

Article

The Impact of Accelerated Aging on Organic, Inorganic, and Food-Nature Biocolorants in Biodegradable Polymer Films

Petková Mária ^{1,*}, Hricová Marcela ¹, Jančovičová Viera ¹, Tomčíková Zita ² and Ujhelyiová Anna ¹ 

¹ Institute of Natural and Synthetic Polymers, Faculty of Chemical and Food Technology, Slovak University of Technology in Bratislava, Radlinského 9, 812 37 Bratislava, Slovakia; marcela.hricova@stuba.sk (H.M.); viera.jancovicova@stuba.sk (J.V.); anna.ujhelyiova@stuba.sk (U.A.)

² Research Institute for Man-Made Fibres a.s., Štúrova 2, 059 21 Svit, Slovakia

* Correspondence: maria.petkova@stuba.sk

Highlights

What are the main findings?

- A comparison evaluation of the effect of selected pigments from the groups of inorganic, organic, and natural food-grade pigments on the processing properties of the material, the coloration of the packaging material achieved by adding the selected pigments into the polymer matrix, and the assessment of color stability by means of accelerated aging of the material.
- In order to preserve the biodegradability of the material and to ensure suitability for applications such as food packaging, natural food-grade pigments are of particular interest. However, both organic and inorganic pigments also met the requirements for maintaining the material's biodegradability. In this study, they served as comparison materials for evaluating the performance and properties of natural food-grade pigments.
- Natural food-grade pigments exhibited lower color intensity compared to organic and inorganic pigments. Moreover, under accelerated aging conditions, color fading occurred earlier and to a greater extent in materials containing natural food-grade pigments. A greater decrease in mechanical properties was also observed in materials containing these pigments.

What is the implication of the main finding?

- However, if natural food-grade pigments are intended for use in food packaging applications, long-term outdoor color stability would not be a critical requirement for this type of material. The achieved results can be considered satisfactory, and therefore, it can be concluded that natural food-grade pigments represent a promising and interesting area for further research and development.



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Abstract

This work presents the preparation and obtained results of the properties of biodegradable-oriented systems of dyed polymer by biocolorants in mass. The oriented systems (films) were prepared from biodegradable material Nonoilin. Our applied research is focused on preparing masterbatches using inorganic, organic, and food-nature pigments to prepare films as packaging materials. Inorganic pigments, such as iron and titanium oxide, and organic pigments were selected to maintain the biodegradability of the polymer mixture, as the manufacturer declares the biodegradability of the selected pigments. The food-natural pigments are extracted from plants and food pigments, such as chlorophyll, caramel, and violets. First, rheology was evaluated to verify the processing conditions of the

materials, and then the properties of the prepared films were examined. Mechanical properties, supermolecular structure, and coloristic properties were assessed for the pure and dyed films. We investigated color fastness after accelerated thermal-light aging using Q-SUN equipment. Food-nature pigments showed sufficient colorability after preparation, although the coloration was lost relatively quickly after accelerated light aging. If they are used as food packaging materials, these pigments would be highly safe for health, in addition to being biodegradable. The color stability of inorganic and organic pigments reached high stability values even after accelerated aging.

Keywords: biodegradable polymer; dyeing in mass; accelerated aging; mechanical properties

1. Introduction

Our previously published work [1] focused on the preparation of biodegradable fibers based on masterbatch formulations, which were subsequently processed into different types of oriented systems, including fibers and films. In the present study, we report and discuss results obtained from oriented systems prepared in the form of films. This approach was adopted because, for a comprehensive comparison of inorganic, organic, and food/nature pigments, fiber preparation was not feasible in all cases. Specifically, food/nature pigments did not exhibit suitable rheological properties required for fiber spinning; therefore, an alternative oriented system in the form of films was prepared and used for further investigation. Researchers have been finding ways to replace synthetic polymers with more environmentally friendly versions for some time. Biodegradable polymers represent a rapidly growing group of materials with significant potential in medicine, environmental technologies, and sustainable materials engineering. Several review papers have focused on biodegradable polymers and their potential applications. Therefore, many of them highlight various possible applications rather than focusing on a single specific use [2–8]. Their ability to undergo degradation into non-toxic byproducts makes them highly attractive alternatives to conventional, non-degradable polymers [9]. These polymers are defined by their ability to degrade through natural processes—enzymatic or hydrolytic—into smaller, non-toxic compounds such as water, carbon dioxide, or natural metabolites [10,11]. An overview of the most widely used biodegradable polymers is discussed, along with their processing and functional properties. It highlights the advantages of employing polymer blends for their newly acquired properties. Biodegradable plastics made from renewable resources are being extensively investigated, and the results indicate that they can serve as a full replacement and competitor to traditional synthetic polymers in certain fields. Many research studies focus particularly on the medical applications of biodegradable materials, such as the development of 3D-printed scaffolds [12], and others utilize them in orthopedic applications [13]. Some studies, on the other hand, point to the potential use of biodegradable materials for packaging applications [14]. The increasing demand for sustainable materials has stimulated intensive research into biodegradable polymers. Applications range [15] from packaging films, electrochemical applications [16,17], to biomedical devices and controlled drug delivery [18]. Understanding the behavior of individual polymers, as well as the opportunities offered by polymer blends, is essential for optimizing their performance in specific contexts.

We often encounter polylactic acid (PLA), which, however, has its limitations not only in terms of the possibility of obtaining properties for various applications but also in terms of biodegradability [19]. Lactic acid can be produced from renewable resources, such as corn starch and sugarcane, so it is considered a green material. It offers good mechanical

strength and processability by conventional methods, such as extrusion and injection molding. However, its brittleness and relatively slow degradation under physiological conditions limit its use in various applications, such as packaging materials, scaffolds for tissue engineering, and biodegradable fibers. Nevertheless, polylactic acid is biodegradable under specific conditions, such as industrial compost [20].

