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Modelling the degradation kinetics of naturally fermented table olives: Influence of packaging, brine, and temperature

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ABSTRACT

This study evaluated the effects of packaging material (glass jars vs. plastic bags) and brine type (filtered fermentation vs. reconstituted) on the shelf-life of fermented table olives (cv. *Nocellara Messinese*). Olives were pasteurised and stored at 20, 30, and 40 °C for 210 days. Quality parameters were monitored in both olives and brines through physicochemical, colorimetric, antioxidant, and texture analyses.

Significant declines in colour (L*, a*, b*, C*, BI), total polyphenols, antioxidant activity, and firmness were observed, particularly in plastic packaging. Glass jars with filtered fermentation brine better preserved phenolic content, antioxidant capacity, and visual/textural quality. pH remained below 4.2 in all samples, ensuring microbiological safety.

Degradation kinetics were modelled using zero- and first-order reactions and the Arrhenius equation.

Most parameters followed first-order kinetics, with temperature as a key factor. A novel integrated quality index (Texture/b*) was introduced to highlight imbalances between visual and mechanical degradation, showing high sensitivity to temperature-driven changes.

Overall, glass jars combined with filtered fermentation brine proved the most effective strategy for preserving sensory and functional attributes. Additionally, reusing fermentation brine emerged as a sustainable, cost-effective practice that reduces waste and enhances quality. The kinetic model supports shelf-life prediction and eco-innovative practices in the table olive industry.

1. Introduction

Table olives are among the most widely consumed fermented foods globally, valued for their distinctive sensory profile and nutritional properties. During the 2023/2024 season, global table olives production reached approximately 2828 million tons (IOC, 2024a). About one-quarter of this amount was produced and consumed in Europe, particularly in Spain, Greece, Italy, and Portugal, where the average annual per capita consumption within the EU is around 2.0 kg (IOC, 2024b).

According to the International Olive Oil Council (IOC, 2004c), table olives are classified based on commercial treatments and processing methods into natural olives, treated olives, dehydrated and/or shrivelled olives, olives darkened by oxidation, and specialties. These processing methods are primarily applied to reduce the natural bitterness caused by oleuropein, using techniques such as alkaline hydrolysis, brining/salting, fermentation by lactic acid bacteria, Gram-negative bacteria and yeasts, or acidification (Garrido-Fernández et al., 1997; Hurtado et al., 2012).

During fermentation, sugars are converted into lactic acid, leading to a decrease in pH, the production of aromatic compounds, improved microbiological safety, and enhanced durability and flavour. Thanks to their numerous sensory and nutritional qualities, table olives are considered a cornerstone of the Mediterranean diet, as highlighted by a study conducted by Conte et al. (2020).

Table olives can be preserved either by natural fermentation or thermal processing, with or without preservatives, and are typically packaged with or without a liquid covering. Previous studies conducted on the reuse of fermentation brine for olive preservation have demonstrated that this approach not only maintains product quality but also reduces environmental impact and production coast (Sánchez Gómez et al., 2006; Romeo et al., 2009). This makes brine reuse a valuable area of research for the food industry.

Packaging plays a key role in expanding the market and increasing

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the commercial value of table olives. Various strategies have been applied, including the use of additives to regulate pH, inhibit browning, or provide antioxidant protection (Casado et al., 2010; Arroyo-López et al., 2010; Benítez-Cabello et al., 2022), as well as modified atmospheres, heat treatments (Tzamourani et al., 2022; Piscopo et al., 2016), and innovative approaches such as high-pressure processing (HPP) (Conte et al., 2020; Martín-Vertedor et al., 2022). One of the main challenges in the table olive processing industry is to package the product post-fermentation while maintaining its quality throughout its shelf-life. Product stabilization must ensure that the olives retain their key characteristics until the declared expiration date. Consequently, reliable prediction of shelf-life is critical. Accelerated shelf-life testing (ASLT) combined with kinetic modelling represents an effective strategy to predict long-term stability.

In this work, naturally fermented olives in brine (*Nocellara Messinese* cv) were packaged using different container types (glass jar and plastic bags), two types of filling brine (reconstituted brine with 6 % NaCl acidified to pH 4; same fermentation brine after filtration), pasteurised and stored at three different temperatures (20, 30, 40 °C). The aim of this work is to evaluate the shelf-life of table olives stored under different storage conditions and to develop a predictive kinetic shelf-life model using an ASLT approach.

2. Material and methods

2.1. Samples and experimental design

The experimental plan was based on the use of naturally fermented olives in brine (*Nocellara Messinese* cv) after a 10-month fermentation period. Following fermentation, olives and brine were packaged either in 314 mL glass jars or in polyamide/polypropylene (PA/PP) plastic bags (20 × 30 cm). Each package contained 300 g of product, maintaining a 1:1 wt ratio (w:w) between olives and brine (i.e. 150 g of olives and 150 g of brine).

Two types of brine were used: a) corresponding fermentation brine (FFB) of experimental olives, filtered through filter paper (Whatman, pore size 7–12 μm. Merck, Germania); and b) reconstituted brine (RB), prepared by dissolving NaCl (6 % w/v) in water and acidifying the solution with food-grade lactic acid until a final pH of 4.0 was reached, as measured with a calibrated digital pH meter (Crison pH-meter, basic model 20).

The samples were organized as follows: olives in RB were packaged both in glass jars (sample A) and in plastic bags (sample B); olives in FFB were also packaged in glass jars (sample C) and plastic bags (sample D). Corresponding brine samples were collected and identified as follows: RB from glass jars (sample BA) and from plastic bags (sample BB); FFB from glass jars (sample BC) and from plastic bags (sample BD).

Finally, all samples were pasteurised at 70 °C for 8 min in an industrial oven (Angelo Po Combistar FX, Carpi, Modena, Italy), with core temperature monitoring performed using a MicroW data logger (Milan, Italy). The packages were then stored in dark conditions at 20, 30, and 40 °C in thermostatic chambers and monitored at different storage times.

2.2. Physicochemical analysis

Physicochemical analyses were carried out on both brine and olives samples. The determination of pH and titratable acidity (TA) in brines were carried out using the methods described by .(Sánchez et al., 1997) To measure olive pH, 10 g of homogenised sample were diluted in 50 mL of deionised water, kept under agitation for 1 h at room temperature, and then measured with a pH meter (Crison pH-meter, basic model 20).

For TA, extracts were centrifuged (NF 1200R, Nüve, Ankara, Turkey) at 8000 rpm for 5 min at 4 °C. The resulting supernatant was filtered, through filter paper, and 5 mL of filtered extract were titrated with 0.01 N NaOH using phenolphthalein as indicator. Results were expressed as % of lactic acid.

