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ORIGINAL ARTICLE

Sensory and physicochemical changes in gluten-free oat biscuits stored under different packaging and light conditions

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Abstract The influence of different packaging films and their thickness (polyethylene terephthalate/casting polypropylene (PET/CPP_34), biaxially oriented polypropylene (BOPP_40), polyvinylchloride (PVC_12), BOPP/CPP_50 and polyethylene/ethylene vinyl alcohol/polypropylene (PE/EVOH/PP_50) on the quality of gluten-free oat biscuits was evaluated for a storage period up to 3 months under light and darkness conditions. Periodically (day 30, 60 and 90), physical parameters, peroxide value, texture and microbiological parameters together with sensory attributes (surface colour, smell, taste, crunchiness and off-flavour) were assessed. Moisture and water activity of biscuits decreased during storage in all packages. The highest peroxide value was obtained for biscuits packed in PVC_12, while the lowest was for the PE/EVOH/PP_50, for both storage conditions. The biscuits' colour changed from yellow–brown to light yellow and the change was more pronounced in the light as compared to the dark storage conditions. The hardness value decreased ($p > 0.05$) during the storage period. The electronic nose system showed that the distinct volatile composition of the biscuits stored in the light was correlated with the higher scores of the off-flavour attribute and with the peroxide values. The sensory data showed that BOPP/CPP_50 preserved the colour of the biscuits, while PE/EVOH/PP_50 kept the initial crunchiness of the biscuits up to 90 days of storage in both light and dark conditions. The study suggested that BOPP/CPP_50 and PE/EVOH/PP_50 can be used for gluten-free oat biscuits' packaging and storage up to 90 days for both conditions studied, without adversely affecting their physicochemical and sensory properties.

Keywords Gluten-free oat biscuits · Packaging materials · Time · Physicochemical parameters · Sensory analysis

Introduction

Among bakery products, biscuits are the most popular food products and widely consumed by a range of people, due to their varied taste, long shelf-life and relatively low cost (Nagy et al. 2012; Vitali et al. 2009).

Fat-rich biscuits are affected by their fat content due to environmental factors such as light and oxygen during storage, reducing the quality and nutritional value of biscuits (Lu and Xu 2010). Rancidity is a major problem in food systems which occurs through lipid oxidation and significantly affects quality of foods, deteriorating their appearance, taste and smell (Chatterjee et al. 2014). The main effect of lipid oxidation, the most common processes in the food system, is the development of off-flavours and odours which shorten the shelf-life of foodstuffs and make them unacceptable for consumption (Bialek et al. 2016).

In a previous work, the role of lipid oxidation and humidity absorption on the shelf-life of wheat flour biscuits was studied based on a mathematical model (Patrignani et al. 2014). The results indicated that an increase in the peroxide level, even at a low level of lipid oxidation, increases Young's modulus and leads to a harder biscuit, while the humidity absorption lowers the fracture stress and Young's modulus.

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Besides chemical parameters (such as: peroxide value, conjugated dienes or para-anisidine value), sensory analysis is a suitable methodology for measuring oxidative damage and determining the shelf-life of foods. Some studies proposed correlations between sensory scores and chemical measurements of lipid oxidation products (Zajdenwerg et al. 2011).

Packaging materials have a major impact on the quality and shelf-life of biscuits (Robertson 2011). Changes in the quality of packed foods, mainly, discolouration defect and off-flavour formation, are initiated in the presence of light and at a long exposure.

According to the permeability to gases and water vapour, different types of packaging can protect from the degradation of lipids in biscuits. Jan et al. (2017) showed that the packaging material and the storage conditions influenced the quality attributes of gluten-free extrudates and cookies. They found that moisture content, water activity, free fatty acids and peroxide value of stored products increased in all packages, while the hardness, L values and sensory scores reduced. A higher increase in moisture, free fatty acids, peroxide value and microbial level (visible colonies of yeasts and moulds, CFU/g) were observed in low density polyethylene, in comparison to laminated pouches of metalized polyester polyethylene.

Worldwide, there is an increase in the consumption of gluten-free biscuits that will contribute to the growth of the gluten-free bakery products market. Thus, the global gluten-free product market is projected to reach USD 7.60 billion by 2020 (Statistica 2019). To our knowledge, no information is available on the storage changes of the gluten-free oat biscuits. With this in mind, the aim of the study was to monitor the physicochemical and sensory changes related to oxidation during storage of the gluten-free oat biscuits up to 90 days. The biscuits were stored in different types of packaging materials, widely used in food industry and under light (to simulate samples' exposure in retail stores) and darkness conditions at room temperature.