PLA is one of the most widely studied biodegradable polymers, but there are others that have an important place in research. Polyglycolic acid (PGA) exhibits high crystallinity and excellent mechanical strength, with a significantly faster degradation rate than PLA. Its hydrolytic degradation yields glycolic acid, which is naturally metabolized by the body. Polycaprolactone (PCL) is a semi-crystalline polymer with excellent flexibility and slower degradation rates compared to PLA and PGA. It is highly miscible with other polymers, making it suitable for blending systems. Its applications include long-term drug release systems and tissue engineering scaffolds. A single biodegradable polymer rarely fulfills all mechanical, thermal, and biological requirements. Therefore, blending strategies are often applied. PLA/PCL blends combine the rigidity of PLA with the flexibility of PCL, yielding materials with improved toughness and adjustable degradation. As already mentioned, PLA is rigid but brittle, while polycaprolactone (PCL) is flexible and ductile. Their blends provide a compromise between strength and toughness. Ostafinska et al. in their study reported strong synergistic effects in PLA/PCL blends with finely dispersed PCL particles, which led to a 16-fold increase in toughness compared to neat PLA, particularly when a high-viscosity PLA matrix was used [21]. A comprehensive review highlighted that PLA/PCL blends are most effective at ratios around 80/20, and that both PCL particle size and PLA crystallinity are decisive factors for toughness [22,23]. PLA/PHB blends could improve thermal stability and processability. Blending also allows tuning hydrophilicity, crystallinity, and degradation kinetics, making such systems attractive for highly specific applications. Polyhydroxybutyrate (PHB) is highly biodegradable but suffers from brittleness and poor thermal stability. When blended with PLA or reinforced with fillers, improved performance can be achieved. A study on PLA/PHB/cellulose nanocomposites showed that the materials exhibited improved thermal stability and mechanical strength and were stable during extrusion and 3D printing at ~200 °C [24]. Composites of PHB with PLA and natural fillers such as lignin, polyethylene glycol (PEG), or microcrystalline cellulose (MCC) improved tensile strength and processability compared to pure PHB [25]. Since it is an available polymer, several research papers deal with the applications and achieved properties of polylactic acid or mixtures of polylactic acid with additives or other polymers. Scientific work devoted by Hussain M. et al., a review on PLA-based biodegradable materials for biomedical applications, PLA, from its synthesis, through properties to biodegradation, is also a reflection of the interest in finding new possibilities for using biodegradable polymers in different applications [26]. Review by Trivedi et al. about the PLA-based biocomposites says that PLA biocomposites could be considered as the best source of sustainable products. PLA's mechanical and thermal properties can be enhanced by reinforcing the nano- and micro-sized natural fibers and cellulose [27]. A group of scientists modified PLA masterbatches with montmorillonite, which they had previously modified, to observe the effect of this modification on the photooxidation of PLA [28]. Byrne F. et al. published the effect of masterbatch additions on various properties of PLA [29]. They point to the addition of another polymer to achieve the desired property improvement, citing many other works that deal with this very topic. Environmental, economic, and safety challenges have forced scientists and industrial producers to partially substitute petrochemical-based polymers. The interest in PLA and PLA composites is attested by a large number of scientific papers and reviews.

In several scientific publications, NONOILEN[®] is mentioned as a commercial material prepared from a blend of PLA and PHB; however, the number of studies specifically devoted to this material remains limited. Since our work is based on experimental research using this commercial polymer blend, we also include available references in this area to provide context and support for the discussion of our results. Scientific studies focused on the polymer blend NONOILEN[®] address its processability, properties, and application in 3D printing technology. One study investigated the effect of repeated thermomechanical processing on the thermal, mechanical, and molecular properties of a PLA/PHB blend, confirming its sufficient stability and suitability for reprocessing. Another study focuses on the composition and processing of this polymer blend derived from renewable resources and confirms its suitability for additive manufacturing. Both studies clearly declare that NONOILEN[®] is a biodegradable polymer material based on PLA and PHB, which degrades without the formation of microplastics and represents a promising material for environmentally sustainable applications [30,31].

The largest application of thermoplastics is the packaging market, because in 2022, they accounted for approximately 40% of global plastic production, reflecting the need to find new materials that are acceptable for the ecological sustainability of the planet [32]. Packaging is a highly important feature for increasing the shelf life of perishable food products because it decreases contamination and provides food safety assurance [33].

The coloration of biodegradable polymers represents an emerging field at the interface of materials science, polymer chemistry, and sustainability. Different classes of colorants—including inorganic pigments, synthetic organic dyes, and natural pigments—offer distinct advantages and challenges when incorporated into biodegradable matrices such as PLA, PCL, or polyhydroxyalkanoates (PHAs). Inorganic pigments, such as TiO₂, ZnO, iron oxides, and carbon black, are widely applied through masterbatch techniques and provide high color stability, UV protection, and, in some cases, antibacterial functionality, although they may alter crystallinity and rheological behavior of the native polymer [34,35]. Synthetic organic dyes, including disperse and reactive azo dyes, provide a broad color spectrum and high chromatic intensity; however, their degradation products may compromise environmental safety [36,37]. Benetti et al. focused on describing the difference in the impact of the choice of inorganic and organic pigments in the preparation of masterbatches [38]. Dyeing and spectroscopic properties of natural dyes on PLA and PET fabrics are described by a group of authors in the article “Dyeing and Spectroscopic Properties of Natural Dyes on Poly (Lactic Acid) and Poly (Ethylene Terephthalate) Fabrics” by Sriumaoum et al. [39]. Also, other scientific work describes natural dyes [40].

We can observe the rise in scientific works that deal with the topic of the coloring of PLA or other biodegradable materials. As is known, the properties and ecology of the material are important for all of us, but for sales marketing, color is important, especially for packaging materials.

However, we focused on coloring with different groups of pigments—inorganic, organic, and natural—so that it is the least harmful material. Natural pigments are very important to prevent health risks during the storage and protection of food; there should be no contact with chemicals. Inorganic pigments are not available in a wide color range, but they have an irreplaceable presence in pigments, adding value through the properties they possess. Organic pigments are associated with a wide range of colors, but their underlying chemistries are frequently linked to non-benign environmental profiles. However, this myth is no longer valid, as there are suppliers of polymer pigments that declare that the pigments are suitable for biocompatible polymer matrices.

2. Materials and Methods

2.1. Materials Used

The following materials were used for experimental research work (Table 1):

Table 1. Overview of pigments and polymers used for the preparation of colored films.

Polymer	Nonoilen	
Pigments 1 wt. %.	Inorganic	F303M TiO ₂ PXA
	Organic	GY FR FB
	Food/Nature	Caramel Chlorophyll Violet

Polymer: Nonoilen IM 3056-2 (NOil) is a thermoplastic material based on biodegradable polymer blends made of 100% renewable raw materials, which undergo biodegradation under various natural conditions. The material is supplied in pellet form. Its complex viscosity at 180 °C, determined using an oscillatory rheometer, amounted to 441 Pa·s, density is 1.2 g/cm³, and melting point is 170 °C (produced by Panara Ltd., Nitra, Slovakia).