The colour analysis of the drupe surface was measured with a Minolta CM-700d Spectrophotometer (CIE L*a*b* coordinates). For brine, measurements were taken in a Minolta glass cell. For olives, forty measurements were taken across twenty drupes. Chroma (C*), hue angle (h°), and browning index (BI) were calculated according to standard equations:

$$C^* = \left(a^2 + b^2\right)^{1/2} \quad (1)$$

$$h^\circ = \arctan(b^* / a^*) \quad (2)$$

$$BI = \frac{100\left(\frac{(a^*+1.75 \text{ x } L^*)}{(5.645 \text{ x } L^*+a^*-0.3012 \text{ x } b^*)} - 0.31\right)}{0.17} \quad (3)$$

2.3. Extraction of antioxidant compounds from olives

The extraction of antioxidant compounds from olive pulp was performed according to the method reported by De Bruno et al. (2021), with some modifications. Two grams of olive pulp were combined with 2 mL of hexane, used to remove the lipid fraction, and 10 mL of a Methanol/Water solution (70:30, v/v), suitable for the extraction of phenolic compounds. The resulting mixture was homogenised using an Ultra-Turrax (IKA T 25 digital ULTRA-TURRAX Staufen, Germany) and then centrifuged at 9000 rpm for 5 min at 4 °C. Following centrifugation, the hydroalcoholic phase, containing the phenolic fraction, was carefully collected using a syringe and filtered through a regenerated cellulose (RC) membrane filter prior to analysis.

2.3.1. Pulp and brine polyphenols

The concentration of total polyphenols (TPC) on both olives and brine was determined according to the method reported by De Bruno et al. (2020), with some modifications. In a 25 mL volumetric flask, a defined aliquot of extract was mixed with 2.5 mL of a MeOH/H_20 solution (80:20), followed by the addition of 0.625 mL of Folin Ciocalteau and 1.250 mL of Na_2CO_3 (20 %). This was made up to volume with deionised water. The samples once prepared were stored in the dark overnight. The absorbance was read at 725 nm. Results were expressed as mg gallic acid per 100 g^{-1} of olives dry weight (mg 100 g^{-1} dw) and per 100 mL^{-1} of brine.

2.3.2. Antioxidant activity

Antioxidant activity was assessed by DPPH and ABTS assays. DPPH assay was conducted following Brand-Williams et al. (1995). A mixture of 2.980 mL methanolic DPPH and 20 μL extract was incubated in the dark at room temperature for 30 min under stirring, and absorbance was read at 515 nm (PerkinElmer UV–Vis λ2). The ABTS assay was tested adding an aliquot of olive extract or brine added to the ABTS solution as reported by Gattuso et al. (2023). The absorbance was measured at 734 nm. For the assays, results were expressed as μmol Trolox g^{-1} of olives dry weight (μmol Trolox g^{-1} dw) and per μmol Trolox 100 mL^{-1} of brine.

2.4. Textural analysis

The texture analysis was carried out using the Texture Analyser (TA-XT plus - Stable Micro Systems, Godalming - UK), in combination with the data integration software 'Exponent 6.1.4.0' (Stable Micro Systems, Godalming - UK), for processing the data obtained from the measurements. For the execution of the penetration test, a cylindrical probe of 2 mm diameter (P/2, Stable Micro Systems) was used. The operating parameters set in the texture analyser were as follows as reported by Romeo et al. (2009) with some modification: pre-test speed: 1.0 mm/s, test speed: 2.0 mm/s, post-test speed: 10.0 mm/s, distance: 6.0 mm, trigger Force: 0.05 g. The measurements were carried out on 10 olives

per sample. The firmness was expressed in Newton (N).

2.5. Degradation kinetics

The results obtained from the three storage temperatures (20°-30°-40 °C) in colour changes and firmness in olives during storage adapting different kinetic models that describe quality changes. These changes were modelled as zero or first order reaction, as reported for food product (Labuza, 1984) according to the following equations:

For the zero order:

$$dA / dt = -k\,t \quad (4)$$

integrating equation (4):

$$A = A_0 - k^*t \quad (5)$$

For the first order:

$$dA / dt = -k^*A \quad (6)$$

integrating equation (6):

$$\ln(A / A_0) = -k^*t \quad (7)$$

where.

- dA/dt is the quality parameter (L*, a*, b*, C*, BI, firmness, firmness/b*) degradation per unit of time;
- k is rate constant (day^{-1});
- A is the parameter value at any given time;
- A_0 is the quality parameter value at the initial time (t = 0, day);
- t is the storage time (day^{-1}).

In addition, it has been considering the Arrhenius model to determine activation energy (Ea) of the degradation reaction as follow:

$$k = k_{-}(0)\ \exp\hat{}(-Ea / (R^*T)) \quad (8)$$

and linearizing equation (8):

$$\ln(k) = \ln(k_0) - (Ea / R)^*(1 / T) \quad (9)$$

here.

- k_0 is the pre-exponential factor;
- Ea is the activation energy (kJ/mol);
- R is the universal gas constant (8.314 J/mol K);
- T is the absolute temperature (Kelvin).

Standard error (SE) was also reported.

2.6. Statistical data analysis

All analyses were conducted in triplicate (n = 3), and the experimental results for samples stored at 20 °C are presented as mean ± standard deviation. Significant differences between mean values ($p < 0.05$) were assessed using one-way analysis of variance (ANOVA) and Multivariate analysis with SPSS Software (Version 15.0, SPSS Inc., Chicago, IL, USA). Multiple comparisons were performed using Tukey's post hoc test to identify individual significant differences ($p < 0.05$).

3. Results and discussion

3.1. Physicochemical properties of packed olives and brine

3.1.1. pH and titratable acidity (TA) in olive and brine samples

One of the key parameters useful for evaluating the quality and safety of table olives in brine is pH. In this study, of the pH values of both olives and brine remained consistently below 4 throughout the storage period (Fig. 1). These conditions are considered microbiologically safe, as they inhibit the growth of spoilage and pathogenic microorganisms that typically proliferate at pH ≥ 4.2. These values were kept below this threshold throughout the storage of samples in brine and olives, while respecting the limit values for olives in brine packed in hermetically sealed containers (IOC, 2004c). These characteristics indicate that the product was stable, and that secondary fermentation did not occur. This valuable aspect can be attributed to the thermal stabilization treatment and the adequate sealing of the containers.

The TA expressed as % of lactic acid, also play a critical role in ensuring the safety and shelf stability of preserved olives. Although the addition of edible acids to correct brine is a widely used operation (Medina-Pradas et al., 2017), a significant decrease was observed in samples A and B after 210 days showing the strong influence of the type of brine and less than the type of packaging for this parameter (Fig. 1). This decrease could be due to a diffusion of some acid compounds from olives to brine, this is proved by the concomitant increase of acidity in

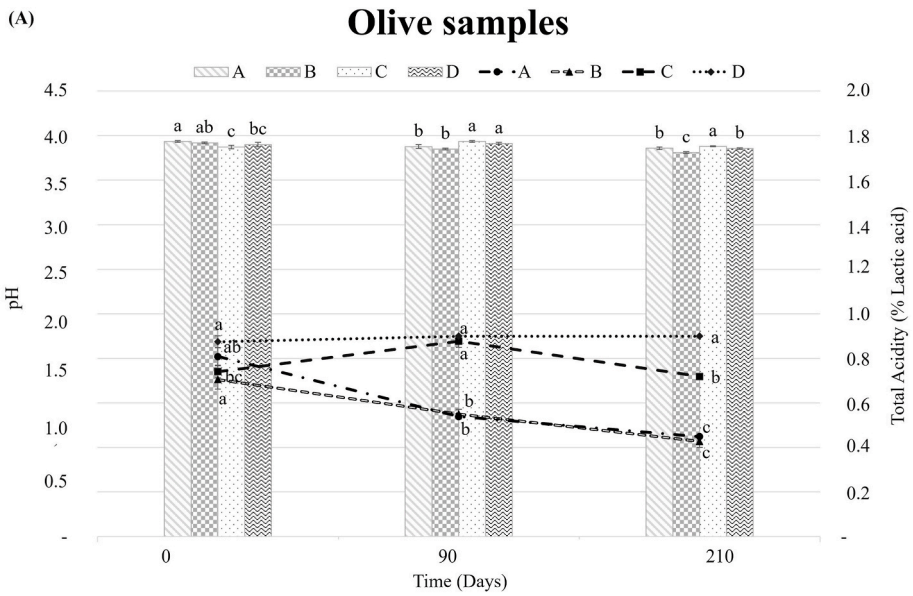

Fig. 1.A. Changes in pH and TA in olives during storage period: histograms (left Y-axis) for pH; lines (right Y-axis) for TA. Small letters differences as assessed by Tukey's post hoc test among samples.