The analysis of the odour profile of gluten-free oat biscuits using an electronic nose system was also performed.

Materials and methods

Gluten-free oat biscuits' preparation

The gluten-free oat biscuit formulation was: 1:1 gluten-free oat flour and gluten-free oat bran (Glebe Farm Foods LTD, UK), vegetal shortening containing 60% fat (26.6%), sugar (21.9%), eggs (33.3%), sodium bicarbonate (0.6%), ammonium bicarbonate (0.6%), citric acid (0.4%) and water (25%). The percentage of the ingredients was calculated based on the dry mixture (oat flour and oat bran). Fat, sugar, eggs and raising powders were purchased locally. Fat and sugar were creamed in a spiral mixer with two speeds with a kneading capacity of 60 kg (SP60, Avancini SPA, Italy) for 10 min, then eggs were added and mixed continuously for 10 min at speed 2, then the rising agents dissolved in water and the flour were added and mixed for another 10 min. Then, biscuits of 8 cm length, 2 cm width and 1 cm height, weighing 8 g, were obtained using a dropping machine (Mimac Italia Srl, Piovene Rocchette, model MINIDROPPRO 400-Y7). The biscuits were placed on trays and baked in a rotative conventional oven (Gima Forni, Italy) for 40 min at 190–210 °C.

After reaching ambient temperature, the biscuits were packed in different types of packaging and stored under controlled conditions. The proximate composition of fresh oat biscuits was determined as described in a previous study (Duta and Culetu 2015).

Packaging and storage

After cooling, 150 g biscuits were introduced in polyethylene terephthalate trays (160 mm length × 95 mm width × 50 mm depth; 0.132 mm thickness) which were wrapped with different transparent films, using a sealing machine (Impulse Bag Sealer, Lovero, Korea). The films used were (the abbreviation included the films' thickness in µm): 12 µm polyethylene terephthalate/22 µm casting polypropylene (PET/CPP_34) and biaxially oriented polypropylene (BOPP_40) from Fimplast Impex (Ploiesti, Romania), polyvinylchloride (PVC_12) (M&M Product, Brasov, Romania), 20 µm BOPP/30 µm CPP (BOPP/CPP_50) (Fimplast Impex, Ploiesti, Romania) and polyethylene/ethylene vinyl alcohol/polypropylene (PE/EVOH/PP_50) (Coexpan, Spain). These films were selected for testing due to their wide application in food packaging. Oxygen transmission rate (OTR) measurement on films was performed using a manometric gas permeation analyzer (Lyssy L100-5000, PBI Dansensor, Switzerland) according to ISO 2556 (1974). Water vapour transmission rate (WVTR) was performed by means of a water vapor permeation analyzer (Lyssy L80-5000, PBI Dansensor, Switzerland) according to ISO 15106-1 (2003).

The effect of storage time of the packed gluten-free oat biscuits at room temperature (20 °C and 44.5% relative humidity) was investigated under two different conditions: light (L) to simulate samples' exposure in retail stores and dark (D), respectively. The packed samples were lined on a table one by one. The source of light used was a Philips TL-D 18 W lamp (Extreme Cool Daylight Snow White, Poland) placed 1.6 m above the samples. The darkness conditions were assured by covering the top of the trays with large aluminium foil. Gluten-free oat biscuit samples were analyzed initially after baking (day 0, for the unpacked biscuit) and at regular intervals for chemical, microbiological and sensory parameters up to 90 days at day 30 (30 d), day 60 (60 d) and day 90 (90 d), respectively. The same analysis procedure was carried out for all the packagings considered. One new tray with 150 g of biscuits samples was used for each analysis.

Moisture content and water activity determination

Gluten-free oat biscuit samples were ground in a mill (model SM-1, MRC Ltd., Israel) and the moisture content was determined using a Moisture Analyzer (Mettler Toledo LJ16, Greifensee, Switzerland). Water activity of the milled biscuits was determined at 25 °C with an a_w-Value Measuring Device type Aquaspector AQS-2-TC (Nagy Messsysteme GmbH, Gäufelden, Germany) (ISO 18787 2017b). All samples were analyzed in triplicate.

