



Influence of modified atmosphere packaging on post-harvest physiology, overall quality, and bioactive compounds during cold storage and shelf-life of 'Tondo Nero' figs (*Ficus carica* L.)

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ABSTRACT

The impact of four polymers [(a) oriented polypropylene film (OPP); b) polyolefin heat-shrinkable film (BOLPH); c) micro-perforated OPP film (MICRO); and d) macro-perforated OPP film (MACRO)] was tested on 'Tondo Nero' figs, cold stored at 1 °C for 7 or 14 d (CS) plus 3 d at 20 °C in simulate marketing conditions (SMC). In-package ethylene increased as film permeability decreased, peaking in OPP-packages. CO₂ partial pressure of OPP-packages, peaking at 15 kPa in CS conditions and at about 30 kPa at the end of SMC, and O₂ partial pressure tending to 0 kPa, negatively affected the overall acceptability. In-package CO₂, never exceeding 8 kPa, and O₂, never dropping 12 kPa, of MICRO and MACRO films did not affect fructose and glucose concentration, but reduced the loss of phenolic compounds, ascorbic acid and antioxidant activity of the peel as well as of the pulp, showing a better performance compared to the other films.

1. Introduction

Fig (*Ficus carica* L.) represents an important crop worldwide, particularly in the Mediterranean region. Many cultivars produce two crops a year; the first one, known as "breba crop", normally matures in early summer; the second one, known as "fig or main crop" matures in late summer (Stover et al., 2007). The first crop represents an important source of income for the high prices it gets on the fresh market compared to the main crop. Fig fruit, consisting of a complex inflorescence called syconium, are eaten peeled or unpeeled as a fresh or dried fruit or are used as an ingredient for the preparation of various desserts, jams, and ice creams.

Figs play an important role in nutrition being an important source of fibers, sugars, vitamins, polyphenols such as flavonoids, anthocyanins, and other nutraceuticals (Lim, 2012; Barolo et al., 2014). As reported in ancient texts, figs have largely used in traditional medicine to improve the health of the elderly and as a prevention or remedy against various diseases (Chessa, 1997; Shamkant et al., 2014). Generally, fruits with dark peel contain higher levels of these compounds resulting in higher antioxidant activity compared to fruits with green peel (Solomon et al., 2006).

Chemical composition and consumers' acceptance depend on the

degree of ripeness reached at harvest time, as sugars as well as organic acids do not increase after harvest (Rodov et al., 2002; Crisosto & Kader, 2004; Crisosto et al., 2010; Byeon & Lee, 2021).

The classification of fig fruits is controversial, although they are generally considered as climacteric (Ferguson et al., 1990). This because the ripening period in fruit is very short and due to the botanical complexity of the syconium, the ripening process of the individual part of the fruit can be asynchronous. As a result, it is very difficult to detect the climacteric peak of respiration and ethylene (D'Aquino et al., 2015; Freiman et al., 2015).

Unfortunately, when full ripe, fresh figs have a short post-harvest life due to their high perishability. At that stage the whole fruit and the peel are very sensitive to impacts and pressures, which can cause cuticle removal, bruising and injuries that favor pathogens' infections and commercial depreciation (Kong et al., 2013; Villalobos et al., 2017; Ertan et al., 2019).

Microbiological spoilage is further favored by the presence of cracks in the skin and the entrance of insects and microorganisms from the ostiole which, as ripening progresses, increases its diameter and, in some varieties, tends to open and lose juice (Ferguson et al., 1990; Michailides et al., 1996).

The shelf-life of figs depends on pre- and post-harvest handling

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practices, which should be done carefully, in order to minimize physical damages and delay senescence and microbiological infections.

Figs are not chilling sensitive and can be stored at 0–2 °C without any disorders induced by exposure to low temperature (Crisosto & Kader, 2007). Thus, cold storage either alone or in combination with modified atmosphere packaging (MAP) is the most important technology capable of preserving quality and controlling microbiological spoilage (Kader, 1986; Colelli et al., 1991; Church, 1994; Opara et al., 2019).

Modified atmosphere results from the interaction between the respiratory activity of the fruit and gas transmission rate of the package, which can be mediated by the introduction of a gas mixture (Oliveira et al., 1998).

In package oxygen must not be below 2 kPa while carbon dioxide should not be above 15–20 kPa to avoid anaerobic respiration (Colelli et al., 1991; Colelli & Kader, 1994; Crisosto & Kader, 2004; Colelli & Amodio, 2020).

The optimal storage conditions reported in literature for fresh figs, is modified atmospheres with a combination of 5–10 kPa oxygen and 15–20 kPa carbon dioxide and temperatures in the range of –1.5 °C with a relative humidity of 90–95% (Colelli & Amodio, 2020).

However, optimal storage conditions depend on various factors such as the variety and the degree of ripeness at harvest. If the storage temperature and/or modified atmosphere are not adequate there may be alterations in the flavor and in chemical constituents due to the accumulation of ethanol (Colelli & Kader, 1994).

Despite the massive bulk of scientific work dealing with nutritional and nutraceutical composition of fresh or dried figs, little information is available on quality and compositional changes occurring after harvest in packaged-cold-stored figs (Villalobos et al., 2014; Villalobos et al., 2015a; Villalobos et al., 2016; Villalobos, 2018).

Therefore, the aim of this study was to provide and implement the knowledge on the impact that different modified atmospheres may have on postharvest physiology, sensory quality, nutritional and nutraceutical compositions of fig fruit. As the optimal range of air composition in terms of CO₂ and O₂ partial pressure is not well defined, depending also on cultivar and environment conditions, in this experiment we used four different films with marked differences in their barrier properties in order to achieve different gases composition inside the packages.

2. Materials and methods

2.1. Plant material and experimental design

'Tondo Nero' figs (*F. carica* L.), (an appreciated cultivar of the Sardinian germplasm, with a purple skin crossed by green streaks, whose creamy-white flesh lodges the flowers immersed in a pinkish juice) were used for this study. The fruit, from the first crop, were harvested at the commercial ripening stage on June 30th from Agris Sardegna research station located in Ussana, South Sardinia (Lat. 39°23' N, Long. 9°04' E). Within two hours from harvest the fruit were transported to the ISPA-CNR laboratory located in Sassari (north Sardinia) in refrigerated

conditions.

Sound and uniform fruit on average weighing about 100 g, were placed in polypropylene trays (18 × 12 × 4.5 cm) in number of 4 and sealed within bags (30 × 25 cm) made with four different polymers (Table 1). Polypropylene trays of control fruit were not packaged. All trays were divided into two groups: group A was stored for 7 d at 1 °C and then transferred to 20 °C for 3 d, to simulate marketing conditions (SMC); group B was stored at 1 °C for 14 d and then transferred to 20 °C for 3 d to SMC.

In previous experiments conducted in our laboratory with first crop figs, we noted that, despite the high susceptibility to microbiological spoilage, generally molds or other visual alterations caused by pathogens start to develop after 3–4 days at room temperature. For this reason, we limited the SMC to 3 d.

To monitor the in-package and storage room temperature and humidity, a data logger (RHT10 Humidity/Temperature USB Datalogger, Extech instruments, FLIR System, Townsend West, USA) was placed inside three packages of each treatment or in different locations of the storage room and set to record temperature and relative humidity every six hours.

A total of 180 trays (nine trays for each sampling time and treatment) were prepared to determine in-package air composition, chemical analysis, and evaluate overall acceptability.

2.2. Respiration, in-package gas composition and ethylene production rate

Respiratory activity was determined on 10 un-packaged fruit treated with a 600 mg L⁻¹ fludioxonil emulsion (Scholar, Syngenta Crop Protection, Milan, Italy) to prevent decay. It was determined at harvest time at 20 °C, after 1, 7 or 14 d of cold storage at 1 °C, and after 1 or 3 d of SMC at 20 °C following 7 or 14 d at 1 °C. Measurements were carried according to a closed system: individual fruit were placed in 1 L jars, whose lids were fitted with two silicon septa and closed for 4 h for fruit stored at 1 °C and for 2 h for those held at 20 °C. CO₂ was determined by a combined CO₂/O₂ analyzer connected with each jar by two tubes, each one ending with a needle inserted in one of the two septa, to form a closed system. (Combi Check 9800–1, PBI-Dansensor A/S, Rinsted, Denmark) (D'Aquino et al. 2016). Respiratory activity was expressed as μg CO₂ kg⁻¹ s⁻¹.

In-package gas composition was determined after 1, 7 and 14 d at 1 °C and after 1 and 3 d at 20 °C following 7 or 14 d at 1 °C (six trays for each sampling time and treatment) using a hand-held analyzer (Check Point, PBI-Dansensor, Italia, Milan, Italy) for combined measurements of oxygen and carbon dioxide. Ethylene concentration was determined according to the procedure described by Palma et al. (2015).

2.3. Weight loss and sensory evaluation

Thirty-six fruit, initially individually weighed, were re-weighed at the end of each storage time and SMC. Weight loss was expressed as the

Table 1
Barrier properties of the used plastic films.

Film	Film characteristics	Commercial name	Thickness	O ₂ permeance cc/m ² /24 h atm	CO ₂ permeance cc/m ² /24 h atm	WVTR g/m ² /24 h.
OPP	Oriented polypropylene film	Coralene SWAF 400	25 μm	2150 ^a	8600 ^b	7
BOLPH	Polyolefin heat shrinkable film	Bolphane BY	25 μm	6500 ^a	26000	20
MICRO ^c	Laser micro-perforated oriented polypropylene film	Coralife SWAF 400	25 μm	63845	49112	7.2
MACRO ^d	Laser macro-perforated oriented polypropylene film	Coralife SWAF 400	25 μm	–	–	–

⁵WVTR: Water Vapor Transmission Rate.