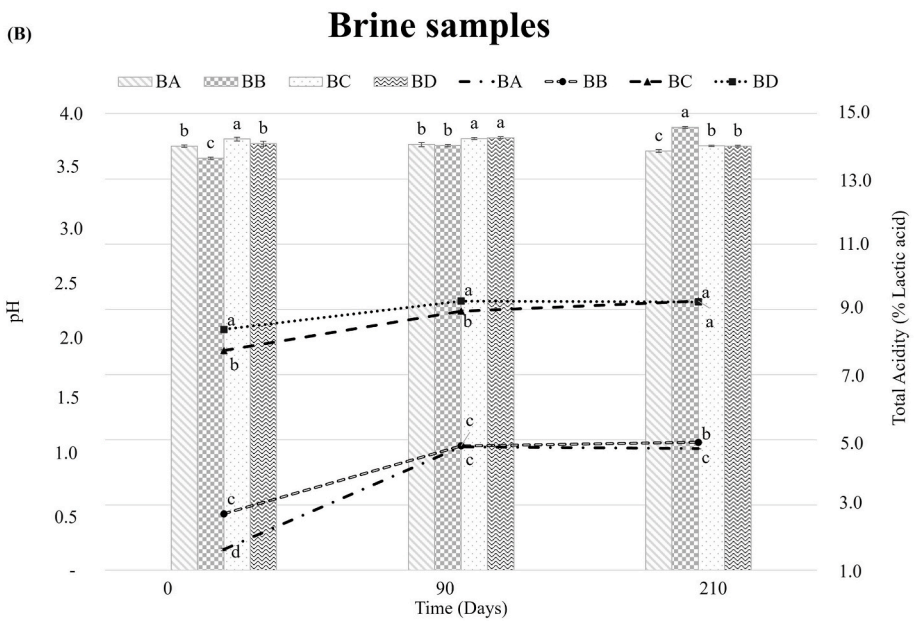

Fig. 1.B. Changes in pH and TA in brines during storage period: histograms (left Y-axis) for pH; lines (right Y-axis) for TA. Small letters differences as assessed by Tukey's post hoc test among samples.

reconstituted brine. The reconstitution of brine at pH 4 with lactic acid only has not brought to a stable system, otherwise the fermented brine used as governing brine is more stable and has not given evidence of diffusion processes. Considering the brine acidity, results showed the acidification effect during storage with significant increases in all samples, highlighting a higher acidity in the fermentation brine. This is related to a higher concentration of acids and compounds probably resulting from biochemical processes such as fermentation. These acidity variations did not markedly affect pH, owing to the buffering effect of brine (Panagou, 2004).

3.1.2. Colour changes in olive and brine samples during storage

Analysis of lightness (L*) (Table 1) revealed significant differences across treatments and storage times, except at the initial measurement ($p > 0.05$). Olives stored in glass jars (samples A and C) maintained the highest L* values, showing stable values over time. This parameter is probably strongly influenced by the packaging materials, as the glass provides better protection against oxidative and thermal stress. In contrast, a moderate decrease in brightness was observed in samples stored in plastic PA/PP (B and D) bags which are comparable at the end of the storage period.

The redness coordinate (a*) increased significantly in all samples, particularly in plastic bags (B and D), highlighting the effect of packaging material on colour evolution. Conversely, yellowness (b*), showed a significant decrease over time in all samples. Confirming the previous colour evidence, a greater degradation of the yellow pigments was observed in samples B and D, those packed in plastic bags. The combined use of filtered fermentation brine and glass jar (sample C) resulted in a better preservation of the carotenoid pigments.

Generally, changes in colour are probably related to the different heat transmission and oxygen permeability favouring a greater degradation of the pigments responsible for the colour. The colorimetric coordinates indicate a "darkening" of the surface colour of olives (Rejano et al., 1995), which as has been observed by other authors may be due to degradation of pigments, in particular compounds derived from chlorophyll, such as pheophytin (Mínguez-Mosquera et al., 1991).

The derived parameters chroma (C*) and hue angle (h°), which describe the saturation and tone of colour respectively, also showed a significant reduction over time. The most marked decline occurred in samples stored in plastic bags, confirming a greater colorimetric degradation compared to glass-packaged olives, which retained better colour stability. This change indicates a colorimetric degradation of the olives towards darker shades depending on the packaging used. The colour resulted by the changes of fermentation decreased following the trend of C* and h°.

Significant differences were also observed in the three colorimetric coordinates L*, a* and b* in the brine (Table 1). These alterations are probably related to different chemical transformation factors such as oxidation, the migration of pigments and other organic compounds such as polyphenols from olives. After 90 days of storage, a slight increase in lightness was recorded, indicating a clarification and stabilization of brine. At the end of storage period, sample BC and BD showed the highest and most similar values. A reduction in BA was observed, showing that among the samples, reconstituted brine maintained better lightness although with very similar values. As regards redness (a*) a fluctuation during storage was observed in all samples with an initial increase followed by a reduction in all samples. BB and BD showed the same redness after 210 days. All samples maintained negative values of b* during storage, with the highest values in samples with ex novo brine to T0. In addition, this parameter followed the same trend with a significant reduction for all samples with values close to zero indicator of brine's yellowing probably related to the oxidation of the pigments. The main differences in the colour of the brine are certainly related to the factors described above, particularly the type of brine used. It is well known that fermentation brine contains phenolic compounds and other constituents that migrate from the olives, thereby altering its colour (Ardic & Aktas, 2023; Minguez-Mosquera et al., 1989).

3.1.3. Phenolic content and antioxidant activity of olives and brines

Regarding the total polyphenol content, a significantly higher reduction was observed in the sample stored in glass jar and regenerated brine (sample A) after 90 days of storage, after which it remained stable until the end of the experiment. In contrast, stable TPC were observed in samples B and C, with the highest value detected in sample C after 210 days (807.13 mg GAE 100 g^{-1} dw). Sample D showed a gradual decrease over time (21 %) yet maintained a high TPC value (725.86 mg GAE 100 g^{-1} dw). This loss of polyphenols may be due either degradation and oxidation phenomena or to the transfer of the compounds into the brine. In fact, it can be observed that TPC increases in the brine samples with ex novo brine (samples BA and BB) and decreases significantly ($p > 0.05$) in

Table 1
Evolution of colour parameters in olive and brine samples during storage.