Pigments

Pigment selection followed European Standard EN 13432:2000 (Packaging—Requirements for packaging recoverable through composting and biodegradation—Test scheme and evaluation criteria for the final acceptance of packaging, European Committee for Standardization (CEN); Brussels, Belgium, 2000) which defines requirements for biodegradable polymer blends, including colorants, to ensure that their inherent biodegradability is retained.

- Inorganic pigments are obtained from natural sources and are non-synthetic.
 - Fepren TP 303M (F303M): Particle size < 50 nm (BET) (Procheza, Praha, Czech Republic);
 - Titanium dioxide AV01SF batch 167112 (TiO₂): Primary particle size (TEM) is 21 nm (Procheza, Praha, Czech Republic);
 - Printex Alpha (PXA): Average primary particle size is 20 nm (Orion S.A., Spring, TX, USA).
- Organic: All of them are synthetic.
 - Graphtol Yellow 3GP (GY): Particle size distribution D50 = 2 µm, chemical formula is C₃₄H₃₂N₆O₁₂, chemical name of the components is Alcohols, C11-14-iso-, C13-rich, and is ethoxylated;
 - PV Fast Red D3G (FR): Particle size distribution D50 = 0.495 µm, and substance name is Pyrrolo [3,4-c]pyrrole-1,4-dione, 3,6-bis (4-chlorophenyl)-2,5-dihydro-;
 - PV Fast Blue A4R (FB): Average particle size is 46 nm.
 (All are from Clariant, Muttenz, Switzerland.)
- Food/Nature: These are the edible pigments of organic origin, extracted from natural sources: caramel, chlorophyll, and violet (Food Colours Perczak Sp. J., Piotrków Trybunalski, Poland).

Silicone oil V350 as a dispersing agent (produced by Azelis Slovakia, s.r.o., Bratislava, Slovakia).

Color Masterbatches and Films Preparation

Nonoilen was used for the preparation of color masterbatches. Color pigments with Nonoilen and a dispersing agent were mixed by a laboratory line with twin-screw extruder WP ZDSK (Werner & Pfleiderer, Dinkelsbühl, Tamm, Germany). Melting was performed on the screw extruder with a diameter of 28 mm, in the temperature range of 180–210 °C. The extruded polymer string was cooled and pelletized. The concentration of the pigments in the masterbatches was 3 wt.%, and the dispersing agent was contained at 0.6 wt.%.

In the next step, the pellet of pure NOil and color masterbatches were dried in a laboratory oven for 3 h at 60 °C. Subsequently, blends of pure NOil and a specified quantity of color masterbatches with selected pigments were mixed. The cast films were made from these prepared blends, resulting in dyed films with 1.0 wt.% pigment content. The cast films were prepared on a laboratory line using an extruder with a diameter of 18 mm and a slot width of 48 mm.

The prepared films had an average width of 5 cm and an average thickness of approximately 100 µm.

2.2. Methods Used

2.2.1. Rheological Properties

The rheological behaviors of the Nonoilen and the color masterbatches were investigated using a rotary rheoviscosimeter Physica MCR 101 (Anton Paar, Graz, Austria). On a rotational rheometer, the torque required to impose a given angular velocity on the rotor was detected. The molten sample filled the gap between two parallel plates. Viscosity and shear stress were subsequently computed from the measured torque and angular velocity by the instrument software Anton Paar RheoCompass 1.24. Experiments were conducted in an air atmosphere. A parallel plate geometry was used with a diameter of 25 mm. The gap was set at 1 mm. All rheological measurements were performed at 190 °C, with a 1% strain from 0.1 to 100 rad/s.

2.2.2. Mechanical Properties

Mechanical properties such as tenacity and elongation at break were measured under the following conditions: tenacity range 5000 N; elongation rate 20 mm/min, and clamping length 200 mm; at a temperature of 20–21 °C and humidity 63–68%. The number of measurements for one sample was 5. Statistical analysis was performed on the dataset, from which the standard deviation was calculated. The EN ISO 13934-1:2013 (Textiles—Tensile properties of fabrics—Part 1: Determination of maximum force and elongation at maximum force using the strip method. ISO: Geneva, Switzerland, 2013) standard was used to determine the tenacity and elongation at break of the prepared cast films.

2.2.3. Supermolecular Properties

Microscopic evaluation of the cast films was performed using an Nf-Meopta Prague microscope (Meopta, Prague, Czech republic) with a 250× magnification.

2.2.4. CIELab Coordinates of Colorimetric

Colorimetry deals with the measurement and characterization of colors. Each color can be expressed in the CIELab in colorimetric trichromatic coordinates (L, a, and b), which were obtained according to ISO 12647-2:2013. L is the parameter of specific lightness, and it is the position between black and white on the axis of color space. Parameter a determines colors from green to red, and parameter b characterizes colors from blue to yellow. The L, a,

and *b* parameters were measured ten times for each sample, and subsequently, the standard deviations were calculated. The total color difference in two colors is defined based on the color difference in the sample according to the CIE 1976 standard (ΔE). The color deviation, in this case, characterizes a change in color shade due to thermal-light aging of the dyed sample. In the calculation, the sample after a defined aging duration is compared to the sample before accelerated aging. The total color difference ΔE was calculated from Equation (1).

$$\Delta E = \sqrt{[(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]} \quad (1)$$

The coloristic properties of the dyed prepared films were evaluated by using the TECHKON SpectroDens instrument (TECHKON GmbH, Königstein, Germany) with settings of illumination type D65, standard observer 10°, and absolute white standard. Before thermal-light aging, the parameters *L*, *a*, and *b* were determined. Subsequently, the films were placed in the Q-sun instrument for thermal light aging. At time intervals of 1 h and 3, 6, 12, 18, 24, 48, and 72 h, samples were removed from the chamber, and CIELab coordinates were measured again.

2.3. Process of Aging

Accelerated light aging was performed with a radiation intensity of 300–800 nm at 494 W/m² in the Q-SUN Xe-1-S chamber (Q-LAB, Westlake, OH, USA), where the temperature was regulated at 65 °C (black panel). Measurement conditions were radiation intensity at 340 nm—0.68 W·m^{−2} and light intensity at 142,000 lx. The samples were evaluated before, during, and after aging. A 72 h period of accelerated light-heat aging corresponds to approximately 3.45 years of real external aging. The aging period corresponds to continuous exposure of materials to conditions of increased temperature, and intense UV radiation and visible light (midday sunlight). Therefore, the actual, total time of use of the materials exceeds the calculated laboratory aging period.