^a Data, provided by the manufactures

^b Not provided by manufacturer; calculated considering a CO₂/O₂ ratio equal to 4.

^c Calculated by adding to Coralene SWAF permeance to CO₂ and O₂ and transmission rate to water vapor the effect of 400 laser perforations per meter square. Diameter of each laser perforation equal to 80 μm. O₂ permeability through perforations was considered 1.30 times that of CO₂.

^d Holes averaging 6 mm in diameter. A total of 2 holes per package.

percentage reduction of the initial weight. The same fruit were used to assess overall appearance first and carry out chemical analyses then.

Six trained laboratory technicians judged the fruit for overall acceptability, which included texture, and taste. At the end of each sampling time two fruit from each tray, for a total of 12 fruit per treatment, were used. Each fruit was divided longitudinally into 4 pieces and presented to panelists in anonymous form. The judgement was based on a subjective scale ranging from 1 to 9, where 1 = very poor, 3 = poor, 5 = good (limit of marketability), 7 = very good, and 9 = excellent.

2.4. Chemical analysis

2.4.1. Sample preparation

Acetonitrile, trifluoroacetic acid and methanol were of high-performance liquid chromatography grade (Merck, Darmstadt, Germany); other reagents, of analytical grade, were: sodium carbonate (Merck, Darmstadt, Germany); 2,2-diphenyl-1-picryldazyl (DPPH), Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid), flavonoids, organic acids standards and Folin–Ciocalteu phenol reagents from Fluka (Buchs, Switzerland); cyanidin 3-rutinoside, apigenin glucoside and quercetin 3-glucoside from Extrasynthese (Genay, France); ascorbic acid, glucose, fructose and sucrose from Sigma-Aldrich Co. (Milan, Italy).

The fruit were manually peeled, and both the peel and the pulp were immediately transferred to $-80\text{ }^{\circ}\text{C}$. Before analyses, the peel and the pulp were homogenized separately using an immersion blender (model RCSM-350–400 P, Royal Catering, Italy).

Total soluble solids (TSS) were measured directly in the homogenized, while the other chemical parameters were determined on extracts obtained in accordance with the following procedure: 10 g aliquots of homogenate were transferred into a glass tube and placed in contact with an extracting solution methanol/water (80/20). After 2 h in agitation, in dark and at room temperature the suspension was centrifuged for 15 min at 13,000 g (Centurion Scientific Ltd, West Sussex, England) and the supernatant, filtered through a 0.45 mm acetate cellulose filter, was employed for the analyses, which were performed in triplicate at harvest, after 7 or 14 d of cold storage and at the end of each respective 3-d period of SMC at $20\text{ }^{\circ}\text{C}$.

2.4.2. TSS, carbohydrates, organic acid, flavonoids, anthocyanin, ascorbic acid, total phenol content, and antioxidant activity

TSS were measured by a digital refractometer (Mod. PR-101, Atago, Tokyo, Japan) and expressed as %. For flavonoids and anthocyanin quantification, the HPLC system (LaChrom Merck-Hitachi liquid chromatograph, Hitachi Ltd., Tokyo, Japan) consisting of a D-7000 system manager, a L-7100 pump and a L-7200 autosampler, was coupled with L-7455 photodiode detector (DAD) and a C18 Prevail column (250 mm \times 0.4.6 mm, 5 μ , Alltech, Milan, Italy), with a Alltech C18 precolumn (7.5 mm \times 4.6 mm I.D.). HPLC elution was carried out at $30\text{ }^{\circ}\text{C}$. The solvent gradient was performed by varying the proportion of solvent A (H_2O with 0.1% trifluoroacetic acid) and solvent B (CH_3CN with 0.1% trifluoroacetic acid) as follows: initial condition 2% B; at 15 min, 5% B; at 30 min, 25% B; at 45 min, 5% B; at 50 min, 2% B; flow rate of 1 mL/min. The chromatogram was monitored simultaneously at 280, 360 nm for flavonoids and 510 nm for anthocyanins. Calculation of concentrations for chlorogenic acid, apigenin 7-glucoside, quercetin 7-rutinoside catechin and cyanidin 3-rutinoside was based on external standards while the concentration of the other compounds was expressed as catechin equivalents. Flavonoids and anthocyanins were identified by LC-electrospray ionization (ESI) MS analysis using an Agilent Technologies (Palo Alto, CA, USA) 1100 series LC/MSD equipped with a diode-array detector (DAD). A ChemStation HP A.10.02 was used for data analysis.

Carbohydrates analyses were performed according to the procedure described by Palma et al. (2018). Stock standard solutions of each carbohydrate were prepared in ultrapure water and their quantifications, in

peel and pulp, were calculated according to the linear calibration curves of standard compounds.

Simultaneous separation and determination of organic acids and ascorbic acid were done by a chromatographic method according to the procedure described by Palma et al. (2013). Peaks of organic acids and ascorbic acid were identified and quantified by comparing their retention times with those of external standards.

Total phenolic content was determined according to the Folin–Ciocalteu colorimetric method (Singleton & Rossi, 1965). The absorbance was achieved at 760 nm by a UV–vis spectrophotometer (Varian Cary 50, Netherlands). Total phenolic content (TPC) was expressed as mg kg^{-1} of gallic acid equivalents. Antioxidant activity was assessed using the free radical DPPH (2,2-diphenyl-1-picrylhydrazyl). The mixture containing 3 mL of a methanol solution of 0.16 mM DPPH and 100 mL of sample, was allowed to react for 15 min in a cuvette. The absorbance of the DPPH solution was determined at 515 nm by a UV–vis spectrophotometer. Antioxidant activity was expressed as mmole kg^{-1} of Trolox equivalent antioxidant capacity (TEAC).

2.5. Statistical analysis

Statistical analysis was performed using Statgraphics Centurion software (Herndon, VA, USA), version XV Professional statistical program. Analysis of variance (ANOVA) was carried out for each storage time and mean comparisons among treatments were performed using Duncan's multiple range test at $P \leq 0.05$.

To evaluate the correlation between antioxidant activity, total phenolic content, ascorbic acid content and treatments, the Pearson's coefficients were used.

3. Results and discussion

3.1. Respiratory activity

Figs are climacteric fruit with a moderate respiratory activity (Crisosto & Kader, 2004; Crisosto et al., 2011). At harvest ($20\text{ }^{\circ}\text{C}$), respiratory activity was $17.1\text{ }\mu\text{g CO}_2\text{ kg}^{-1}\text{ s}^{-1}$ and dropped to $2.47\text{ }\mu\text{g CO}_2\text{ kg}^{-1}\text{ s}^{-1}$ after 1 d at $1\text{ }^{\circ}\text{C}$ (Fig. 1). Significant changes did not occur during the 7 or 14 d at $1\text{ }^{\circ}\text{C}$, but following transfer to SMC, respiratory activity increased reaching values like those found at the harvest time.

This burst in respiratory activity normally occurs when fruit from low temperatures are moved to warm temperatures. However, generally a decreasing trend follows, unless physiological stresses or microbiological infections would occur during cold storage (Lyons & Breidenbach, 1990; D'Aquino et al., 2010).

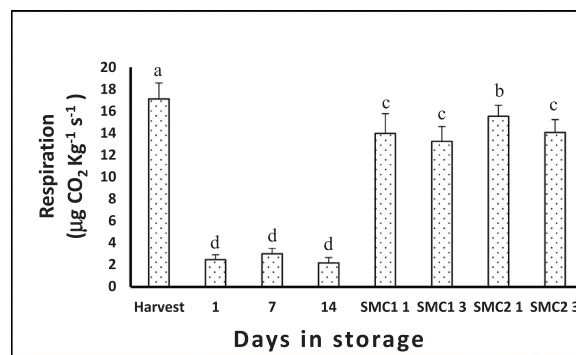


Fig. 1. Respiratory activity as carbon dioxide release in 'Tondo Nero' figs at harvest and after 1, 7 or 14 d of storage at $1\text{ }^{\circ}\text{C}$ and in SMC at $20\text{ }^{\circ}\text{C}$ following 7 or 14 d storage at $1\text{ }^{\circ}\text{C}$ (SMC1 1 = 1 d; SMC1 3 = 3d; SMC2 1 = 1 d and SMC2 3 = 3d). Columns with different letters are significantly different at $P \leq 0.05$ according to ANOVA analysis. Vertical bars represent the standard deviation ($n = 10$).

Waghmare et al. (2014) reported that temperature and time significantly affected respiration of fresh figs. Particularly, they found a linear relationship between temperature and respiration, which approximately decreased by 3–4 times when temperature was lowered from 30° to 10° C.

After a decline occurring 24 h following harvest, respiratory activity in ‘Craxiu de Porcu’ figs stored at 20 °C increased first gradually and then sharply during the six days of simulated marketing conditions, but the authors attributed this increase to latent infections of microorganism rather than a climacteric pattern (D’Aquino et al., 1998).

Regardless the cause, an increase in respiratory activity when fruit are transferred from refrigerated conditions to room temperature, represents an aspect of extreme importance for packaged fruit that which needs be considered when designing a package, to prevent potential anaerobic conditions.