Determination of peroxide value in oat biscuits

Peroxide value (PV) of the lipid fraction extracted from the milled gluten-free oat biscuits was determined based on AOAC (2005). Briefly, lipids were extracted with chloroform and 10 mL of the extracted lipids were dissolved in 15 mL glacial acetic acid and 1 mL of saturated KI aqueous solution. The sample was stirred and left for 5 min in darkness, followed by the addition of 75 mL of water and 1 mL of a starch solution. Formed iodine was titrated with 0.01 N sodium thiosulfate. The PV was expressed as miliequivalents of active oxygen per kilogram of lipids (meq O_2/kg of lipids). All samples were analyzed in triplicate.

Colour measurement

The surface colour of the gluten-free oat biscuits was measured using a Chroma Meter CR-410 (Konica Minolta Sensing, Inc., Osaka, Japan) with a D65 illuminant and a 2° observer angle. Parameters L* (where L* = 0 is black and L* = 100 represents white), a* (green–red) and b* (blue–yellow) were determined. Before measurements, the instrument was calibrated against a white tile. Readings were performed on 10 different points of each sample during storage in different film packagings. The colour difference (ΔE) between fresh and stored biscuits as well as between biscuits stored under light and dark conditions was calculated as: $\Delta E = \sqrt{\left(L_C^* - L_S^*\right)^2 + \left(a_C^* - a_S^*\right)^2 + \left(b_C^* - b_S^*\right)^2}$, where subscripts C refer to the control biscuits, while S refer to the stored biscuits (Francis and Clydesdale 1975).

Texture analysis

The texture properties of gluten-free oat biscuits were measured with an Instron Texture Analyzer (model 5944, Illinois Tool Works Inc., USA) using a compression test according to the procedure described by Duta and Culetu (2015). Eight biscuits from each sample were analyzed by compression test.

Headspace-electronic nose

The headspace of the gluten-free oat biscuits samples was performed on an electronic nose system combined with HS100 auto-sampler together with α-Soft version 8.0 software for data processing (Alpha M.O.S.—model FOX 4000, Toulouse, France). The system consists of an array of 18 different metal oxide sensors placed in three controlled temperature chambers.

One g of ground gluten-free oat biscuits, accurately weighed, was placed in a 10 mL vial, hermetically sealed with a polytetrafluoroethylene (PTFE)/silicone septum and incubated for 600 s at 80 °C under agitation (500 rpm) to allow the volatilization of flavour compounds into the headspace. Synthetic air and nitrogen (Linde Gas, Romania) were used as carrier gas with a flow of 150 mL/min. 1500 µL of the biscuit sample headspace were injected into the measuring chamber of the electronic nose, with an acquisition time of 120 s. All samples were run in triplicate and the individual signals recorded were used for statistical analysis. Principal component analysis (PCA) was used to detect the differences in the overall volatile composition among oat biscuit samples stored for different times (30 d, 60 d and 90 d) and conditions (light and dark). A discrimination index was calculated by the software. The higher the discrimination index, the better is the discrimination (Alpha MOS 2002).

Sensorial evaluation

Sensory testing was carried out in a sensory laboratory equipped with 6 individual booths according to ISO 8589 (2007). 10 samples of gluten-free oat biscuits packed in different films and stored under light and dark conditions were analyzed after 30 d, 60 d and 90 d. A panel of 10 trained panelists (ages 24–60 years) evaluated the samples for the quantitative descriptive attributes of biscuits. Panelists were selected and trained in accordance with ISO 8586 (2012). Training was performed on the basis of product characteristics and the panelists were trained with selected sensory characteristics for gluten-free oat biscuits and criteria for sensory evaluation. Biscuits were evaluated for surface colour (scale from 1-pale, light to 5-well-baked, intense colour), smell and taste (scale from 1-tasteless, odourless to 5-intense), crunchiness at the first bite (scale from 1-very smooth, gummy, wet to 5-very crunchy) and off-flavour (atypical flavour covering flavour from package, rancidity; scale from 1-not identified to 5-very strong). The samples were identified with a three-digit code. At each session the panelists received a whole biscuit from each sample. The panelists were instructed to rinse their mouths with water between sample evaluations. A set of six samples was evaluated firstly and after 30 min break the second set of 4 biscuits was evaluated to avoid fatigue (Serrem et al. 2011).