		Time	A	B	C	D	Sign
Olive	L*	**0**	48.46 ± 4.3^{a}	44.98 ± 3.2^{bAB}	48.3 ± 3.38^{a}	48.2 ± 3.4^{aA}	**
		90	47.88 ± 1.97^{b}	46.12 ± 1.34^{cA}	49.82 ± 2.33^{a}	46.15 ± 1.90^{cB}	**
		210	47.87 ± 2.3^{a}	43.98 ± 2.22^{bB}	49.79 ± 3.24^{a}	43.23 ± 1.79^{aC}	**
		Sign	ns	**	ns	**	
	a*	**0**	5.16 ± 0.97^{abB}	4.65 ± 0.84^{bC}	5.4 ± 0.91^{aB}	5.39 ± 1.04^{aB}	**
		90	5.82 ± 0.67^{A}	6.04 ± 0.69^{B}	5.98 ± 0.9^{A}	6 ± 0.72^{A}	ns
		210	5.53 ± 1.03^{cAB}	6.47 ± 0.77^{aA}	5.99 ± 0.66^{bA}	6.29 ± 0.68^{abA}	**
		Sign	**	**	**	**	
	b*	**0**	17.01 ± 2.65^{bA}	14.99 ± 2.7^{cA}	18.65 ± 3.01^{aA}	18.18 ± 3.2^{abA}	**
		90	14.67 ± 2.42^{bB}	12.3 ± 1.95^{cB}	16.33 ± 2.27^{aB}	12.32 ± 1.76^{cB}	**
		210	14.14 ± 1.73^{bB}	11.16 ± 2.35^{cB}	16.02 ± 2.03^{aB}	10.51 ± 1.79^{cB}	**
		Sign	**	**	**	**	
	C*	**0**	17.8 ± 2.7^{bA}	15.74 ± 2.54^{cA}	19.46 ± 2.87^{aA}	18.99 ± 3.16^{abA}	**
		90	15.83 ± 2.22^{bB}	13.75 ± 1.74^{cB}	17.43 ± 2.17^{aB}	13.73 ± 1.67^{cB}	**
		210	15.23 ± 1.66^{bB}	12.96 ± 2.17^{cB}	17.13 ± 1.91^{aB}	12.27 ± 1.78^{cC}	**
		Sign	**	**	**	**	
	h°	**0**	73.07 ± 2.55^{A}	72.29 ± 4.94^{A}	73.51 ± 3.87^{A}	73.26 ± 3.55^{A}	ns
		90	67.93 ± 4.4a^{B}	63.47 ± 4.49^{bB}	69.67 ± 3.73^{aB}	63.82 ± 3.68^{bB}	**
		210	68.51 ± 4.38^{aB}	59.36 ± 5.12^{bC}	69.3 ± 3.29^{aB}	58.83 ± 3.32^{bC}	**
		Sign	**	**	**	**	
	BI	**0**	50.74 ± 8.81^{bcA}	48.14 ± 9.88^{cA}	56.67 ± 10.23^{aA}	55.28 ± 11.14^{abA}	**
		90	44.96 ± 5.8^{bB}	40.36 ± 5.98^{cB}	47.94 ± 5.5^{aB}	40.06 ± 3.87^{cB}	**
		210	43.12 ± 5.29^{bB}	39.95 ± 7.96^{bcB}	47.15 ± 5.76^{aB}	38.02 ± 4.72^{cB}	**
		Sign	**	**	**	**	
Brine		**Time**	**BA**	**BB**	**BC**	**BD**	**Sign**
	L*	**0**	34.91 ± 0.15^{aB}	34.81 ± 0.16^{bB}	34.87 ± 0.29^{C}	34.9 ± 0.07^{aC}	ns
		90	35.29 ± 0.07^{aA}	34.97 ± 0.02^{cBA}	35.25 ± 0.16^{B}	35.06 ± 0.07^{bB}	**
		210	34.48 ± 0.39^{cC}	35.05 ± 0.31^{bC}	35.49 ± 0.4^{A}	35.52 ± 0.07^{aA}	*
		Sign	**	**	ns	**	
	a*	**0**	−0.11 ± 0.02^{aB}	−0.12 ± 0.02^{aC}	−0.16 ± 0.04^{bB}	−0.23 ± 0.04^{cB}	**
		90	0.05 ± 0.06^{aA}	0 ± 0.05^{bA}	0.01 ± 0.12^{abA}	−0.06 ± 0.03^{cA}	**
		210	−0.15 ± 0.05^{bC}	−0.05 ± 0.03^{aB}	−0.2 ± 0.05^{cB}	−0.05 ± 0.04^{aA}	**
		Sign	**	**	**	**	
	b*	**0**	−1.73 ± 0.1^{cC}	−1.75 ± 0.06^{cC}	−1.39 ± 0.1^{aC}	−1.62 ± 0.06^{bC}	**
		90	−0.55 ± 0.1^{bB}	−0.58 ± 0.06^{bB}	−0.41 ± 0.23^{aB}	−0.55 ± 0.03^{bB}	**
		210	−0.11 ± 0.08^{bA}	−0.07 ± 0.07^{abA}	−0.16 ± 0.04^{cA}	−0.05 ± 0.06^{aA}	**
		Sign	**	**	**	**	

Small letters within a column and capital letters within a row show significant differences as assessed by Tukey's post hoc test. Abbreviations: **, significance at $p < 0.01$; *, significance at $p < 0.05$; ns, significance at $p > 0.05$.

sample BD without statistical differences (Fig. 2). The phenol content detected were consistent with those reported by Timpanaro et al. (2023) in *Nocellara Messinese* olives.

In filtered fermented brines (BC and BD), higher TPC were detected compared to BA and BD throughout the entire storage period. Brine composition also plays a key role in TPC retention. After 210 days, between the two types of brine the glass showed higher values than the same brine preserved in PA/PP bags. FFB demonstrated greater stability compared to RB, likely due to its original composition, which is naturally enriched with phenolic compounds, such as hydroxytyrosol, released by olives during the spontaneous fermentation process. These phenolic compounds are known to exert antimicrobial and antioxidant effects, contributing not only to the microbiological stability of the medium, but also to the protection of olive quality over time (Kara, 2023). In contrast, RB, composed solely of water and salt, lacks these bioactive compounds at the start, and can only acquire functional properties progressively through compound migration from the olives. This difference in composition and functional activity may explain the superior performance of FFB in preserving phenolic content, colour, and texture of the product during storage.

The antioxidant capacity of olives and brines was measured by DPPH and ABTS assays, which measure the radical scavenging capacity of antioxidants (Fig. 3a and b). Results for both tests were expressed as μmol Trolox g^{-1} dw, with generally higher values observed in the ABTS assay. This is because the DPPH assay reacts with larger classes of both hydrophilic and lipophilic compounds. The DPPH assay showed values ranging from 7.88 μmol Trolox g^{-1} dw in sample B at initial time, and just over 15 μmol Trolox g^{-1} dw in samples C and D. A significant increase over time was observed in all samples, likely due to the hydrolysis of phenolic compounds, which enhances free radical reactivity. This increase is attributed e to the diffusion of phenolics from the olive into the brine during fermentation as observed by Chranioti et al. (2018).