3.2. In-package gas composition

In accordance with the polymers’ barrier properties (Table 1), the highest CO₂ and lowest O₂ concentrations were detected in OPP packages, followed by BOLPH, MICRO and MACRO ones (Figs. 1A, 1B, 1C). Apparently, neither CO₂ nor O₂ partial pressure reached a steady state condition in OPP packages; both gases were quite stable during the first 7 d at 1 °C (CO₂ ~ 10 kPa; O₂ ~ 11 kPa). The marked changes detected at day 14 (CO₂ = 14.9 kPa; O₂ = 6.9 kPa) (Fig. 2A; 2B), could result by water condensation onto the inner side of the packages. Chen et al. (2014), reported that oxygen transmission rate of BOPP film decreases when RH increases and a further restriction to gas exchange both O₂ can be caused by the layer of water covering the film.

In MACRO, MICRO and BOLPH packages air composition underwent slight changes over cold storage period, with final values partial pressures for CO₂ of 0.16, 1.26, 9.06 kPa and for O₂ of 20.15, 19.38 and 10 kPa, respectively (Fig. 2B).

An overall increase in CO₂ alongside with a decrease in O₂ occurred in all packages when fruit were moved to SMC, but changes of individual films followed different patterns. In OPP packages CO₂ increased to about 21 kPa at the end of SMC1 and exceeded 30 kPa at the end of SMC2, concomitantly O₂ partial pressure dropped to about 1 kPa at the end of SMC1 and was almost undetectable at the end of SMC2. The results indicate that at 20 °C the increased demand of O₂ to sustain the aerobic metabolism could not be matched by the O₂ transmission rate of OPP packages and likely a shift to anaerobic respiration took place. In contrast, the lower barrier to gases of BOPP allowed to generate an in-package air composition with a CO₂ partial pressure always below 17 kPa and an O₂ partial pressure ranging from 5 to 8 kPa, a composition that falls within a range generally considered optimal (CO₂ = 15–20 kPa CO₂; O₂ = 5–10 kPa) or at least not at risk of anaerobiosis (Crisosto & Kader, 2004; Colelli & Amodio, 2020), although Turk et al. (1994) for controlled atmosphere storage of ‘Bursa Siyahi’ figs recommended 3–5 kPa CO₂ and 3–5 kPa O₂.

In MICRO packages the increase in temperature had a moderate impact on gases composition with an average CO₂ and O₂ partial pressure of 8 kPa and 13.5 kPa, respectively. Similar results were reported by Villalobos et al. (2015b) in ‘San Antonio’ figs packed with micro-perforated films (3 holes per packages Ø = 100 µm) and stored for 17 d at 0 °C. In ‘Cuelo Dama Blanco’ figs stored in the same conditions, a steady state condition was reached by day fourteen. When the number of holes per package was increased to 16, in ‘Cuelo Dama Blanco’ figs the steady state condition was reached after 7 d. These authors concluded that the gaseous balance within the packages was not only due to the packages permeability, determined in this case by the number of micro-perforations, but also to respiration rate of the fig cultivars. Appreciable changes in gas composition did not occur in MACRO perforated packages during the SMC periods. (Figs. 1A, 1B).

Overall, in-package ethylene evolution increased with storage, particularly when fruit were moved to SMC and reflected the barrier

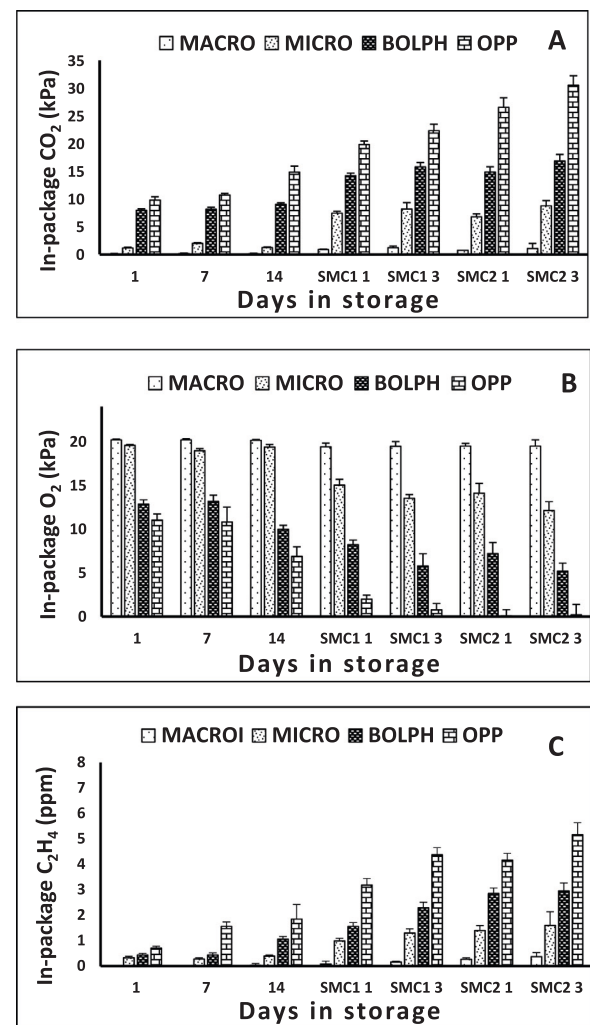


Fig. 2. In-package CO₂ (A), O₂ (B) and C₂H₄ (C) concentrations of ‘Tondo nero’ fruit packaged with macro-perforated (MACRO), micro-perforated (MICRO), Bolphane (BOLPH) and OPP films during cold storage at 1 °C after 1, 7 or 14 d and in SMC at 20 °C following 7 or 14 d storage at 1 °C (SMC1 1 = 1 d; SMC1 3 = 3d; SMC2 1 = 1 d and SMC2 3 = 3d). Vertical bars represent the standard deviation (n = 6).

properties of the tested films as its concentration increased as film permeability decreased (Fig. 2C). Colelli & Amodio (2020) report that a combination of 5–10% O₂ and 15–20% CO₂ are effective in reduction of ethylene production rates in fig fruit. High-CO₂ exposure reduced the ethylene production and decay incidence of fruit if CO₂ levels do not exceed the limits tolerated by the fruit. Bahar & Lichter (2018), found in Ottomanit figs stored in controlled atmosphere with 15 kPa of CO₂ a disintegration of the peel and internal browning which was interpreted as CO₂ injury.

Despite fig fruits are generally categorized as climacteric, showing a rise in respiration and with a moderate ethylene production rate (1–10 µl/kg/hr at 20 °C) and sensitive to ethylene (Ferguson et al., 1990; Crisosto and Kader, 2004), they differ greatly from all other climacteric species in that they share features common to both climacteric and non-climacteric groups. At the ripening onset both ethylene and respiration rates increase, but differently than other species of the same group, as apples and tomatoes, the ripening process does not occur if fruit are picked before the onset of the ripening process; yet, fruit reach their final size during the climacteric, which last only 3 d (Freiman et al., 2015). Generally, one strategy to prolong the postharvest life of climacteric fruit is to harvest the fruit in their pre-climacteric stage, store

them at low temperature to prevent the onset of the climacteric and induce the ripening process upon transfer to warm temperatures. Unlikely, in figs this strategy does not work, because fruit would never develop their optimal quality features in terms of flavour, size, texture, aroma and color if were harvested in their pre-climacteric phase (Flaishman et al., 2008). On the other hand, even a small delay beyond the optimal maturity would dramatically reduce the postharvest life and make the fruit highly susceptible to microbiological decay (Crisosto et al., 2011; Flaishman et al., 2008).

As fruit were harvested at the optimal maturity stage, likely after the climacteric peak, the moderate increase in respiration rate alongside with the more marked rise of in-package CO₂ and ethylene, cannot be attributed to the ripening process; rather it might result from the effect of incipient infections. In fact, the particular botanical structure of the syconium makes the tissue susceptible to infections starting both from the peel, through wounds and micro-cracks, and the syconium cavity visited by wasps or other insects (D'Aquino et al., 2015; Crisosto et al., 2011), even if by limiting the SMC to only 3 d we did not find, as expected, no mould or sign of microbiological deterioration.

3.3. Effect of packaging on sensory quality and weight loss

The influence of the different packaging systems on overall acceptability is shown in Fig. 3. Sensory quality of fresh fruit is the result of a combination of taste (sweet, sour, bitter, presence or absence of off-flavor), aroma, off-odors, and textural properties (Nunes & Emond, 2007). In this study, overall acceptability declined in all types of packaging.

After 14 d of cold storage, unwrapped fruit were judged at the limit of acceptability while at day 14 packaged fruit were rated higher than 7, except for those packaged with OPP film, which were rated 6.5. The highest score was given to fruit packaged with the MACRO and MICRO perforated films, followed by BOLPH and OPP films.

At the end of SMC following 14 d of cold storage, unwrapped fruit and those packaged with OPP film were judged below the limit of acceptability, while the highest values were attributed to fruit packaged with MACRO and MICRO perforated films; BOLPH group reached values close to the limit of acceptability.