3. Results and Discussion

A rotational viscometer equipped with a plate–plate geometry was employed to characterize the rheological behavior of the investigated systems, specifically the relationship between viscosity and shear rate. The impact of different classes of pigments—inorganic (Figure 1a), organic (Figure 1b), and food-natural pigments (Figure 1c)—on the rheological properties of pure and color NOil masterbatches was evaluated. This evaluation was carried out through the analysis of flow curves, which were constructed based on the dependence of viscosity on shear rate.

For all masterbatches, as well as for pure Nonoilen, the viscosity exhibited a decreasing trend with increasing shear rate, demonstrating the typical behavior of polymer melts. The reduction in viscosity observed in the masterbatches containing pigments was more pronounced compared to pure Nonoilen, indicating a notable influence of the pigment particles on the flow properties of the matrix. Particularly in the case of natural pigments (Figure 1c)—such as caramel, chlorophyll, and violet pigments—the effect was even stronger, which could suggest enhanced particle–polymer interactions or morphological changes in the melt that amplify the change in viscosity as a function of increasing shear rate.

The course of viscosity dependence on shear rate for the evaluated masterbatches with all types of pigments obtained from rheological measurements confirmed the experience and conclusions from the evaluation of technological stability, where it was stated that masterbatches with pigments can be used in dyeing Nonoilen masterbatch. Masterbatches with pigments have rheological behavior similar, with some decrease, to pure Nonoilen, but despite this, their use in dyeing Nonoilen matrix can complicate the technological

process of preparing masterbatches or subsequently products manufactured from them. Therefore, it is necessary to consider the processing options for these masterbatches for individual technologies.

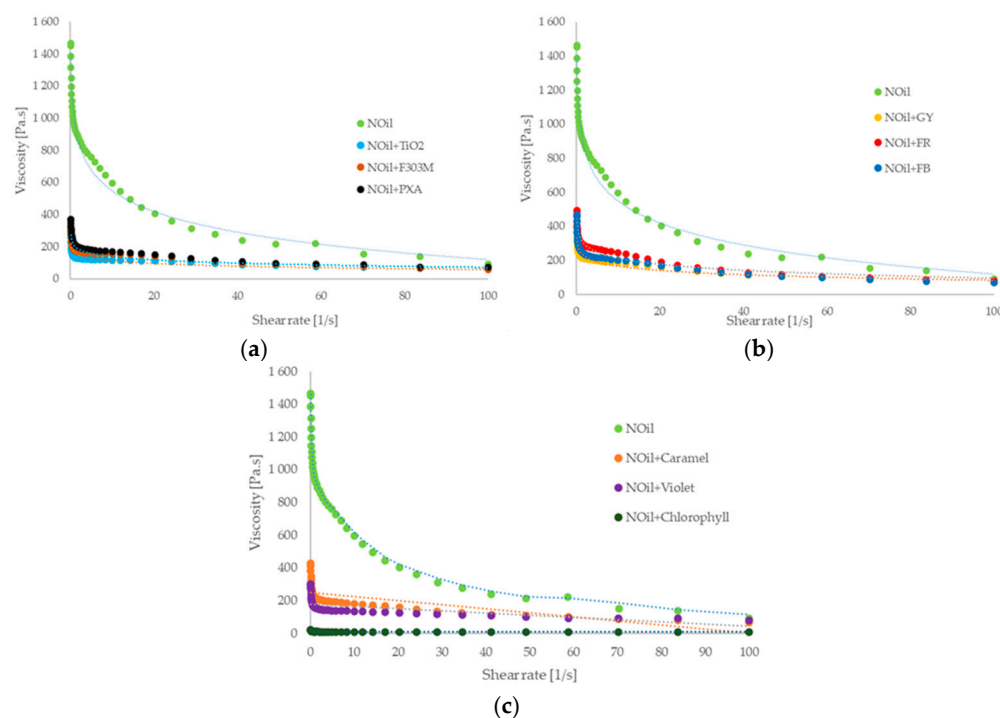


Figure 1. Dependence of viscosity on shear rate for pure NOil and color masterbatches with 1 wt.% content of pigment. (a) inorganic pigments. (b) organic pigments. (c) natural pigments.

For all prepared NOil cast films, the mechanical performance was evaluated in terms of tenacity and elongation at break (Table 2). The experimental results clearly demonstrate that the incorporation of pigments reduces the overall mechanical performance, as the colored films do not attain the tenacity at break observed in the pure NOil film. In most cases, the elongation at break of the cast NOil films follows a similar trend to their tenacity at break; that is, film from pure NOil exhibits higher strength, and also achieves a higher elongation value. The NOil films containing pigments achieve lower elongation at break, but on the other hand, a much higher measurement of mistakes.

Mechanical properties of films containing inorganic and organic pigments revealed that cast films with inorganic pigments exhibited more uniform and slightly higher values of tenacity and elongation at break compared to films containing organic pigments. Among all the tested cast-colored films, higher tenacity at break was achieved in films incorporating the inorganic pigment TiO₂, whereas the lowest values were observed in films containing the organic red pigment. Despite these observed differences, the mechanical properties of all pigmented cast films remained inferior to those of the film prepared from pure NOil.

The better properties of TiO₂-filled films can be attributed to the fine particle size, high thermal stability, and relatively good interfacial compatibility of this inorganic pigment with the polymer matrix, which collectively promote better stress transfer under load. In contrast, organic pigments tend to exhibit lower compatibility with the hydrophobic polymer matrix, which can result in less homogeneous dispersion and the formation of micro-defects acting as stress concentrators. These structural irregularities likely explain the diminished tenacity and elongation at break in films containing organic pigments, particularly in the case of the red pigment.

Table 2. Mechanical properties of pure NOil and masterbatches with inorganic, organic, and natural pigments.