About the ABTS assay, over initial values were detected in sample B. Samples stored in regenerated brine exhibited a significant decrease in antioxidant capacity over time, ultimately showing the lowest values among all tested samples. This decline may be attributed to the higher solubilization and diffusion of antioxidant compounds into the brine. In contrast, samples preserved in their original fermentation brine did not exhibit significant changes over the storage period. The results obtained for the antioxidant properties were like those reported by Cano-Lamadrid et al. (2017).

A similar trend to TPC was observed for the DPPH assay in which the decrease of antioxidant activity was significant for BB ($p > 0.05$) and BD ($p > 0.01$) while no significant change was recorded in BA and BC confirming the protection effect of the glass jar. The antioxidant activity analysed by ABTS increased significantly for all samples over time. This indicates that the biochemical transformations taking place in the bioactive compounds present in the brine, improve the radical scavenging properties.

3.2. Texture properties of packed olives

The structure integrity of food products is one of the main quality indicators. Turgidity and tissues crunchiness are directly associated with product freshness and perceived healthiness, making them key sensory attributes valued by consumers. Objective assessment of these properties can be achieved through texture analysis using specialized equipment and suitable probes. In this study, firmness was used as the main texture parameter.

Firmness data collected at initial time (T0), after heat treatment, showed no statistically significant differences among the tested samples. However, a progressive decline in firmness was observed in all samples during storage (Fig. 4). Notably, significant reductions were recorded in samples B and D, both packaged in plastic bags, suggesting a relationship between packaging type and texture degradation. For instance, Lombardi et al. (2018) observed a reduction in texture during storage of green table olives, and similar trend was also found by (García-García et al., 2014) in packed ripe table olives who highlighted packaging-dependent firmness loss.

The packaging material emerged as a critical factor in firmness retention, with glass jars (samples A and C) showing superior

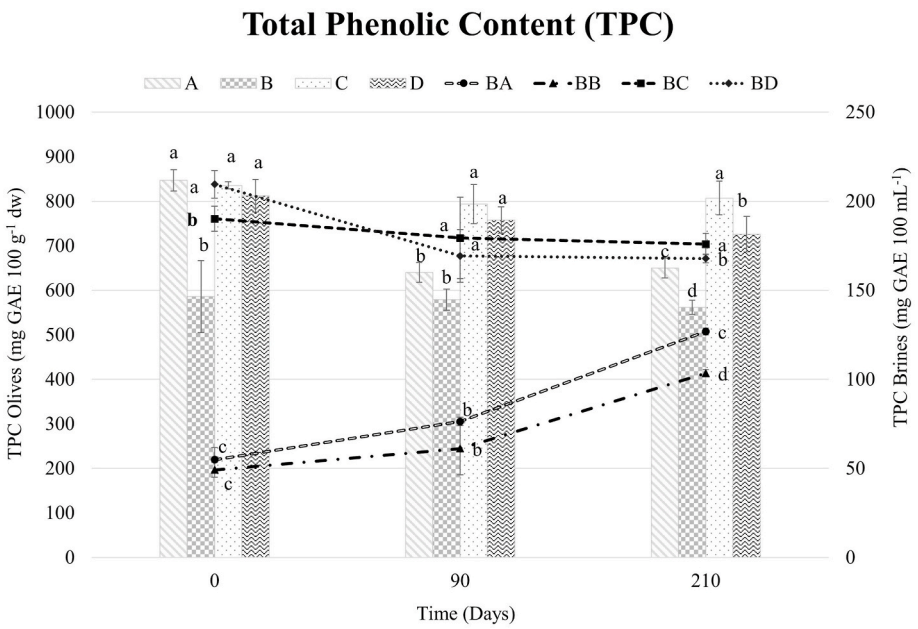

Fig. 2. TPC results in olives and brine during storage period: histograms (left Y-axis) for olives; lines (right Y-axis) for brine. Small letters differences as assessed by Tukey's post hoc test among samples.

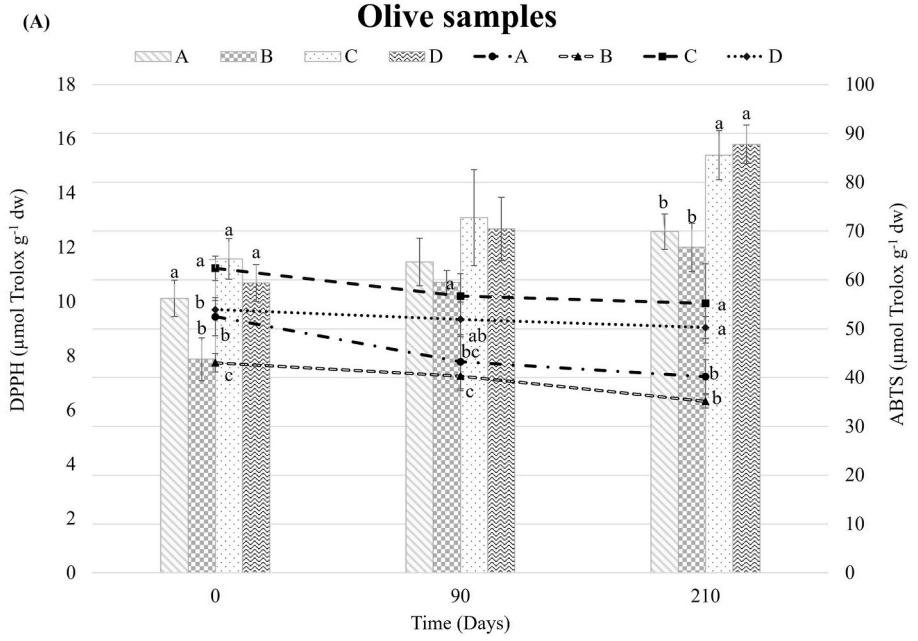

Fig. 3.A. DPPH and ABTS assays' results in olives during storage period: histograms (left Y-axis) for DPPH; lines (right Y-axis) for ABTS. Small letters differences as assessed by Tukey's post hoc test among samples.

performance in preserving structural integrity. Additionally, the use of filtered fermented brine appears to have contributed positively to the preservation of olive texture.

The multivariate analysis (Table 2) highlighted that the main factors sample, brine, and packaging significantly affected all quality parameters ($p < 0.01$). In contrast, time alone had a limited effect, showing significance only for pH ($p < 0.05$), indicating that its influence emerges primarily in interaction with other variables.

3.3. Study of kinetic degradation

The evolution of all quality parameters was modelled using zero-order and first-order kinetics. For each model, the degradation rate constants (k), intercepts, coefficients of determination (R^2), and standard errors were calculated at three storage temperatures (20 °C, 30 °C, 40 °C). From the velocity rates obtained from the three temperatures, moreover, the activation energy and R^2 were calculated by mean of Arrhenius' equation. These data make it possible to better investigate the course of degradation at three different temperatures.