The worst performance achieved in fruit packaged with the OPP film was mainly due to the development of off flavors induced by high concentration of CO₂ and the decline of O₂ below the critical limit that triggers anaerobic metabolism and for peel alterations (Villalobos et al., 2018). Studies conducted by Bahar & Lichter, (2018), with 'Ottomanit' fig fruit stored in controlled atmosphere with 15 kPa of CO₂, reported an

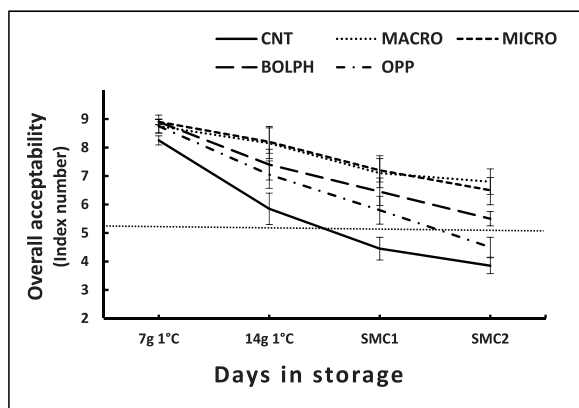


Fig. 3. Overall acceptability of 'Tondo nero' fruit packaged with macro-perforated (MACRO), micro-perforated (MICRO), Bolphane (BOLPH) and OPP film after 7 or 14 d at 1 °C or at the end of SMC at 20 °C following 7 d or 14 d storage at 1 °C (SMC1 = 3 d; SMC2 = 3d). Point 5 represents the limit of marketability; vertical bars represent the standard deviation (n = 36).

alteration of the peel, interpreted as CO₂ injury, and Flaishman et al., (2008) reported a development off flavor in 'Nazareth' breba figs stored in MAP with 10–12 kPa CO₂, which did not occur in those exposed to 4–6 kPa CO₂.

In our study, the lowest score given to unwrapped fruit was mainly due to the worsening of freshness due to the high weight loss. Most fruit and vegetables lose their freshness when the water loss approaches 3–10% of the initial weight (Ben-Yehoshua & Rodov, 2003; Nunes & Emond, 2007; D'Aquino et al., 2016; Afsah-Hejri et al., 2021).

In the present study, weight loss increased with storage in all cultivars, particularly in unwrapped fruit, being around 4.5% and 7.5% after just 7 and 14 d of storage and ending at 16% and 20% in SMC after 7 or 14 d of cold storage, respectively. In contrast, in wrapped fruit, weight loss was lower than 2% (Fig. 4). The positive effect of wrapping in reducing weight loss was also observed in macro-perforated film, which by ensuring adequate humidity inside the packages, allowed to preserve freshness. (Table S1 in Supplementary Material).

However, overall results of this study, including all the chemical parameters which will be shown and discussed below, clearly show that until fruit are cold stored high levels of in-package CO₂ associated with low concentrations of O₂ substantially do not affect negatively the overall acceptability and chemical composition, but when fruit are moved to warm temperatures the less the in-package gas composition differs from normal air and the better the overall quality is maintained. In other words, it seems that quality maintenance depends more on a high level of humidity; conditions created by MICRO and MACRO films. Packages that lead to high levels of CO₂ and reduced concentrations of O₂, as those made with OPP and BOLPH films, can stimulate anaerobic respiration and accelerate the degradation of respirable substrates.

3.4. Effect of MAP on chemical parameters evolution during storage and shelf life

3.4.1. TSS, Carbohydrates and organic acid

The initial mean value of TSS was 16.5% in pulp and 14.8% in the peel (Table 2).

In pulp the largest decrease in TSS, occurred in BOLPH and OPP films after 14 d of storage (14%) and the subsequent SMC (18%).

In the peel, TSS decreased by about 22% over the whole storage time in all packages (Table 2).

A similar trend was reported by Kaynak et al. (1998) in figs packaged in plastic boxes and stored at 0–1 °C: under those conditions they found that TSS did not change until the fourteenth day but decreased after day 20. Likewise, Tsantili et al. (2003) reported that in ambient conditions or in modified atmospheres with O₂ and CO₂ concentrations of 2% and 0.05%, respectively, TSS did not undergo significant changes. In

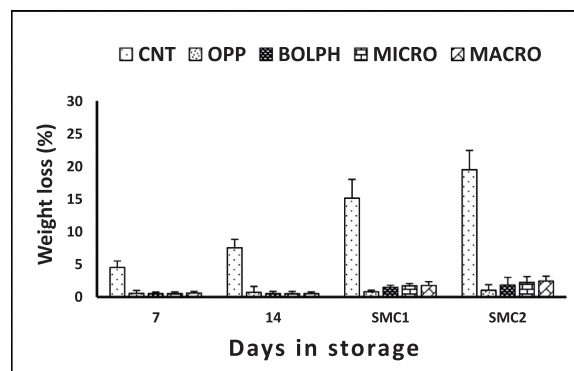


Fig. 4. Weight loss of 'Tondo nero' fruit packaged with macro-perforated (MACRO), micro-perforated (MICRO), Bolphane (BOLPH) and OPP films after 7 or 14 d at 1 °C or at the end of SMC at 20 °C following 7 d or 14 d storage at 1 °C (SMC1 = 3 d; SMC2 = 3d). Vertical bars represent the standard deviation (n = 36).

Table 2
Sugar content in 'Tondo Nero' figs stored at 1 °C for 7 d or 14 d plus 3 d in SMC at 20 °C.

	Fructose g kg ⁻¹		Glucose g kg ⁻¹		Sucrose g kg ⁻¹		TSS %	
	Pulp	Peel	Pulp	Peel	Pulp	Peel	Pulp	Peel
Harvest	68.95 ± 0.62	52.45 ± 3.83	60.32 ± 2.85	47.41 ± 2.04	2.33 ± 0.06	5.28 ± 1.08	165.1 ± 0.36	148.0 ± 0.91
7 d at 1 °C								
CNT	63.73 ± 1.01 a	51.87 ± 2.78 a	52.25 ± 2.61 a	45.26 ± 3.19 a	1.12 ± 0.08 b	3.06 ± 1.27 a	15.0 ± 0.69 a	13.6 ± 0.76 a
OPP	65.14 ± 2.08 a	49.69 ± 2.32 a	54.91 ± 1.04 a	42.63 ± 3.11 ab	1.23 ± 0.13 b	1.99 ± 0.47 ab	14.7 ± 0.26 a	13.1 ± 0.91 ab
BOLPH	64.64 ± 1.48 a	45.90 ± 0.70 b	52.71 ± 2.21 a	38.87 ± 1.01 b	1.10 ± 0.16 b	2.16 ± 0.45 ab	14.6 ± 0.47 a	12.4 ± 0.61 ab
MICRO	65.10 ± 0.97 a	44.16 ± 1.93 b	53.40 ± 1.36 a	40.32 ± 2.96 ab	2.29 ± 0.02 a	1.91 ± 0.25 ab	15.0 ± 0.25 a	12.1 ± 0.73 b
MACRO	64.73 ± 2.75 a	44.85 ± 1.51 b	53.68 ± 1.26 a	39.82 ± 2.18 b	2.19 ± 0.32 a	1.66 ± 0.24 b	15.0 ± 0.25 a	11.8 ± 0.58 b
7 d at 1 °C plus 3 d in SMC at 20 °C								
CNT	62.15 ± 0.68 a	49.18 ± 0.83 a	51.16 ± 1.37 a	42.06 ± 0.40 a	0.94 ± 0.05 b	1.87 ± 0.29 a	14.5 ± 0.43 a	12.1 ± 0.88 a
OPP	58.61 ± 0.51 BCE	46.91 ± 0.56 ab	46.68 ± 1.11c	37.76 ± 1.89 BCE	1.13 ± 0.17 b	1.43 ± 0.08 ab	13.7 ± 0.17 b	12.9 ± 1.01 a
BOLPH	57.85 ± 1.67c	48.13 ± 0.73 ab	45.79 ± 1.12c	35.69 ± 1.69c	0.99 ± 0.04 b	1.21 ± 0.41 b	13.6 ± 0.05 b	12.0 ± 0.91 a
MICRO	60.15 ± 1.12 ab	46.76 ± 2.18 ab	49.06 ± 1.23 b	39.79 ± 0.74 ab	2.13 ± 0.16 a	1.12 ± 0.24 b	13.5 ± 0.10 b	12.3 ± 0.77 a
MACRO	60.65 ± 1.05 ab	45.65 ± 3.06 b	47.17 ± 0.34 BCE	40.29 ± 1.32 ab	2.12 ± 0.06 a	0.88 ± 0.25 b	13.1 ± 0.49 b	11.8 ± 0.95 a
14 d at 1 °C								
CNT	57.56 ± 3.25 a	48.99 ± 1.26 a	50.86 ± 1.13 a	42.69 ± 1.06 a	–	–	14.6 ± 0.52 ab	12.5 ± 0.62 a
OPP	55.39 ± 1.95 a	45.80 ± 1.87 a	50.22 ± 1.53 a	40.66 ± 1.48 a	–	–	14.2 ± 0.15 b	11.2 ± 1.02 a
BOLPH	54.51 ± 2.00 a	46.07 ± 1.05 a	50.56 ± 0.65 a	40.73 ± 1.13 a	–	–	14.2 ± 0.20 b	11.3 ± 1.11 a
MICRO	58.21 ± 2.55 a	48.04 ± 1.76 a	51.55 ± 1.22 a	42.19 ± 1.90 a	–	–	14.7 ± 0.15 a	11.6 ± 0.70 a
MACRO	58.07 ± 1.86 a	47.18 ± 1.99 a	52.44 ± 1.22 a	41.83 ± 2.57 a	–	–	14.8 ± 0.10 a	11.8 ± 0.86 a
14 d at 1 °C plus 3 d in SMC at 20 °C								
CNT	55.01 ± 2.08 ab	44.56 ± 1.28 a	48.35 ± 1.01 a	38.26 ± 1.89 a	–	–	14.13 ± 0.20 a	11.45 ± 0.73 a
OPP	50.89 ± 1.52 c	42.13 ± 0.94 a	41.27 ± 2.01 b	33.53 ± 1.32 b	–	–	13.63 ± 0.05 b	10.75 ± 0.67 a
BOLPH	51.88 ± 2.14 BCE	42.08 ± 0.65 a	42.31 ± 1.92 b	34.07 ± 1.26 b	–	–	13.53 ± 0.25 b	10.63 ± 0.63 a
MICRO	56.01 ± 1.45 a	43.18 ± 1.42 a	45.96 ± 1.51 a	35.34 ± 2.02 ab	–	–	13.96 ± 0.11 a	11.17 ± 0.86 a
MACRO	55.66 ± 1.15 a	42.74 ± 1.49 a	46.01 ± 1.52 a	35.41 ± 2.19 ab	–	–	14.03 ± 0.11 a	11.58 ± 0.96 a