Pigment	Samples	Tenacity at Break [N]	Standard Deviation [N]	Elongation at Break [%]	Standard Deviation [%]
-	NOil	146.6 ^a	2.6	2.0 ^a	0.2
Inorganic	NOil + TiO ₂	85.6 ^{bcd}	10.3	1.2 ^{ab}	0.4
	NOil + F303M	67.9 ^{cd}	13.4	1.7 ^{ab}	0.5
	NOil + PXA	67.5 ^d	6.3	1.4 ^{ab}	0.4
Organic	NOil + GY	57.8 ^{cd}	14.5	1.0 ^b	0.2
	NOil + FR	57.0 ^{bcd}	22.1	1.0 ^b	0.4
	NOil + FB	66.6 ^d	5.0	1.2 ^b	0.3
Natural	NOil + Caramel	108.0 ^b	6.9	1.1 ^b	0.1
	NOil + Violet	86.9 ^c	5.7	0.9 ^b	0.1
	NOil + Chlorophyll	82.2 ^{bcd}	16.4	0.6 ^b	0.3

Values are mean $\pm$ SD (n = 5). Within each column, values that do not share a common superscript letter are significantly different (Welch one-way ANOVA followed by Games–Howell post hoc test, $p < 0.05$). Letters (^a, ^b, ^c, and ^d) represent a compact letter display: values sharing at least one letter are not statistically different, whereas values without a common letter differ significantly ($p < 0.05$). Letters are evaluated separately for each column. Statistical analysis: Welch ANOVA followed by the Games–Howell post hoc test, $\alpha = 0.05$, assuming n = 5.

Data are reported as mean $\pm$ SD (n = 5). Differences in mechanical properties among formulations were evaluated using one-way Welch ANOVA to account for unequal variances, followed by Games–Howell post hoc multiple comparisons. Statistical significance was set at $p < 0.05$. Pigment addition significantly affected tenacity at break (Welch ANOVA: $F(9, 15.90) = 153.75$, $p = 9.67 \times 10^{-14}$) and elongation at break (Welch ANOVA: $F(9, 16.00) = 12.30$, $p = 1.24 \times 10^{-5}$).

This behavior can be attributed to the effect of pigment loading on the structural homogeneity of the polymer matrix. At some point, pigments' agglomeration and non-uniform dispersion within the polymer phase increase. Such inhomogeneities act as stress concentrators, thereby weakening the matrix continuity and reducing both tenacity and elongation at break. This interpretation is further supported by microscopic image analyses, which confirmed the presence of local irregularities in the pigment distribution. Consequently, the deterioration of mechanical parameters in highly pigmented films can be directly associated with the reduced structural integrity of the polymer network caused by poor pigment dispersion.

Microscopic evaluations of NOil films containing different types of pigments revealed a distinct distribution of pigment (Figure 2), which exerted a direct influence on all evaluated properties of the prepared films. Inorganic and organic pigments, in comparison to natural pigments, generally formed fewer and less pronounced agglomerates. Nevertheless, a fully homogeneous dispersion of these pigments within the polymer matrix was not consistently achieved, as minor irregularities in their distribution could still be detected.

However, films containing natural pigments exhibited more evident and larger pigment agglomerates, particularly in the case of chlorophyll. This poor dispersion was closely associated with the most pronounced decrease in viscosity observed in the rheological measurements, indicating strong particle–particle interactions rather than with the polymer matrix, resulting in disrupted flow behavior. Similarly, the mechanical properties confirmed that films with natural pigments displayed reduced tenacity and elongation at break. Moreover, a higher experimental error was recorded in these measurements, which also presents a situation of worse dispersion and greater agglomeration.

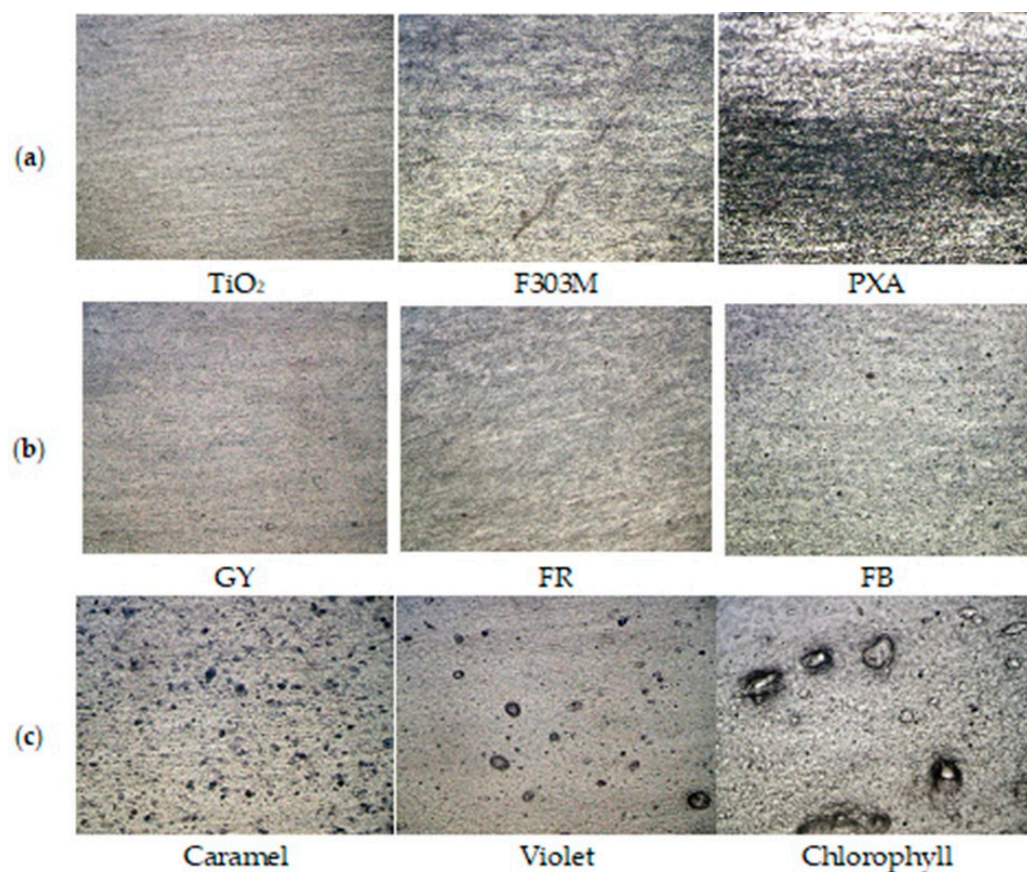


Figure 2. Microscopic images of films prepared from Nonoil and masterbatches with a pigment content of 1.0 wt.% pigments, with a magnification of 250 $\times$. (a) Inorganic pigments. (b) Organic pigments. (c) Natural pigments.