In the case of zero-order kinetics, the speed is independent of the concentration of the parameter analysed, whereas in first order, the speed is proportional to the value of this parameter. In addition, through the Ea, obtained from the Arrhenius model indicates how sensitive this phenomenon is to temperature. In general, as already noted, colour is a

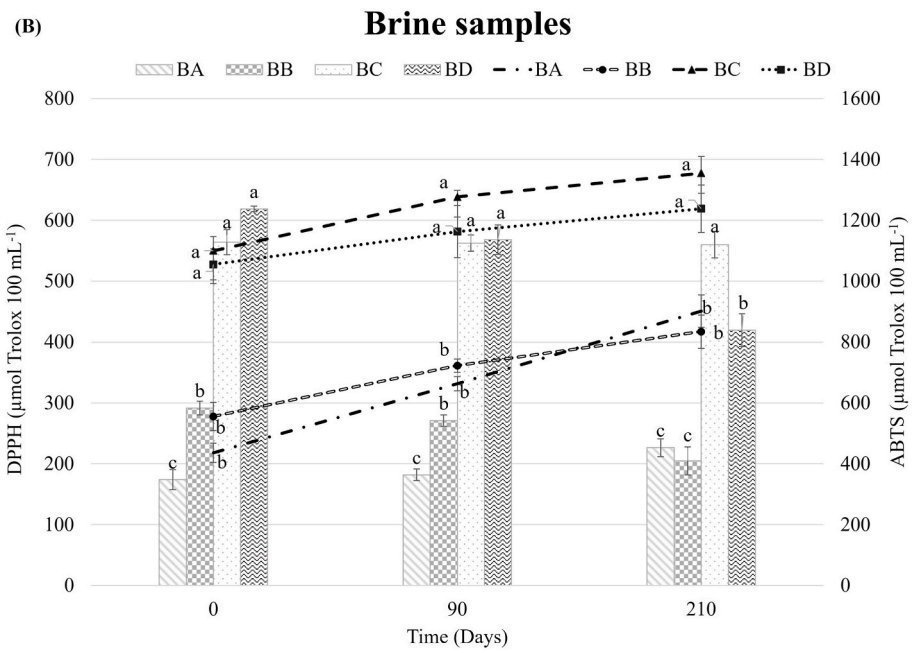

Fig. 3.B. DPPH and ABTS assays' results in brines during storage period: histograms (left Y-axis) for DPPH; lines (right Y-axis) for ABTS. Small letters differences as assessed by Tukey's post hoc test among samples.

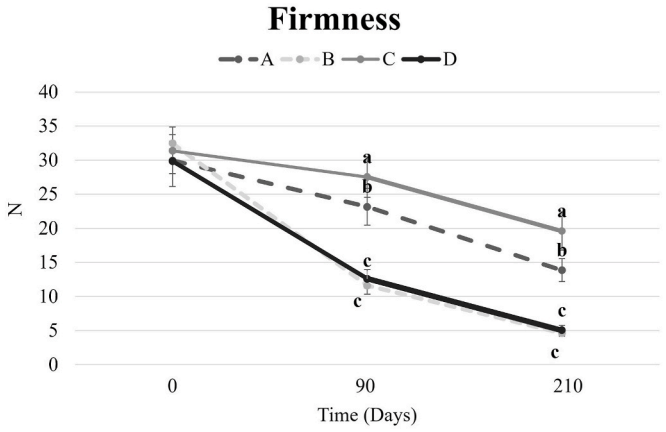

Fig. 4. Firmness properties of olives during storage. Small letters differences as assessed by Tukey's post hoc test among samples.

key parameter to verify the shelf-life of products. Olives are subject to browning phenomena, related to oxidation, degradation of pigments and other chemical physical transformations, thus determining significant changes over time that can be carefully studied.

For the b* parameter in sample A, a better fit was observed at 30 °C ($R^2 > 0.900$), although acceptable correlation ($R^2 > 0.700$) was observed at 20 and 40 °C for both kinetic orders, with higher Ea for the first reaction order (Table 3). Sample B showed a consistent fit with first-order kinetics at all temperatures ($R^2 = 0.778$ at 20 °C; 0.794 at 30 °C; and 0.933 at 40 °C). Less linear values were found for the sample C at 20 °C, although at higher temperatures the values of R^2 were quite good (between 0.884 and 0.899) and a trend that would seem to show a similar trend, but with a greater orientation towards the first order. For sample stored in plastic bags and original brine (sample D), a good fit was found for both orders but R^2 above all temperatures for the first order.

As observed for this parameter, the rate of degradation (k) increases significantly with increasing temperature, and the kinetics of degradation seems to follow a first order kinetics, with R^2 values generally higher, a good similarity between the Ea values of the samples, and a

Table 2
Multivariate statistical analysis of olive qualities.

Dependent Variable	pH	TA	L*	a*	b*	C*	h°	BI	TPC	DPPH	ABTS	Texture
Sample	**	**	**	**	**	**	**	**	**	**	**	**
Brine	**	**	**	**	**	**	**	**	**	**	**	**
Pack	**	**	**	**	**	**	**	**	**	**	**	**
Time	*	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns
Sample*Brine	**	**	**	**	**	**	**	**	**	**	**	**
Sample*Pack	**	**	**	**	**	**	**	**	**	**	**	**
Sample*Time	**	**	**	**	**	**	**	**	**	**	**	**
Brine*Pack	**	**	**	**	**	**	**	**	**	**	**	**
Brine*Time	**	**	**	**	**	**	**	**	**	**	**	**
Pack*Time	**	**	**	**	**	**	**	**	**	**	**	**
Sample*Brine*Pack	**	**	**	**	**	**	**	**	**	**	**	**
Sample*Brine*Time	**	**	**	**	**	**	**	**	**	**	**	**
Sample*Pack*Time	**	**	**	**	**	**	**	**	**	**	**	**
Brine*Pack*Time	**	**	**	**	**	**	**	**	**	**	**	**
Sample*Brine*Pack*Time	**	**	**	**	**	**	**	**	**	**	**	**

Significance: n.s. not significant; ** Significance at $p < 0.01$; * Significance at $p < 0.05$.

Table 3
Kinetic modelling of b and Chroma (C) degradation in olive samples: zero-order, first-order, and Arrhenius model
T = Temperature (°C); k = rate constant (day^{-1}); Intercept = linear regression intercept; R^2 = coefficient of determination; SE = standard error; Ea = activation energy from Arrhenius plot (kJ/mol).