Values in column for each storage time not followed by the same letter are significantly different at $P \leq 0.05$ according to Duncan's multiple range test; "±" stands for standard deviation of the mean (n = 3).

addition, Bouzo et al. (2012) also found that in MAP packages TSS were lower or similar than in control. In contrast, Villalobos et al. (2014 and 2015b) and Bahara & Lichter (2018), in different fig cultivars stored under MAP observed an increase in TSS value. The discrepancies of these results may be attributed to the different cultivars or maturity stage at harvest or to different storage conditions.

In agreement with Caliskan & Polat (2011), Viuda-Martos et al. (2015) and Veberic & Mikulic-Petkovsek (2016), fructose and glucose were the most abundant sugars in the pulp as well as in the peel, while sucrose was very low or absent (Table 2). As previously reported (Viuda-Martos et al. 2015), fructose content was higher than glucose with an average ratio at harvest in pulp and peel of 1.14 and 1.10 g kg⁻¹, respectively (Table 2).

Fructose and glucose decreased during storage, but differences among the different film-packages were negligible. Moreover, the fructose content of pulp decreased by about 5.6% and 18.5% after 7 or 14 d of cold storage, respectively. In the same way, fructose detected in the peel decreased by about 11.5% at the end of 14 d of cold storage. After 14 d of storage in MICRO and MACRO packaged fruit, the sugar losses were lower than in the other packages, even if the differences were not significant.

Glucose content followed the same trend as fructose with a decrease of about 13% in the pulp and 16% in the peel after 14 d of cold storage, while after 7 d sucrose was no longer detectable. During the SMC periods, fructose and glucose decreased in all treatments with small differences between the different packages.

Several studies report that modified atmospheres influence sugars' metabolism even if it has not been fully clarified what is the fate of individual sugars such as fructose, sucrose, and glucose. The different responses reported in the literature may be a function of different oxygen and carbon dioxide concentrations (Cukrov et al., 2019; Brizzolaro et al., 2020). The best performances of MICRO and MACRO films can be attributed to the atmosphere generated inside the packages with CO₂ and O₂ which remained at optimal levels, while in BOLPH and OPP films, the higher concentration of CO₂ detected inside the packages after 14 days of cold storage (9.6 and 14.8 kPa respectively), but most of all at

the end of the two SMC periods (15.8 and 16.8 kPa for BOLPH and 22.3 and 30.5 kPa for OPP), might have negatively influenced the carbohydrate content (Bahar & Lichter, 2018; Colelli & Amodio, 2020).

Table 3 shows organic acids content at harvest and their changes during storage in the pulp and in the peel. Four organic acids (malic, citric, fumaric and oxalic) were detected. Except for fumaric acid, the concentration of organic acids was higher in the pulp. Malic acid was the most abundant acid both in the pulp and in the peel, followed by citric acid: together accounted for 99% and 82%, in pulp and peel respectively, of the total organic acids (Oliveira et al., 2009; Viuda-Martos et al., 2015; Veberic & Mikulic-Petkovsek, 2016).

All organic acids decreased during storage and in SMC in pulp and peel in all packages but, the different types of packaging showed a different response. In fruit wrapped with MACRO and MICRO films, the decrease of malic and citric acid detected in pulp and peel, was lower than in those wrapped with OPP and BOLPH, while, as a general trend, little changes occurred in fumaric and oxalic acid content.

In fruit of several species, the decrease of organic acids is significantly affected by MAP. In pomegranate arils stored under MAP, Belay et al. (2018) found a decrease of organic acids concentration, probably due to their involvement in the respiratory process. Similarly, Holcroft & Kader (1999) in strawberry fruit stored under controlled atmosphere found a decrease of organic acids in fruit treated with 20 kPa CO₂.

In this study the best results were achieved with MACRO and MICRO films, maybe due to the slight reduction of O₂ combined with a moderate increase of CO₂. In contrast, the heavy losses of organic acids detected in sample of BOLPH, and OPP packages can be due to the high level of CO₂ and the low partial pressure of O₂, which might have shifted respiration from aerobic to anaerobic and consequently to fermentative processes with probable involvement of organic acids in catabolic processes (Holcroft & Kader, 1999; Plotto et al., 2020).

A reduction of organic acid also occurred in un-packaged fruit, although at a slower rate.

In this case, the changes can be attributed in part to an increase of juice concentration and in part to a decrease in the rate of consumption of the substrates for the metabolic processes that occur in tissues

Table 3
Organic acids in ‘Tondo Nero’ figs stored for 7 d or 14 d at 1 °C plus 3 d in SMC at 20 °C.

	Malic acid mg kg ⁻¹		Citric acid mg kg ⁻¹		Fumaric acid mg kg ⁻¹		Oxalic acid mg kg ⁻¹	
	Pulp	Peel	Pulp	Peel	Pulp	Peel	Pulp	Peel
Harvest	745.21 ± 30.22	674.86 ± 36.06	537.21 ± 10.87	136.23 ± 8.91	82.59 ± 6.27	128.99 ± 9.16	104.40 ± 10.41	24.55 ± 2.84
7 d at 1 °C								
CNT	775.45 ± 19.17 a	686.3 ± 9.62 a	549.01 ± 11.45 a	107.25 ± 8.08 a	69.73 ± 8.27 a	118.03 ± 6.53 a	99.23 ± 8.53 a	19.08 ± 3.08 ab
OPP	571.48 ± 15.14c	609.53 ± 4.5 b	375.09 ± 11.18c	101.73 ± 5.44 a	70.19 ± 7.48 a	84.15 ± 7.98c	108.84 ± 13.64 a	22.52 ± 0.63 a
BOLPH	593.19 ± 23.19c	604.18 ± 29.9 b	366.49 ± 5.447c	97.99 ± 7.664 a	72.51 ± 4.39 a	91.37 ± 16.76 BCE	95.33 ± 8.50 a	20.78 ± 3.03 ab
MICRO	717.17 ± 6.77 b	655.05 ± 14.4 a	516.31 ± 14.27 b	100.47 ± 5.49 a	71.02 ± 10.2 a	108.70 ± 7.71 ab	108.16 ± 11.82 a	18.24 ± 2.31 ab
MACRO	727.64 ± 23.78 b	668.86 ± 20.6 a	505.38 ± 10.83 b	99.05 ± 10.22 a	79.15 ± 5.50 a	96.57 ± 10.70 BCE	104.69 ± 9.18 a	17.86 ± 1.00 b
7 d at 1 °C plus 3 d in SMC at 20 °C								
CNT	743.10 ± 17.61 a	744.20 ± 22.76 a	549.05 ± 15.29 a	111.24 ± 5.86 a	67.56 ± 7.28 a	67.32 ± 9.97 ab	103.05 ± 3.91 a	21.99 ± 2.21 a
OPP	499.24 ± 24.54 d	506.37 ± 18.74c	355.43 ± 7.591 d	110.40 ± 8.51 a	59.14 ± 8.97 abc	75.86 ± 14.0 ab	99.25 ± 5.01 ab	22.29 ± 1.71 a
BOLPH	553.38 ± 8.425c	533.99 ± 9.55 BCE	378.02 ± 6.526c	103.62 ± 4.90 a	65.99 ± 6.93 ab	63.64 ± 6.86 b	99.46 ± 4.80 ab	21.70 ± 2.05 a
MICRO	642.72 ± 15.17 b	543.92 ± 6.81 b	518.77 ± 5.779 b	114.07 ± 7.11 a	52.96 ± 7.71 BCE	63.65 ± 6.91 b	86.50 ± 10.0c	20.60 ± 1.50 a
MACRO	639.31 ± 20.69 b	568.18 ± 26.8 b	525.79 ± 10.58 b	109.65 ± 8.03 a	48.59 ± 3.12c	83.14 ± 8.67 a	86.68 ± 9.88c	18.45 ± 2.40 a
14 d at 1 °C								
CNT	539.35 ± 27.02 b	736.81 ± 18.57 a	345.01 ± 6.09 a	104.7 ± 10.55 a	75.84 ± 6.69 a	49.21 ± 8.47 a	114.43 ± 11.43 a	21.96 ± 2.03 a
OPP	415.19 ± 16.04 d	508.07 ± 27.70 d	286.37 ± 9.91 b	85.65 ± 7.62 BCE	76.62 ± 6.05 a	47.10 ± 7.74 a	115.69 ± 8.256 a	16.14 ± 2.04 b
BOLPH	478.39 ± 18.90c	565.88 ± 31.49c	304.59 ± 9.73 b	79.16 ± 7.01c	74.65 ± 4.98 a	41.01 ± 8.55 a	107.49 ± 14.61a	17.16 ± 3.53 b
MICRO	599.07 ± 50.38 a	630.15 ± 19.46 b	337.84 ± 15.75 a	96.46 ± 8.91 ab	75.39 ± 6.25 a	45.75 ± 2.38 a	98.565 ± 8.092 a	16.01 ± 2.01 b
MACRO	578.91 ± 19.64 ab	621.32 ± 14.50 b	339.10 ± 1.64 a	95.69 ± 6.45 ab	72.51 ± 4.72 a	44.99 ± 5.84 a	107.54 ± 1.68 a	16.66 ± 1.67 b
14 d at 1 °C plus 3 d in SMC at 20 °C								
CNT	468.14 ± 26.75 a	590.45 ± 8.57 a	282.67 ± 8.37 a	92.15 ± 9.04 a	67.34 ± 6.71 a	42.70 ± 3.11 a	91.54 ± 7.71 a	16.57 ± 1.64 a
OPP	338.82 ± 10.79c	414.79 ± 21.68 d	239.0 ± 9 2.06c	65.85 ± 5.61c	62.29 ± 5.49 a	41.01 ± 4.87 a	91.55 ± 7.14 a	12.24 ± 2.04 b
BOLPH	379.38 ± 20.89 b	459.37 ± 19.80c	250.34 4.26c	65.00 ± 5.60c	60.39 ± 7.63 a	34.81 ± 4.29 a	84.32 ± 5.95 a	12.39 ± 2.30 b
MICRO	492.59 ± 28.79 a	510.78 ± 10.07 b	280.27 ± 9.63 ab	77.17 ± 4.23 b	64.97 ± 4.68 a	39.93 ± 4.96 a	83.85 ± 5.34 a	14.80 ± 1.87 ab
MACRO	476.46 ± 11.06 a	500.39 ± 14.13 b	267.95 ± 6.94 b	76.89 ± 3.22 b	61.34 ± 4.11 a	39.32 ± 2.62 a	89.37 ± 4.70 a	13.66 ± 1.91 ab