The tendency of natural pigments to form larger agglomerates can be attributed to their inherent chemical structure. Many natural pigments, such as chlorophylls, possess polar functional groups and limited thermal stability, which reduce their compatibility with the relatively hydrophobic biopolymer matrix. This mismatch in polarity promotes poor interfacial adhesion and aggregation of pigment particles, ultimately impairing their dispersion. Additionally, natural pigments often contain molecules with varying solubility and stability, further contributing to structural inhomogeneity within the polymer films. These factors collectively explain the stronger deterioration in both rheological and mechanical performance observed for films containing natural pigments.

One of the objectives of our research was to investigate the stability of the coloristic properties of cast films prepared from pigmented masterbatches. For this reason, attention was focused on assessing the effect of thermo-light aging on cast films containing inorganic (Figure 3), organic (Figure 4), and natural (Figure 5) pigments. The coloristic properties of the prepared NOil-based cast films were evaluated by determining the CIE Lab parameters, where L, a, and b are said about color characterization in colorimetric trichromatic coordinates (Table 3). In Table 3, there are L, a, and b parameters and a calculated standard deviation for showing only pigment TiO₂. Other pigments are characterized in the attachment (Tables A1–A8). Subsequently, the total color difference (ΔE) was calculated before and after thermo-light aging at different aging times (1, 3, 6, 12, 24, 48, and 72 h). The calculation quantifies the change in film color as a function of accelerated aging duration compared to the as-prepared film (before accelerated aging). The pre-aging sample is taken as the reference standard for computing the total color difference (ΔE).

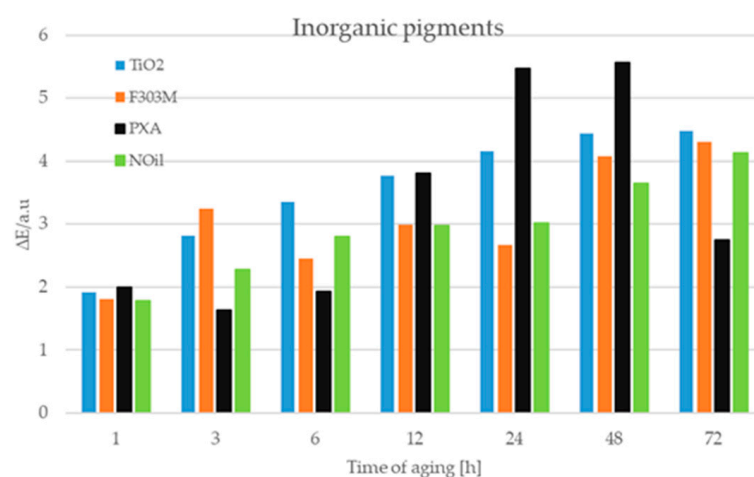


Figure 3. Dependence of color deviations on time of accelerated aging for NOil films with inorganic pigments.

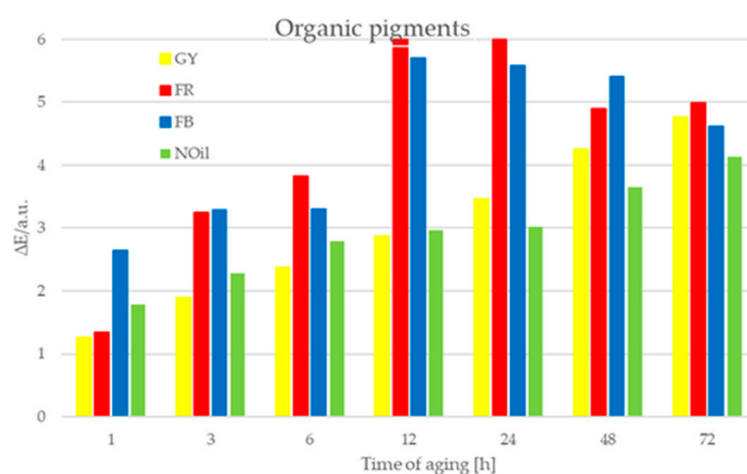


Figure 4. Dependence of color deviations on time of accelerated aging for NOil films with organic pigments.

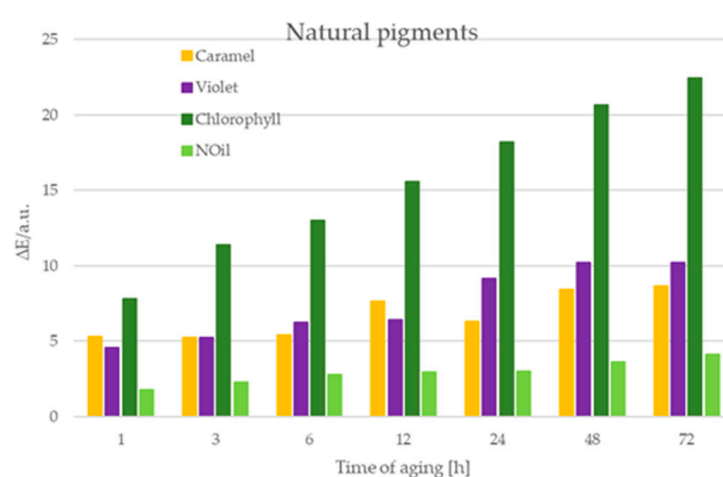


Figure 5. Dependence of color deviations on time of accelerated aging for NOil films with natural pigments.

The total color difference (ΔE) incorporates all three parameters—L, a, and b—and their changes during the aging process. Since accelerated aging is being applied, these results are intended to simulate the effects of prolonged real-time aging and to demonstrate

how each pigment behaves over time. To support this demonstration, we provide actual photographs of the prepared films.

Table 3. L, a, and b parameters from colorimetric trichromatic coordinates prepared film with inorganic pigment TiO₂.