b*														
	Zero order					Arrhenius Plot		First order					Arrhenius Plot	
	T	k	Intercept	R^2	SE	R^2	Ea	T	k	Intercept	R^2	SE	R^2	Ea
A	20	−0.0134	16.50	0.704	0.85	**0.893**	72.55	20	−0.0009	2.80	0.717	0.05	**0.889**	77.29
	30	−0.0203	16.84	0.910	0.64			30	−0.0013	2.82	0.917	0.04		
	40	−0.0909	16.04	0.830	1.23			40	−0.0066	2.78	0.865	0.08		
B	20	−0.0215	15.14	0.734	1.27	**0.943**	68.15	20	−0.0016	2.72	0.778	0.08	**0.990**	78.74
	30	−0.0371	12.28	0.680	2.11			30	−0.0039	2.50	0.794	0.17		
	40	−0.1292	13.78	0.876	1.45			40	−0.0128	2.64	0.933	0.10		
C	20	−0.0131	18.29	0.671	0.90	**0.706**	67.21	20	−0.0008	2.91	0.681	0.05	**0.705**	69.33
	30	−0.0123	18.36	0.893	0.43			30	−0.0007	2.91	0.899	0.02		
	40	−0.0782	19.24	0.896	0.80			40	−0.0048	2.96	0.884	0.05		
D	20	−0.0297	15.85	0.678	2.00	**0.939**	65.10	20	−0.0022	2.76	0.735	0.13	**0.970**	69.10
	30	−0.0494	15.17	0.704	2.66			30	−0.0043	2.71	0.791	0.18		
	40	−0.1650	16.72	0.897	1.67			40	−0.0135	2.83	0.960	0.08		
Chroma (C*)														
A	20	−0.0129	17.51	0.671	0.88	**0.870**	73.48	20	−0.0008	2.86	0.683	0.05	**0.868**	78.31
	30	−0.0184	17.35	0.633	1.16			30	−0.0011	2.85	0.643	0.07		
	40	−0.0896	17.12	0.877	1.00			40	−0.0061	2.84	0.898	0.06		
B	20	−0.0177	16.20	0.632	1.32	**0.946**	72.53	20	−0.0012	2.78	0.673	0.08	**0.983**	82.54
	30	−0.0320	13.57	0.722	1.65			30	−0.0029	2.61	0.807	0.12		
	40	−0.1193	15.02	0.922	1.03			40	−0.0104	2.72	0.945	0.08		
C	20	−0.0125	19.30	0.661	0.87	**0.724**	69.16	20	−0.0007	2.96	0.673	0.05	**0.724**	71.45
	30	−0.0122	19.32	0.895	0.42			30	−0.0007	2.96	0.898	0.02		
	40	−0.0781	20.27	0.838	1.03			40	−0.0045	3.02	0.832	0.06		
D	20	−0.0263	16.93	0.659	1.85	**0.944**	68.15	20	−0.0018	2.82	0.699	0.11	**0.969**	72.51
	30	−0.0456	16.18	0.676	2.62			30	−0.0035	2.77	0.738	0.17		
	40	−0.1582	17.80	0.925	1.35			40	−0.0118	2.89	0.973	0.06		

great reliability of the Arrhenius model. These results suggest that temperature accelerates degradation, especially in olives stored in plastic bags, which consequently exhibit reduced shelf-life compared to those stored in glass jars. Furthermore, brine type did not significantly affect the shelf-life related to the b* parameter, particularly in glass jars, where the degradation followed a linear trend consistent with first-order kinetics. For the C* value (colour saturation, derived from a* and b*), a strong fit with the Arrhenius model was observed, indicating high temperature sensitivity (Table 3).

In sample A, R^2 values around 0.870 for both orders at 40 °C

Table 4
Kinetic modelling of Browning Index (BI) and texture degradation in olive samples: zero-order, first-order, and Arrhenius model.

Browning Index (BI)														
	Zero order					Arrhenius Plot		First order					Arrhenius Plot	
	T	k	Intercept	R^2	SE	R^2	Ea	T	k	Intercept	R^2	SE	R^2	Ea
A	20	−0.1243	29.27	0.919	3.61	**0.799**	43.65	20	−0.0088	3.47	0.982	0.12	**0.796**	41.62
	30	−0.1372	26.66	0.846	4.85			30	−0.0097	3.32	0.956	0.17		
	40	−0.3944	28.08	0.973	1.97			40	−0.0265	3.43	0.990	0.08		
B	20	−0.0461	47.59	0.540	4.17	**0.973**	77.33	20	−0.0010	3.86	0.552	0.09	**0.995**	86.38
	30	−0.0983	40.92	0.663	5.81			30	−0.0029	3.71	0.744	0.14		
	40	−0.3522	45.99	0.916	3.19			40	−0.0100	3.84	0.938	0.08		
C	20	−0.0500	55.83	0.695	3.24	**0.774**	56.91	20	−0.0010	4.02	0.710	0.06	**0.779**	58.28
	30	−0.0541	55.34	0.867	2.11			30	−0.0011	4.01	0.875	0.04		
	40	−0.2255	58.40	0.891	2.36			40	−0.0045	4.07	0.880	0.05		
D	20	−0.0680	49.50	0.605	5.37	**0.960**	69.46	20	−0.0015	3.90	0.631	0.11	**0.980**	73.58
	30	−0.1265	48.39	0.754	5.99			30	−0.0032	3.88	0.823	0.12		
	40	−0.4225	52.37	0.937	3.27			40	−0.0104	3.97	0.976	0.05		
Texture (Firmness)														
A	20	−0.0777	30.50	0.994	0.61	**0.734**	44.51	20	−0.0037	3.45	0.980	0.05	**0.711**	46.18
	30	−0.0778	28.21	0.888	2.29			30	−0.0036	3.35	0.914	0.09		
	40	−0.2529	25.66	0.711	4.82			40	−0.0127	3.23	0.797	0.19		
B	20	−0.1208	27.24	0.853	4.91	**0.803**	46.00	20	−0.0090	3.38	0.988	0.10	**0.762**	42.79
	30	−0.1349	25.36	0.709	7.16			30	−0.0094	3.21	0.847	0.33		
	40	−0.4078	25.45	0.708	7.83			40	−0.0279	3.22	0.890	0.29		
C	20	−0.0631	33.66	0.887	2.20	**0.693**	44.10	20	−0.0025	3.53	0.899	0.08	**0.697**	45.73
	30	−0.0594	30.80	0.916	1.49			30	−0.0023	3.43	0.932	0.05		
	40	−0.2035	29.42	0.824	2.81			40	−0.0083	3.38	0.848	0.10		
D	20	−0.1243	29.27	0.919	3.61	**0.799**	43.65	20	−0.0088	3.47	0.982	0.12	**0.796**	41.62
	30	−0.1372	26.66	0.846	4.85			30	−0.0097	3.32	0.956	0.17		
	40	−0.3944	28.08	0.973	1.97			40	−0.0265	3.43	0.990	0.08		

T = Temperature (°C); k = rate constant (day^{-1}); Intercept = linear regression intercept; R^2 = coefficient of determination; SE = standard error; Ea = activation energy from Arrhenius plot (kJ/mol).

confirmed a good correlation. An Ea >70 kJ/mol further emphasized strong thermal dependence. In sample B, first-order kinetics again appeared more appropriate, with $R^2 > 0.900$ at 40 °C. In sample C, degradation kinetics were less clearly defined, possibly due to a synergistic effect of glass packaging and fermented brine that slowed degradation and blurred the distinction between kinetic orders. Sample D showed high R^2 values at 40 °C for both orders (0.944 for zero-order, 0.969 for first-order), suggesting a slight preference for the first-order model. In summary, first-order kinetics best describe the degradation behaviour of C* in olives stored in plastic bags (samples B and D), with k values increasing systematically with temperature. These findings support the conclusion that C* degradation is a thermally activated process, and storage in plastic significantly accelerates quality loss compared to glass jars.