Values in column for each storage time not followed by the same letter are significantly different at $P \leq 0.05$ according to Duncan's multiple range test; “±” stands for standard deviation of the mean (n = 3).

Table 4

Phenolic compounds detected in 'Tondo Nero' fruit pulp stored for 7 d or 14 d at 1 °C plus 3 d in SMC at 20 °C. Values expressed as mg kg⁻¹. (Van: Vanillic acid glucoside; Cat: Catechin; Caff exo: Caffeoylquinic acid hexoside; Dihy: Dihydroquercetin; Chl: Chlorogenic acid; Ap gluc: apigenin glucoside; Cyan: Cyanidin-3-rutinoside; Fer ex: Ferulic acid hexoside; Que: Quercetin-3-glucoside; Rut: Rutin).

	Van	Cat	Caff exo	Dihy	Chl	Ap gluc	Cyan	Fer ex	Que	Rut
Harvest	2.71	4.81	6.16	5.14	2.49	29.4	5.00	6.68	13.28	15.67
7 d at 1 °C										
CNT	2.27 a	4.02 a	6.05 a	4.67 a	2.45 a	29.51 a	5.02 a	5.66 ab	15.46 b	13.69 b
OPP	2.44 a	3.75 a	5.91 a	3.44 b	2.02 a	23.61 b	4.42 a	4.93 b	15.89 ab	12.63 b
BOLPH	2.32 a	4.36 a	5.54 a	4.26 ab	2.23 a	27.00 ab	4.77 a	6.30 a	16.49 ab	14.06 b
MICRO	2.65 a	4.09 a	6.33 a	4.57 a	2.48 a	28.89 a	4.96 a	6.19 a	17.13 ab	16.41 a
MACRO	2.77 a	3.91 a	6.26 a	4.79 a	2.37 a	28.94 a	4.81 a	6.03 a	17.59 a	15.85 a
7 d at 1 °C plus 3 d in SMC at 20 °C										
CNT	2.05 a	3.92 a	5.44 BCE	3.81 a	2.38 a	27.11 a	4.43 a	5.34 BCE	16.11 a	12.75 b
OPP	2.39 a	3.11 b	5.09c	2.81 a	1.91 a	22.31 a	4.13 a	4.65c	15.69 a	10.37c
BOLPH	2.42 a	3.53 ab	5.85 ab	3.48 ab	2.21 a	24.14 a	4.40 a	6.19 a	17.17 a	13.87 ab
MICRO	2.73 a	3.83 a	6.12 a	3.98 a	2.29 a	26.41 a	4.67 a	5.78 ab	17.40 a	15.59 a
MACRO	2.85 a	3.74 a	6.06 ab	4.18 a	2.16 a	25.76 a	4.83 a	5.90 ab	17.12 a	15.63 a
14 d at 1 °C										
CNT	2.26 b	3.43 ab	5.78 BCE	4.26 a	2.10 a	22.71 ab	4.55 a	5.60 ab	18.89 b	13.36 b
OPP	2.34 b	2.85 b	5.38c	3.30 b	1.90 a	17.42c	4.09 a	4.93 b	15.97 b	9.52c
BOLPH	2.95 a	3.34 ab	5.52 BCE	4.26 a	1.93 a	20.33 BCE	4.63 a	5.98 a	17.31 a	14.98 ab
MICRO	3.05 a	3.74 a	6.21 ab	4.43 a	2.02 a	24.26 a	4.77 a	5.83 a	17.43 a	15.79 a
MACRO	3.06 a	3.77 a	6.68 a	4.72 a	2.10 a	24.70 a	4.80 a	6.11 a	17.31 a	15.33 a
14 d at 1 °C plus 3 d in SMC at 20 °C										
CNT	1.88 b	3.39 a	5.29 ab	3.46 ab	1.94 ab	18.63 a	4.09 a	5.07 BCE	17.81 b	10.69 b
OPP	1.89 b	2.81 b	4.86 b	2.51c	1.55 b	14.14 b	3.99 a	4.74c	18.97 ab	8.75c
BOLPH	2.18 b	3.30 ab	5.35 ab	3.14 b	1.82 ab	15.62 b	4.06 a	5.41 ab	19.40 ab	12.07 ab
MICRO	2.59 a	3.73 a	5.72 a	3.64 ab	2.16 a	19.62 a	4.40 a	5.37 ab	19.69 ab	13.08 a
MACRO	2.70 a	3.64 a	5.76 a	4.02 a	2.05 ab	20.00 a	4.53 a	5.62 a	19.94 a	12.70 a

Values in column for each storage time not followed by the same letter are significantly different at $P \leq 0.05$ according to Duncan's multiple range test.

Table 5

Phenolic compounds detected in 'Tondo Nero' peel stored for 7 d or 14 d at 1 °C plus 3 d in SMC at 20 °C. Values expressed as mg kg⁻¹. (Van: Vanillic acid glucoside; Cat: Catechin; Caff exo: Caffeoylquinic acid hexoside; Dihy: Dihydroquercetin; Chl: Chlorogenic acid; Ap gluc: apigenin glucoside; Cyan: Cyanidin-3-rutinoside; Fer ex: Ferulic acid hexoside; Que: Quercetin-3-glucoside; Rut: Rutin).

	Van	Cat	Caff exo	Dihy	Chl	Ap gluc	Cyan	Fer ex	Que	Rut
Harvest	9.54	23.56	13.63	52.02	9.13	87.91	31.09	44.42	68.36	381.15
7 d at 1 °C										
CNT	9.54 a	21.09c	15.19 a	49.40 a	9.15 a	82.89 a	30.76 a	39.58 a	50.58 BCE	340.04 b
OPP	9.77 a	21.47 BCE	14.74 a	48.74 a	8.56 a	82.44 a	24.19 b	44.67 a	44.14c	346.95 b
BOLPH	9.86 a	23.39 ab	14.45 a	49.42 a	9.41 a	82.28 a	26.30 ab	42.10 a	57.33 ab	353.47 ab
MICRO	10.5 a	24.91 a	15.12 a	51.43 a	9.47 a	88.67 a	30.17 ab	41.81 a	64.28 a	370.06 a
MACRO	10.5 a	25.15 a	14.60 a	50.98 a	9.16 a	87.48 a	30.44 ab	43.13 a	61.58 a	373.73 a
7 d at 1 °C plus 3 d in SMC at 20 °C										
CNT	11.98 a	22.55 b	15.10 a	49.54 a	8.92 a	93.45 a	29.27 a	36.34 a	27.71 b	325.52 ab
OPP	9.045c	22.61 b	12.40 a	46.99 a	7.57 a	91.20 a	19.81 d	30.41 a	31.14 b	310.67 b
BOLPH	10.57 b	26.83 a	14.37 a	50.80 a	8.75 a	92.76 a	21.06 cd	31.41 a	32.13 b	336.49 a
MICRO	10.44 b	25.16 a	13.73 a	51.20 a	9.55 a	96.52 a	24.51 BCE	31.95 a	51.68 a	343.49 a
MACRO	11.12 ab	26.75 a	13.52 a	50.03 a	9.26 a	97.64 a	25.08 ab	33.03 a	46.18 a	341.14 a
14 d at 1 °C										
CNT	11.68 a	21.90 BCE	12.01 a	47.82 a	7.63 ab	80.24 b	31.14 a	38.43 a	37.85 b	322.59 b
OPP	6.841c	19.11c	11.31 a	46.97 a	6.49 b	80.65 b	21.81c	42.34 a	39.32 b	318.74 b
BOLPH	7.955c	22.51 b	11.15 a	46.96 a	9.15 a	81.55 b	26.01 b	37.73 a	42.78 b	340.59 a
MICRO	10.34 b	25.57 a	12.64 a	49.58 a	9.32 a	86.91 a	29.66 a	46.54 a	56.12 a	353.49 a
MACRO	11.25 ab	25.70 a	12.51 a	50.57 a	9.98 a	88.22 a	29.47 a	37.99 a	61.28 a	351.35 a
14 d at 1 °C plus 3 d in SMC at 20 °C										
CNT	11.78 a	22.43 b	11.76 a	46.89 b	7.29 a	84.88 b	27.74 a	35.77 a	31.52 b	312.93 b
OPP	8.00 b	20.31c	11.39 a	46.41 b	7.01 a	86.56 b	18.14c	39.01 a	34.66 b	307.74 b
BOLPH	8.78 b	23.04 b	11.04 a	46.34 b	8.26 a	87.21 b	22.68 b	36.39 a	36.78 b	336.92 a
MICRO	10.83 a	25.11 a	12.38 a	48.89 ab	8.99 a	92.73 a	24.47 ab	41.59 a	50.45 a	345.16 a
MACRO	11.83 a	25.57 a	12.32 a	50.19 a	9.47 a	93.18 a	25.66 ab	37.32 a	55.61 a	343.29 a