Time Aging	L	Standard Deviation	a	Standard Deviation	b	Standard Deviation
0	90.63	0.64	−1.21	0.04	4.26	0.39
1	90.99	0.36	−0.97	0.02	2.39	0.25
3	91.49	0.45	−0.939	0.04	1.58	0.14
6	91.52	0.49	−0.95	0.06	1.04	0.23
12	91.39	0.49	−0.96	0.03	0.58	0.21
24	92.00	0.64	−0.91	0.04	0.34	0.12
48	91.37	0.85	−0.96	0.11	−0.12	0.29
72	91.78	0.63	−0.95	0.03	−0.07	0.21

For the inorganic and organic pigments, visible changes are not as pronounced as with natural pigments; in contrast, the changes for natural pigments are more visible. Therefore, images before and after aging are presented. These real photographs (Figure 6) point out the visible color changes that are quantified by the total color difference (ΔE), which is calculated based on the measured L, a, and b parameters and their mutual relationships, as defined by Equation (1), yielding the resulting ΔE value.

The obtained results indicate that all prepared films exhibited an increasing color change, expressed as an increase in ΔE values, with increasing aging time. The most pronounced color differences were observed in films containing natural pigments. For films with inorganic pigments, the color change with aging time was comparable to the natural color shift in the polymer itself. Films with organic pigments showed a more distinct deviation in color stability after some hours of aging, while films with natural pigments exhibited immediate changes, visible already after the first hour of accelerated aging, associated with the degradation of the natural colorants and a subsequent significant loss of coloration intensity.

Overall, the results confirm that thermo-light aging leads to an increase in color deviation (ΔE) in all films, reflecting a measurable color change. Among inorganic pigments, the highest ΔE was observed in films pigmented with TiO₂, whereas smaller changes were detected for films containing F303M and PXA pigments, consistent with the high thermal and light stability of inorganic pigments. For films containing organic pigments—yellow, red, and blue—the largest color deviation was observed for the blue and red pigments, after 12 h of aging, whereas the yellow pigment showed the greatest stability. The change in its color corresponded to the change in the color of the polymer matrix itself due to thermal-light aging. Films pigmented with organic pigments exhibited relatively high stability (low ΔE values), comparable to inorganic pigments, while films containing the natural pigment showed significantly higher deviations in color.

The monitoring of accelerated aging has been addressed in our previous publications [1], where we investigated the material properties of fibers containing only a single group of pigments. As stated at the beginning of this manuscript, the viscosity limitations of the pigment-filled materials prevented us from processing and directly comparing all pigment types in fibrous form.

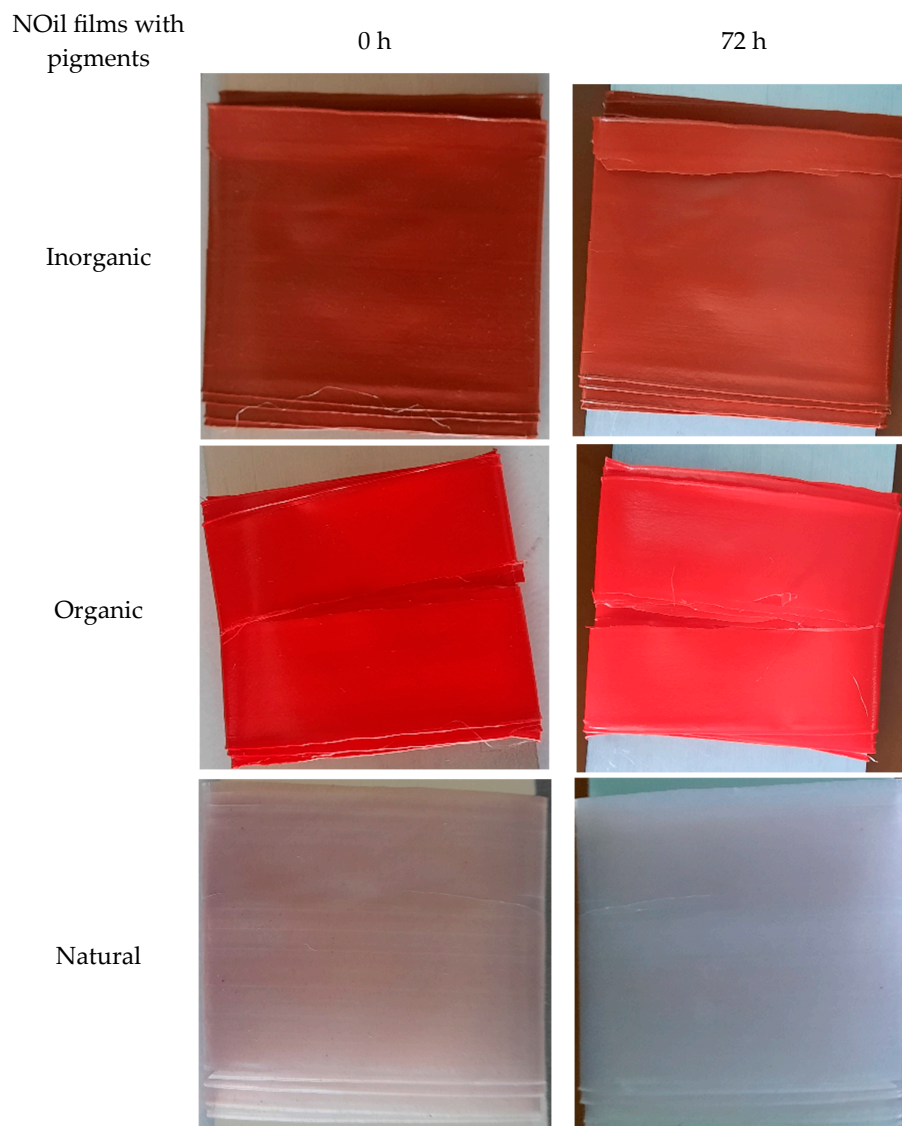


Figure 6. Real photographs of prepared films with the inorganic, organic, and natural-food pigments.

4. Conclusions

This study evaluated the impact of inorganic, organic, and food-natural pigments on the rheological, mechanical, and coloristic properties of NOil-based cast films. Rheological behavior for all pigmented masterbatches was written, where food-natural pigments, particularly chlorophyll, caused the most pronounced decrease in viscosity. Mechanical properties demonstrated that films containing inorganic pigments, especially TiO_2 , have higher tenacity at break; organic pigments achieved slightly lower mechanical properties, while food-natural pigments reduced the values of mechanical properties due to inhomogeneous dispersion and particle agglomeration. But all colored masterbatches achieved lower values of mechanical properties than pure NOil. Microscopic observations confirmed these structural irregularities, particularly in films with food-natural pigments, which acted as stress concentrators.