Among the chromatic variations, browning index was also observed to evaluate the degradative kinetics (Table 4). For sample A (glass jar with reconstructed brine), high coefficients of determination ($R^2 > 0.950$) were recorded for both kinetic models, with a better fit for the first-order reaction at all temperatures. However, the Arrhenius model returned similar R^2 values for both orders (close to 0.800), and the calculated activation energy was relatively low (about 40 kJ/mol), possibly due to the catalytic effect of enzymatic activity (Yildiz, 2009). An irregular behaviour in the sample B kept at 20 °C with a decidedly low R^2 was observed, although the Arrhenius model gives a very high coefficient of determination for both orders (>0.950). In sample C, no clear kinetic order was evident, but the trend appeared to lean toward first-order kinetics. For sample D, the best fit was found at 40 °C, with high R^2 values and an overall first-order kinetic trend (Arrhenius $R^2 = 0.980$). Overall, colour degradation across samples showed consistent behaviour, with a general tendency toward first-order kinetics. For the texture, excellent results in terms of linearity were found for all samples (Table 4). Sample A shows high R^2 especially at 20 °C (>0.980) for both reaction orders. However, first-order kinetics consistently yielded more stable and higher R^2 values across all temperatures, supporting the assumption that degradation follows this model. An excellent fit was observed for sample C which showed high values of the coefficient of determination (especially for the first order reaction), although the values of R^2 of the Arrhenius model are close to 0.700. Sample D showed consistent texture and very high R^2 values between the different temperatures for both models, especially for the first reaction order (between 0.956 and 0.990). The reported data suggest that the consistency of olives in the different samples analysed follows a degradation following a first order kinetics (generally higher R^2). Higher values of Ea have been found for this order in samples A and C, indicating better glass protection for this parameter, requiring a higher Ea for the degradation reaction to take place.

To approach product quality from a multidimensional perspective, we considered the ratio of Texture/b* (Table 5). This novel approach combines two key quality parameters: visual appearance (colour) and mechanical consistency (firmness), to provide a more comprehensive assessment of overall olive quality. The index serves both as a global qualitative descriptor and a diagnostic tool for detecting potential anomalies, particularly when disproportionate degradation occurs between the two parameters. This approach highlights the different response to degradation of the different combinations (brine-packing). The k values increase consistently with increasing temperature and return in almost all cases an exceptionally high R^2 according to the Arrhenius model. This indicates that temperature is a determinant in the degradation reactions and there is a strong correlation, although individual kinetics do not show exceptionally high fit. High R^2 values were observed according to the Arrhenius model for all samples with values especially for the zero order between 0.828 and 0.998 showing an exponential trend of k variation with temperature.

4. Conclusion

This study assessed the combined effects of packaging type and brine composition on the quality of naturally fermented table olives during storage. Colour changes were observed over time, particularly in samples stored in plastic bags, likely due to higher oxygen permeability and chemical interactions with the brine. The results obtained highlighted the crucial role of packaging in maintaining the visual appearance of fermented olives during shelf life. pH values consistently remained below 4.2, ensuring microbiological safety. Colour stability proved strongly dependent on both packaging and brine type, with glass jars and filtered fermentation brine better maintaining colour, preserving bioactive compounds, and improving overall product stability. Similar trends were observed for total phenol content, antioxidant activity, and firmness, confirming the protective effects of combining appropriate packaging and brine. This preservation of health-promoting and sensory attributes aligns with consumer preferences for antioxidant-rich foods. Kinetic modelling showed that most quality parameters, especially colour and texture, followed first-order degradation kinetics, with temperature playing a central role. The proposed texture/b* ratio emerged as a new quality index, reflecting the multidimensional nature of olive quality. Overall, the use of glass jars with filtered fermentation brine proved most effective in maintaining the functional and sensory characteristics of the product. Additionally, reusing filtered brine enhanced quality and offered a sustainable solution by reducing waste and environmental impact. These findings provide practical guidance for optimizing packaging strategies, promoting circular economy practices, and reducing food loss in the table olive industry.

Table 5
Kinetic modelling of Texture/b* degradation in olive samples: zero-order, first-order, and Arrhenius model.

Texture/b*														
	Zero order					Arrhenius Plot		First order					Arrhenius Plot	
	T	k	Intercept	R^2	SE	R^2	Ea	T	k	Intercept	R^2	SE	R^2	Ea
A	20	−0.0037	1.81	0.959	0.11	**0.780**	32.28	20	−0.0028	0.61	0.938	0.06	**0.698**	29.38
	30	−0.0039	1.81	0.794	0.17			30	−0.0027	0.60	0.815	0.11		
	40	−0.0087	1.59	0.642	0.19			40	−0.0060	0.45	0.673	0.13		
B	20	−0.0073	1.82	0.874	0.27	**0.909**	39.95	20	−0.0074	0.66	0.984	0.09	**0.999**	26.87
	30	−0.0165	2.34	0.741	0.55			30	−0.0106	0.86	0.819	0.32		
	40	−0.0205	1.82	0.716	0.39			40	−0.0151	0.58	0.807	0.22		
C	20	−0.0025	1.86	0.598	0.20	**0.855**	27.55	20	−0.0017	0.63	0.654	0.12	**0.828**	27.46
	30	−0.0028	1.80	0.404	0.29			30	−0.0019	0.59	0.486	0.16		
	40	−0.0052	1.53	0.348	0.21			40	−0.0035	0.42	0.314	0.16		
D	20	−0.0068	1.91	0.826	0.30	**0.960**	30.72	20	−0.0066	0.71	0.901	0.21	**0.750**	25.52
	30	−0.0089	1.87	0.771	0.24			30	−0.0067	0.65	0.796	0.17		
	40	−0.0152	1.75	0.905	0.15			40	−0.0130	0.60	0.874	0.15		

T = Temperature (°C); k = rate constant (day^{-1}); Intercept = linear regression intercept; R^2 = coefficient of determination; SE = standard error; Ea = activation energy from Arrhenius plot (kJ/mol).

CRediT authorship contribution statement

Iolanda Cilea: Methodology, Investigation, Formal analysis. **Antonio Gattuso:** Writing – original draft, Software, Data curation, Conceptualization. **Marco Poiana:** Writing – review & editing, Visualization, Validation, Supervision, Resources, Project administration, Funding acquisition, Conceptualization. **Alessandra De Bruno:** Writing – review & editing, Visualization, Validation, Supervision, Data curation, Conceptualization.

Data availability

All data and materials are available from the authors upon request.

Five key references:

1. Lombardi et al. (2018) investigated the effect of storage conditions on natural green table olives. The research highlights how temperature and packaging affect quality over time.
2. Piscopo et al. (2016) studied the termal treatment and the influence of packaging on olives and brines to preserve quality characteristics.
3. Romeo et al. (2009) examined the stability of fermented green olives after fermentation.
4. Sánchez Gómez et al. (2006) investigated how preservation methods affect Spanish-style green olives over time.
5. Timpanaro et al. (2023) examined Spanish-style and natural fermentation green olives highlighting the qualitative differences for the treatments for different cultivars.

Key references point to the need for a study that goes beyond monitoring changes in quality over time. Each of them supported the authors' decision to evaluate chemical-physical but especially kinetic responses at different temperatures, packaging and brine type in order to determine models for shelf-life prediction to fill this gap in the bibliography.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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