Microbiological analysis

Decimal dilutions of gluten-free oat biscuit samples in peptone water were pour plated on Plate Count Agar medium (Biokar Diagnostics, Allonne, Beauvais, France) and incubated for 72 h at 30 °C to examine the total plate count (TPC) (ISO 4833-1, 2013). For *Enterobacteriaceae*, dilutions were poured plated on Violet Red Bile Glucose Agar (Oxoid, Basingstoke, Hampshire, England) and incubated for 24 h at 37 °C (ISO 21528-1, 2017a). Yeasts and moulds were counted on Dichloran 18% glycerol agar (Biokar Diagnostics, Zac de Ther, France) after incubation at 25 °C for 7 days (ISO 21527-2, 2008).

Statistical analysis

Data are expressed as mean ± standard deviation and analyzed by one-way analysis of variance (ANOVA). The significance of mean differences was determined by Tukey's test (Minitab 18). p values lower than 0.05 were considered statistically significant.

Results and discussion

Gluten-free oat biscuits and packaging films

The composition and barrier properties of the films are reported in Table 1

The proximate composition (expressed as % dry matter) of gluten-free oat biscuits before packaging was: protein, 14.2%; fat, 17.5%; ash, 2.3%; sugar, 17.0% and total dietary fibre, 18.2%. The lipid content from the raw oat materials is 7%. Zhou et al. (1999) stated that oat lipids are implicated in the flavour/off-flavour attributes of oat. During storage, the quality of gluten-free oat biscuits could be deteriorated because of the high fat content leading to rancid flavour and taste, caused by oxidation and rancidity of the fat.

Biscuits from the same batch were packed in 5 different films and stored under light and dark conditions. PE/EVOH/PP_50 and PET/CPP_34 are high oxygen-barrier films with low OTR. High values for OTR on BOPP_40, BOPP/CPP_50 and PVC_12 films suggest that oxygen could easily permeate into package leading to high partial pressure of oxygen inside and a higher oxidation rate of cookies during storage (Lu and Xu 2010). PVC_12 has the highest OTR.

The presence of BOPP induced a higher OTR value in the multilayer BOPP/CPP_50 film than PET in PET/CPP_34.

Moisture content and water activity changes

The moisture content of gluten-free oat biscuits in both conditions decreased during storage due to water loss in the headspace of all types of packaging. The decrease in moisture content is up to 21% compared with the initial value (4.2%). Biscuits exhibited a moisture content lower than 4% until 90 days of storage.

The evolution of a_w is in line with the changes in moisture content. Water activity in fresh gluten-free oat biscuits has a value of 0.32 that underwent a slow decrease in all samples. This is probably due to the water loss from the biscuit to the packaging headspace. The decrease in a_w with up to 26% reached after 90 days of biscuit storage is low enough to avoid microbial growth.

Peroxide value evolution

Peroxide value (PV) is an indicator of fat oxidative rancidity or degree of oxidation in food products. The autooxidation of the lipids that are present in biscuits' formulation occurs rapidly when a_w is less than 0.3 (Cauvain and Young 2011). Labuza (1971) reported that the relative rate of lipid oxidation is at a minimum when a_w is in a range between 0.25 and 0.40.

The initial PV for the unpacked gluten-free oat biscuit was determined as 3.1 ± 0.1 meq O_2/kg of lipids. PV of gluten-free oat biscuits measured in different packaging films was compared and data are showed in Table 2. PV tends to increase gradually with the increase of the storage period. A higher PV indicates that more peroxide is produced and the oxidation level is also greater. Storage in dark conditions reduced the PV (rancidity level). Lu and Xu (2009) stated that the speed of fat oxidation is very slow in dark-stored biscuits.

In both storage conditions, the highest PV was obtained for samples in PVC_12 film. For high oxygen-barrier films (low OTR) such as PE/EVOH/PP_50 and PET/CPP_34, the PV of the biscuits increased slowly in contrast with high OTR films. Lu and Xu (2010) reported that for high OTR films, such as BOPP/PE, oxygen permeates easily into the pack, leading to a higher oxidation rate of biscuits during

Table 1 Properties of film packaging used to wrap the trays

Film composition (abbreviation)	Thickness, µm	Oxygen transmission rate (OTR)[a]	Water vapour transmission rate (WVTR)[b]
PET/CPP_34	34	133.8	3.2
BOPP_40	40	1336.5	1.3
PVC_12[c]	12	5000	Impermeable
BOPP/CPP_50	50	1485.5	1.3
PE/EVOH/PP_50	50	9.3	1.5