Values in column for each storage time not followed by the same letter are significantly different at $P \leq 0.05$ according to Duncan's multiple range test.

experiencing excessive transpiration.

Organic acids, even in small amounts, represent important compounds that, together with sugars, markedly affect the taste, balancing the sweetness and enhancing the flavor of the fruit. The sensory properties of organic acids depend on the types of organic acid present and their concentrations and are due to the acidity, bitterness, and astringency they impart to the fruit (Hartwig & McDaniel, 1995). As it happens with organic acids, the impact that sugars have on taste depends on

their concentration as well as on their specific sweetening properties. A higher perception of sweetness can be associated to a higher content of fructose, thanks to its higher Eisenberg index compared to glucose (1.45 – 1.75 vs 0.6 – 0.75) (Caliskan & Polat, 2011). In our study, the combined higher levels of organic acids, fructose, and glucose detected in fruit packaged with MICRO and MACRO films might be the reason why this fruit got a higher score by panelists.

3.4.2. Phenolic compounds

In figs, phenolics (flavonol glycosides, hydroxycinnamic acids, anthocyanins, and phenolic acids) are important compounds which, besides affecting the flavor, play an important nutritional role in the diet thanks to their high antioxidant capacity and nutraceutical properties (Barolo et al., 2014; Palmeira et al., 2019; Adiletta et al. 2019; Adiletta et al., 2022). Phenolics content is greatly influenced by pre-harvest environmental conditions, agronomic practices, cultivar, and post-harvest technologies (Veberic et al., 2008; Ammar et al., 2015a; Palmeira et al., 2019).

Tables 4 and 5 report the qualitative phenolic profile detected by chromatography analysis and the effect of the different MAPs on their changes during storage. Ten phenolic compounds were detected and quantified even if the chromatographic profiles showed some peaks that were not identified for being: a) not completely separated; b) below the detection limits; or c) poorly ionized in mass analysis. However, the area of the detected peaks was 90% of the total area.

In accordance with Ammar et al., (2015b), in fig pulp, the flavones apigenin-O-glucoside was among the most abundant flavonoids followed by quercetin-3-O-glucoside, while in peel the most abundant was rutin, followed by apigenin-O-glucoside (Del Caro & Piga, 2008; Hssaini et al., 2021). Regarding ferulic acid exoside, at harvest significant differences between pulp and peel were found with values of 6.6 and 44.4 mg kg⁻¹ respectively.

Among the phenolic acids, chlorogenic acid ranged between 2 mg kg⁻¹ (pulp) to 9 mg kg⁻¹ (peel) (Veberic & Mikulic-Petkovsek, 2016). In agreement with several data reported in literature, cyanidin-3-O-rutinoside was detected as the prevalent anthocyanin pigment both in the pulp and peel (Solomon et al., 2006; Del Caro & Piga, 2008; Duenas et al., 2008; Vallejo et al., 2012). However, significant differences in the anthocyanins content were found between the pulp and peel (Tables 4 and 5).

As a general trend, overall phenolic compounds decreased as in-package CO₂ increased (Tables 4 and 5). Pulp vanillic acid glucoside, catechin, dihydroquercetin, chlorogenic acid, apigenin glucoside, ferulic acid hexosaide and rutin, decreased at different rates according to the

different MAP generated inside the packages with the highest losses occurring in OPP packages and the lowest in fruit packed with MACRO and MICRO perforated films (Table 4).

Pulp caffeoylquinic acid hexoside content was differently influenced by treatments. After 14 d of storage at 1 °C, its content increased in MICRO and MACRO packages while decreased in OPP and BOLPH packages. At the end of the SMC period, caffeoylquinic acid hexoside content diminished in all packages, especially in OPP ones (21%). In contrast, in MACRO e MICRO perforated films, pulp quercetin-3-glucoside increased by about 31% after 14 d at 1 °C and by 48% at the end of the subsequent SMC period. In OPP film, at the same sampling times, quercetin-3-glucoside increased by about 21% and 31%, respectively (Table 4).

In pulp, cyanidin-3-rutinoside showed an overall decreasing trend with losses of about 4% at the end of cold storage and 20% at the end of SMC (Table 4); the highest losses were detected in fruit packaged with OPP film; in the other packaging treatments differences were not always consistent and statistically different, likely for the intrinsic high variability among individual fruit.

Peel vanillic acid glucoside, catechin, quercetin-3-glucoside and rutin were significantly affected by MAP. After 14 d of storage at 1 °C and after the following SMC periods, vanillic acid glucoside and catechin increased in MACRO and MICRO packages while decreased in OPP and BOLPH packages. Quercetin-3-glucoside and rutin showed a significant decline, especially in OPP and BOLPH packages. Cyanidin-3-rutinoside was stable during cold storage in fruit packed with MACRO and MICRO film, but decreased by about 15% at the end of cold storage and by up to 41% at the end of SMC (Table 5) in OPP and BOLPH. In contrast, slight changes occurred in caffeoylquinic acid hexoside, dihydroquercetin, chlorogenic acid, apigenin glucoside and ferulic acid hexoside (Table 5).

The effect of high carbon dioxide concentrations on inhibiting the synthesis or lowering the stability of anthocyanins in fruit has been reported by several authors. Remon et al. (2004), showed that high levels of in-package CO₂ were associated with a lower anthocyanin content in 'Burlat' cherries, compared with packages with lower levels of CO₂. A

Table 6

Ascorbic acid, total phenolics and antioxidant properties in 'Tondo Nero' fruit pulp and peel stored for 7 d or 14 d at 1 °C plus 3 d in SMC at 20 °C.

