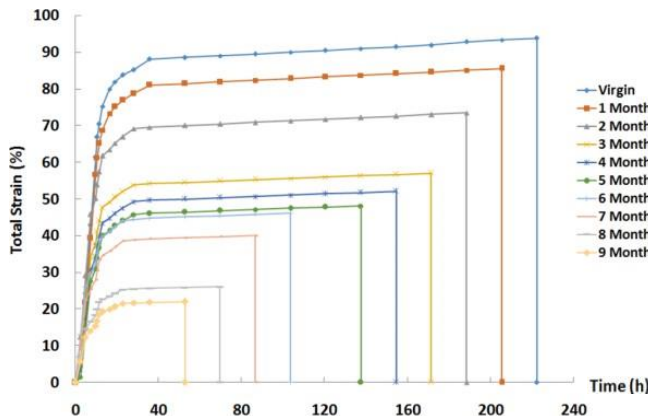


Figure 4. Ageing effects on the creep-recovery of monolayer LDPE.

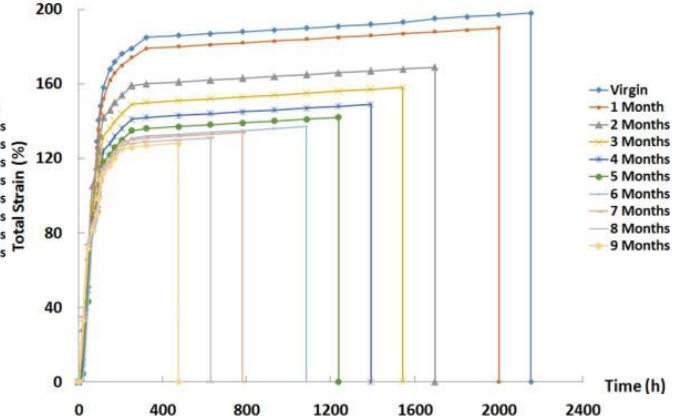


Figure 5. Ageing effects on the creep-recovery of tri-layer LDPE.

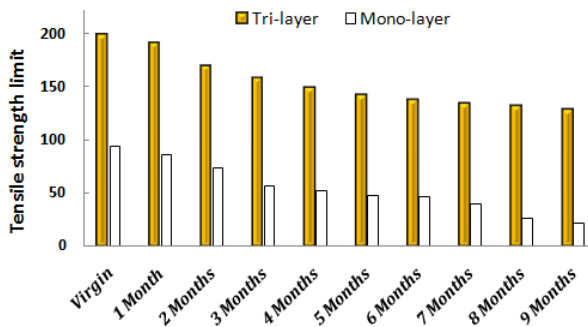


Figure 6. Tensile strength limit during loading vs. creep time.

CONCLUSIONS

The study found that natural ageing affects the performance of LDPE monolayer and tri-layer films. The films' mechanical properties degrade due to weathering and ageing, but they are more stable and can withstand up to 9 months of exposure to UV radiation and high temperatures. The degradation of films is attributed to factors such as temperature, water, and sand particles in the greenhouse environment. The study also suggests that the LDPE film may have a longer lifespan in Europe than in North Africa due to differences in temperature and sand-wind effects, which

can have positive effects on reducing the plastic footprint of agriculture.

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