The present study was primarily focused on processing behavior and on the coloring performance of different pigment types. Even though the addition of pigments led to a certain decrease in the monitored processing-related parameters, the results nonetheless demonstrate that their use is beneficial for technological applications. Particular attention should be drawn to food-natural pigments, which may be especially suitable in this

context. However, future application-oriented research will inevitably be required to evaluate all relevant parameters for packaging films intended for food contact, including potential migration of dispersing agents or pigment components to the surface of the packaging material.

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Appendix A

Table A1. L, a, and b parameters from colorimetric trichromatic coordinates prepared film with inorganic pigment F303M.

Time Aging	L	Standard Deviation	a	Standard Deviation	b	Standard Deviation
0	32.73	0.33	34.91	0.31	31.20	0.47
1	33.22	0.39	34.22	0.36	29.61	0.83
3	32.93	0.5	33.68	0.37	28.21	0.96
6	33.4	0.36	34.0	0.38	29.0	0.6
12	33.50	0.6	33.87	0.36	28.52	1.02
24	33.34	0.57	34.00	0.36	28.77	0.4
48	34.32	0.63	33.75	0.41	27.63	0.82
72	34.34	0.58	33.67	0.34	27.41	0.82

Table A2. L, a, and b parameters from colorimetric trichromatic coordinates prepared film with inorganic pigment PXA.

Time Aging	L	Standard Deviation	a	Standard Deviation	b	Standard Deviation
0	5.70	0.5	−0.30	0.18	−0.32	0.2
1	7.57	1.24	−0.56	0.19	−0.98	0.36
3	7.20	1.01	−0.62	0.2	−0.93	0.35
6	7.49	1.11	−0.61	0.19	−0.98	0.37
12	9.32	2.78	−0.71	0.4	−1.42	0.68
24	10.90	3.16	−1.04	0.58	−1.85	0.84
48	11.04	2.58	−1.02	0.27	−1.72	0.44
72	8.25	1.61	−0.67	0.34	−1.29	0.44

Table A3. L, a, and b parameters from colorimetric trichromatic coordinates prepared film with inorganic pigment GY.

Time Aging	L	Standard Deviation	a	Standard Deviation	b	Standard Deviation
0	76.74	0.19	4.45	0.46	92.88	0.39
1	77.75	0.26	3.74	0.58	93.19	0.39
3	78.26	0.26	3.36	0.39	93.22	0.37
6	78.13	0.46	2.54	0.62	93.17	0.68
12	78.64	0.49	2.29	0.71	92.87	0.93
24	79.28	0.36	2.13	0.42	93.33	0.75

Table A3. *Cont.*

Time Aging	L	Standard Deviation	a	Standard Deviation	b	Standard Deviation
48	79.54	0.7	1.25	0.64	93.14	0.86
72	79.60	0.58	0.68	0.75	92.21	0.99

Table A4. L, a, and b parameters from colorimetric trichromatic coordinates prepared film with inorganic pigment FR.

Time Aging	L	Standard Deviation	a	Standard Deviation	b	Standard Deviation
0	38.80	0.58	63.86	0.48	46.62	1.29
1	39.16	0.48	63.81	0.53	45.33	0.96
3	39.58	0.79	63.42	0.5	43.50	1.36
6	39.70	1.09	63.02	0.45	43.01	2.32
12	40.38	1.4	62.60	0.63	40.77	2.72
24	40.66	1.14	63.09	0.52	40.98	2.56
48	40.16	0.93	63.14	0.6	41.97	1.65
72	39.15	0.88	62.06	1.61	41.97	1.52

Table A5. L, a, and b parameters from colorimetric trichromatic coordinates prepared film with inorganic pigment FB.

Time Aging	L	Standard Deviation	a	Standard Deviation	b	Standard Deviation
0	13.90	2.03	8.40	1.68	−34.15	1.99
1	16.15	1.94	7.03	1.47	−34.40	2.06
3	16.67	1.92	6.69	1.8	−34.56	1.16
6	16.59	2.15	6.50	1.95	−33.87	2
12	18.64	2.63	5.48	2.09	−32.91	2.08
24	18.57	2.95	5.47	2.32	−33.31	2.53
48	18.23	1.38	5.55	1.25	−32.58	2.45
72	17.59	2.19	6.09	1.66	−32.61	2.62

Table A6. L, a, and b parameters from colorimetric trichromatic coordinates prepared film with inorganic pigment violet.

Time Aging	L	Standard Deviation	a	Standard Deviation	b	Standard Deviation
0	46.55	1.5	3.24	0.36	1.86	0.49
1	50.60	1.9	2.80	0.29	−0.19	0.36
3	51.10	1.33	3.14	0.2	−0.70	0.17
6	52.04	1.37	3.12	0.2	−1.10	0.31
12	52.16	1.45	3.21	0.23	−1.29	0.17
24	55.17	1.14	2.88	0.3	−1.14	0.28
48	56.21	1.39	2.26	0.15	−1.40	0.4
72	56.00	1.49	1.96	0.31	−1.84	0.47

Table A7. L, a, and b parameters from colorimetric trichromatic coordinates prepared film with inorganic pigment caramel.

Time Aging	L	Standard Deviation	a	Standard Deviation	b	Standard Deviation
0	29.69	1.97	2.57	0.18	4.66	0.17
1	34.94	1.82	2.16	0.3	4.11	0.22
3	34.89	1.37	2.33	0.19	3.77	0.22
6	35.08	1.42	2.39	0.19	4.00	0.42
12	37.30	1.06	2.80	0.28	4.19	0.46
24	35.98	0.45	2.90	0.17	4.74	0.31
48	38.13	1.48	2.97	0.34	5.08	0.37
72	38.31	1.42	3.07	0.18	5.25	0.25

Table A8. L, a, and b parameters from colorimetric trichromatic coordinates prepared film with inorganic pigment chlorophyll.

Time Aging	L	Standard Deviation	a	Standard Deviation	b	Standard Deviation
0	40.65	1.19	−12.23	0.42	9.81	0.37
1	45.25	1.2	−6.32	0.25	7.50	0.35
3	48.96	2.06	−5.32	0.24	6.27	0.56
6	50.479	2.49	−4.948	0.25	5.394	0.51
12	52.69	2.94	−4.358	0.32	3.849	0.64
24	55.05	1.78	−3.67	0.32	2.71	0.46
48	57.23	2.45	−3.34	0.24	1.22	0.75
72	59.12	1.76	−3.13	0.27	0.85	0.43

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