[a] $cm^3/m^2 \cdot 24$ h·bar, test conditions: 23 °C, 0% RH (according to ISO 2556, 1974)

[b] $g/m^2 \cdot 24$ h, test conditions: 23 °C, 85% RH (according to ISO 15106-1, 2003)

[c] The values for PVC were according to the information from the film supplier

PET/CPP_34: polyethylene terephthalate/casting polypropylene

BOPP_40: biaxially oriented polypropylene

PVC_12: polyvinylchloride

BOPP/CPP_50: biaxially oriented polypropylene/casting polypropylene

PE/EVOH/PP_50: polyethylene/ethylene vinyl alcohol/polypropylene

Table 2 Peroxide value (expressed as meq O_2/kg of lipids) of gluten-free oat biscuits packed in different films and stored in light and dark conditions up to 90 days

Storage		PET/CPP_34	BOPP_40	PVC_12	BOPP/CPP_50	PE/EVOH/PP_50
Period	Condition					
Fresh samples		3.1 ± 0.1				
30 d	L	5.53 ± 0.06[c]	6.03 ± 0.12[b]	7.98 ± 0.24[a]	6.08 ± 0.03[b]	3.73 ± 0.14[d]
60 d		8.17 ± 0.31[c]	9.69 ± 0.10[b]	12.76 ± 0.16[a]	10.18 ± 0.16[b]	7.17 ± 0.15[d]
90 d		14.24 ± 0.20[d]	20.63 ± 0.18[c]	35.39 ± 0.13[a]	24.50 ± 0.26[b]	12.11 ± 0.22[e]
30 d	D	3.80 ± 0.10[c]	4.20 ± 0.10[b]	5.97 ± 0.15[a]	4.32 ± 0.08[b]	3.47 ± 0.12[d]
60 d		5.79 ± 0.25[c]	6.30 ± 0.10[b]	8.27 ± 0.15[a]	6.43 ± 0.21[b]	5.30 ± 0.10[d]
90 d		8.27 ± 0.12[c]	11.33 ± 0.15[b]	13.77 ± 0.25[a]	11.20 ± 0.10[b]	6.12 ± 0.23[d]

The values are expressed as mean ± SD, $n = 3$. Means followed by the same superscript letter in a row are not significantly different ($p > 0.05$) between different samples at the same day of storage

PET/CPP_34: polyethylene terephthalate/casting polypropylene

BOPP_40: biaxially oriented polypropylene

PVC_12: polyvinylchloride

BOPP/CPP_50: biaxially oriented polypropylene/casting polypropylene

PE/EVOH/PP_50: polyethylene/ethylene vinyl alcohol/polypropylene

30 d: day 30; 60 d: day 60; 90 d: day 90

L light, *D* dark

storage. When comparing oat biscuit samples packed in different films at the same day of storage, the PV was significantly different ($p < 0.05$), except for BOPP_40 and BOPP/CPP_50 films. This could be explained by the similar values for OTR and WVTR of both films. Thus, in packed biscuit in BOPP_40 and BOPP/CPP_50 films, there were no significant changes ($p > 0.05$) in PV at the same day of storage, except for 90 d in light conditions where the PV measured in all the biscuits considered was significantly different ($p < 0.05$).

A significant linear correlation ($p < 0.05$) between the logarithm of the average PV and storage time for all packaging films was obtained. Under light conditions, the coefficients of determination (R^2) of the fitted equations were: 0.9935 (PET/CPP_34), 0.9661 (BOPP_40), 0.9821 (PVC_12), 0.9904 (BOPP/CPP_50) and 0.9674 (PE/EVOH/PP_50). The same linear degree of correlation was noticed under dark conditions: 0.9889 (PET/CPP_34), 0.9731 (BOPP_40), 0.9829 (PVC_12), 0.9717 (BOPP/CPP_50) and 0.9233 (PE/EVOH/PP_50), respectively.

Thus, the relation of PV and storage time of gluten-free oat biscuits in all the types of packages studied was linearly dependent.

Colour measurement

During biscuits' storage, fat oxidation takes place and the free radicals formed could react with proteins modifying the product colour (Zamora and Hidalgo 2005).

The change in the colour parameter L* during storage of gluten-free oat biscuits is shown in Table 3.