	Total Phenol mg kg ⁻¹		Ascorbic acid mg kg ⁻¹		Antioxidant capacity TEAC mmoli kg ⁻¹	
	Pulp	Peel	Pulp	Peel	Pulp	Peel
Harvest	296.28 ± 5.84	876.57 ± 26.35	17.77 ± 1.19	20.96 ± 0.83	304.35 ± 8.46	1181.26 ± 68.51
7 d at 1 °C						
CNT	297.66 ± 5.17 ab	855.21 ± 19.17 ab	18.43 ± 1.34 a	21.75 ± 1.09 a	294.45 ± 14.41 ab	1192.00 ± 18.96 a
OPP	287.47 ± 4.31 b	826.63 ± 12.71c	17.19 ± 0.95 ab	21.31 ± 0.73 a	264.81 ± 12.74c	1153.16 ± 11.97 b
BOLPH	293.88 ± 6.35 ab	830.24 ± 14.77 BCE	17.05 ± 0.51 ab	20.47 ± 0.50 a	282.16 ± 8.40 BCE	1161.63 ± 25.02 ab
MICRO	302.40 ± 7.50 a	857.47 ± 17.16 ab	16.74 ± 0.39 b	20.48 ± 1.06 a	296.36 ± 6.53 ab	1193.07 ± 20.92 a
MACRO	304.46 ± 5.08 a	865.11 ± 7.26 a	17.08 ± 0.62 ab	21.01 ± 1.27 a	303.29 ± 4.76 a	1199.19 ± 22.15 a
7 d at 1 °C plus 3 d in SMC at 20 °C						
CNT	306.47 ± 6.63 a	724.55 ± 16.17 ab	18.13 ± 0.89 a	19.65 ± 1.17 ab	306.36 ± 9.63 ab	95.10 ± 36.46 ab
OPP	292.70 ± 4.79 b	705.00 ± 15.15 b	15.56 ± 0.86 b	18.66 ± 0.51 b	294.23 ± 9.48 b	925.098 ± 17.41c
BOLPH	295.73 ± 3.19 b	736.55 ± 20.02 a	16.64 ± 0.39 ab	20.91 ± 1.15 a	295.06 ± 7.40 b	977.42 ± 19.44 b
MICRO	306.47 ± 2.71 a	744.05 ± 13.56 a	16.91 ± 0.59 ab	20.97 ± 1.12 a	312.10 ± 9.51 a	1006.87 ± 21.73 ab
MACRO	307.24 ± 4.25 a	754.49 ± 17.14 a	17.41 ± 1.21 a	20.57 ± 0.58 a	313.95 ± 6.98 a	1037.98 ± 27.63 a
14 d at 1 °C						
CNT	302.71 ± 6.54 ab	815.46 ± 25.57 ab	17.91 ± 0.37 a	20.73 ± 1.62 a	304.53 ± 16.05 ab	1154.26 ± 14.21 b
OPP	293.73 ± 5.87 b	763.43 ± 28.95c	15.67 ± 0.52 b	19.46 ± 1.24 a	289.57 ± 6.56 b	1060.75 ± 15.73 d
BOLPH	304.89 ± 4.71 a	784.29 ± 13.53 BCE	15.55 ± 0.40 b	19.19 ± 0.97 a	304.00 ± 9.79 ab	1112.63 ± 20.30c
MICRO	305.56 ± 5.22 a	822.77 ± 6.75 a	16.57 ± 1.09 b	21.02 ± 0.98 a	310.44 ± 5.23 a	1169.38 ± 11.25 ab
MACRO	309.03 ± 3.89 a	833.67 ± 16.07 a	16.29 ± 0.38 b	20.21 ± 0.93 a	313.89 ± 8.79 a	1197.02 ± 16.79 a
14 d at 1 °C plus 3 d in SMC at 20 °C						
CNT	305.31 ± 7.75 a	712.30 ± 21.73 a	13.92 ± 0.60c	17.37 ± 0.89 ab	297.98 ± 13.17 b	995.92 ± 16.70 b
OPP	277.87 ± 8.96 b	660.44 ± 21.58 b	13.96 ± 0.55c	15.82 ± 1.26 b	273.85 ± 9.51c	895.76 ± 19.47c
BOLPH	304.14 ± 7.63 a	692.84 ± 12.81 ab	14.96 ± 0.55 BCE	18.52 ± 0.70 a	308.37 ± 7.09 ab	990.20 ± 11.89 b
MICRO	306.42 ± 10.93 a	716.89 ± 16.58 a	16.19 ± 0.80 a	19.58 ± 1.35 a	314.77 ± 7.79 ab	1078.45 ± 26.24 a
MACRO	313.61 ± 11.24 a	714.73 ± 27.50 a	15.88 ± 0.53 ab	19.25 ± 1.51 a	324.79 ± 10.29 a	1045.80 ± 14.88 a

Values in column for each storage time not followed by the same letter are significantly different at P ≤ 0.05 according to Duncan's multiple range test; "±" stands for standard deviation of the mean (n = 3).

similar trend was observed by Palma et al. (2015) in ready-to-eat pomegranate arils wrapped with a polypropylene film and stored at 5 °C. Gil et al. (1997) reported that high CO₂ led to a decrease in anthocyanins content of internal tissues of strawberries but did not affect the content of external tissue.

These results confirm that MAPs can preserve content anthocyanin of various agricultural products if CO₂ levels are within tolerated limits (Gil et al. 1995 and 1997).

Babic et al., (1993) and Amanatidou et al., (2000) found an inhibition in the synthesis of phenolic compounds in cut carrots stored in an CO₂ enriched atmosphere compared to samples stored in air. Bahar & Lichter (2018) reported an increase in browning coloration of fig pulp stored in controlled atmosphere when CO₂ level increased from 5% to 10% or 15%, whereas Macheix et al. (1990) reported a decrease in flavonols, caffeoyl tartaric and p-coumaroyl tartaric acids in pulp and peel of grape berries when stored in anaerobic conditions. Similarly, MAP with 12–14% of CO₂ decreased the content of flavonol in minimally processed red lettuce (Gil et al., 1998). In unwrapped fruit, the decrease in phenolic compounds detected in pulp and peel might be a consequence of physiological alterations as membranes deterioration or loss of cell structure due to the excessive water loss (Ben-Yehoshua & Rodov, 2003).

3.4.3. Total phenol content, ascorbic acid and antioxidant scavenging activity

Peel and pulp TPC changes during cold storage and SMC were affected by the different MAPs (Table 6). In OPP packages TPC showed a general decrease, with higher losses detected in peel, which accounted for 12.9%, and 24.6% after 14 d of storage and at the end of SMC, respectively; lower variations occurred in pulp and peel fruit packed with MICRO and MACRO perforated film.

These data were confirmed by the sum of the concentrations of individual phenolic compounds detected by chromatography (data not shown).

In fig fruit packaged with OPP film, the greatest decrease in TPC can be attributed to the high level of CO₂ and low O₂ concentrations, which might counteract the biosynthetic pathway of phenols. An enhanced activity of polyphenol oxidase and phenylalanine ammonia lyase, might have sustained the synthesis and oxidation of phenolics compounds mediated by the decrease of O₂ levels and the increase of CO₂ (Gil et al., 1998; Reyes et al., 2007; Kader, 2009; Cantos et al., 2001; Cukrov et al., 2019). Similar results were obtained by Guillén et al. (2015) and Zidi et al. (2020) who reported that storage under modified atmosphere delays the accumulation of phenolic compounds of figs fruit.

Ascorbic acid content at harvest was 17.77 mg kg⁻¹ in fruit pulp and 20.96 mg•kg⁻¹ in peel (Table 6). Its level declined moderately in pulp during storage and at a higher rate during SMC. The greatest degradation occurred in fruit packed with OPP film, where a loss of about 7% and 11% was detected in the pulp at the end of cold storage and SMC and a reduction of 22% and 24% was detected in the peel at the same sampling time, respectively. Lower losses (about 9% over the whole storage time) were detected in pulp and peel of fruit packaged in MICRO and MACRO films (Table 6). Similar results were observed by Irfan et al. (2013) in figs treated with different calcium chloride solutions and Agar et al., (1997) in berry fruit stored in high CO₂ and low O₂ atmospheres.

Normally, ascorbic acid decreases with storage in most horticultural products at a rate that depends on the genotype and the storage conditions (temperature, in-package CO₂, O₂ and C₂H₄ concentrations). In OPP film the high decrease of ascorbic acid in fruit pulp during the shelf-life can be explained with the high levels of CO₂ and C₂H₄.

At harvest, the peel TEAC value was 1181.26 (Table 5). As shown in Table 5 the antioxidant activity was significantly affected by packaging treatments. A marked decrease of TEAC values was observed during storage in fruit packaged with OPP film with final losses of 24.1% in pulp and 22.1% in peel, compared to harvest time. In the other packaging treatments negligible changes occurred in the pulp, whereas a

significant reduction in the peel occurred only in BOLPH packages (Table 6). Villalobos et al. (2015a) with the DPPH test, found no significant difference in figs packaged with different types of films, while with the ABTS test they found a slight increase in antioxidant activity in figs packaged with micro-perforated film. In another study, Guillén et al. (2015) found that modified atmosphere packaging did not affect the antioxidant activity of figs. In contrast, Zidi et al. (2020) detected a decrease of phenolic compounds and DPPH radical scavenging capacity of fresh Algerian figs packaged with a micro-perforated film.

In this study the results achieved with the OPP film were affected by the high concentration of CO₂, which overcame those considered optimal and which had negative effects on the phenolic compounds and ascorbic acid content the main components that determine the fruit antioxidant activity. (Colelli & Amodio, 2020).

The evolution of antioxidant activity during storage was complementary to the evolution of phenolic compounds and ascorbic acid content. Several studies have reported a close relationship between antioxidant activity and total phenolics (Heim et al., 2002; Silva et al., 2007).

In accordance with Del Caro and Piga (2008), Caliskan & Polat, (2011) and Zidi et al., (2020), the results indicate a good correlation between the TPC and TEAC and ascorbic acid, with *r* values ranging from 0.5 to 0.9, except for the non-significant interaction between the values of TEAC and ascorbic acid in the pulp (Table S2 in Supplementary Material).

4. Conclusion

The main objective of this study was to compare the physiological, nutritional, nutraceutical and overall acceptability during postharvest storage under different modified atmospheres of an important fig cultivar (i.e., 'Tondo Nero') grown in Sardinia to identify the most suitable packaging to preserve fruit quality.

Our results show that the optimal MAPs generated by the MICRO and MACRO perforated films not only extend the shelf-life but also allow to maintain a satisfactory quality, compared to unwrapped figs, even when fruit are stored under simulated marketing conditions.

In contrast, the modified atmospheres generated by the OPP film, with extremely high levels of CO₂ and O₂ concentrations close to 0 kPa, had a negative effect on overall acceptability, which decreased below the limit of marketability just after 7 d of storage at 1 °C followed by 3 d of storage at 20 °C.

These results could be very important from the commercial point of view, considering the extreme perishability of figs after harvesting.

CRedit authorship contribution statement

Dr Palma and Dr. D'Aquino designed the experimental plan, conducted all the laboratory analysis, carried out the statistical analysis and wrote the manuscript, Dr. Muntoni contributed to the experimental design, carried out all the operation related to preharvest treatments, organized the harvesting and critically contributed to the final version of the manuscript.

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.

Data Availability

Data will be made available on request.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.fpsl.2023.101030](https://doi.org/10.1016/j.fpsl.2023.101030).

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