The initial L* value for the unpacked biscuit was 38.21 ± 0.09 and there were no significant differences between fresh biscuits and those stored for 30 d in both illumination conditions. During storage the colour changed from yellow–brown for biscuits exposed to darkness to light yellow for biscuits exposed to lightness conditions. The longer the storage period of gluten-free oat biscuits, the lightness (L*) increased. As a general trend, gluten-free oat biscuits stored in dark conditions were darker (lower L* values) compared with biscuits stored under light (Table 3).

Biscuits stored in all types of film packagings for 60 d and 90 d in light conditions had significant differences in L* values ($p < 0.05$).

No significant differences ($p > 0.05$) in the colour parameter L* was found between biscuits stored for 30 d in dark conditions; L* values of the biscuits remained unchanged regardless of the type of packaging used.

Gluten-free oat biscuits exposed at both storage conditions and packed in the PVC_12 film with high OTR developed a lighter colour than the others. The darkest biscuit was that stored in a package film with low OTR (PE/EVOH/PP_50).

Considering the time of storage for the same type of package, there was a significant increase ($p < 0.05$) for L*. Thus, the biscuits were lighter as the storage period increased (for both light and dark conditions).

The colour differences (ΔE) between the fresh biscuits and all the biscuits stored in dark conditions (for 30 d, 60 d and 90 d, respectively) was lower than 1 (Table 4). For the samples stored for 90 d under light conditions, ΔE was higher than 1, except for PE/EVOH/PP_50 (ΔE = 0.68). For the biscuits packed in PVC_12, the colour difference was greater than 3, which means that the difference is visible to the human eye (Francis and Clydesdale 1975). Regarding the difference between biscuits stored under light and those stored under dark conditions, ΔE was lower than 1, except for PVC_12 at 60 d (ΔE = 1.5) and 90 d (ΔE = 2.4).

Hardness of biscuits

Hardening has a great importance in the shelf-life of biscuits. The hardness value of fresh biscuits was

Table 3 L* values of gluten-free oat biscuits packed in different films and stored in light and darkness conditions up to 90 days

Storage		PET/CPP_34	BOPP_40	PVC_12	BOPP/CPP_50	PE/EVOH/PP_50
Period	Condition					
Fresh samples		38.21 ± 0.09^{C}	38.21 ± 0.09^{C}	38.21 ± 0.09^{C}	38.21 ± 0.09^{C}	38.21 ± 0.09^{C}
30 d	L	38.31 ± 0.10^{bcC}	38.21 ± 0.08^{dC}	38.45 ± 0.07^{aC}	38.33 ± 0.05^{bC}	38.24 ± 0.02^{cdC}
60 d		38.84 ± 0.05^{cB}	38.74 ± 0.04^{dB}	40.09 ± 0.09^{aB}	39.01 ± 0.07^{bB}	38.45 ± 0.03^{eB}
90 d		39.34 ± 0.14^{dA}	39.65 ± 0.05^{cA}	41.33 ± 0.03^{aA}	39.90 ± 0.05^{bA}	38.72 ± 0.06^{eA}
30 d	D	38.23 ± 0.11^{aC}	38.23 ± 0.09^{aC}	38.29 ± 0.05^{aC}	38.21 ± 0.09^{aC}	38.25 ± 0.03^{aC}
60 d		38.40 ± 0.15^{bcB}	38.43 ± 0.06^{bcB}	38.64 ± 0.07^{aB}	38.47 ± 0.07^{bB}	38.35 ± 0.03^{cB}
90 d		38.88 ± 0.10^{cA}	39.02 ± 0.11^{bA}	39.13 ± 0.05^{aA}	38.85 ± 0.03^{cA}	38.42 ± 0.05^{dA}

The values are expressed as mean ± SD, $n = 10$

a–e: Values followed by the same superscript letter in a row are not significantly different ($p > 0.05$) between different samples at the same day of storage

A–C: Values followed by different superscript letter within columns for the same condition storage are significantly different ($p < 0.05$)

PET/CPP_34: polyethylene terephthalate/casting polypropylene

BOPP_40: biaxially oriented polypropylene

PVC_12: polyvinylchloride

BOPP/CPP_50: biaxially oriented polypropylene/casting polypropylene

PE/EVOH/PP_50: polyethylene/ethylene vinyl alcohol/polypropylene

30 d: day 30; 60 d: day 60; 90 d: day 90

L light, *D* dark

Table 4 Colour differences (ΔE) values calculated between gluten-free oat biscuits' samples

Storage Period	Condition	PET/CPP_34	BOPP_40	PVC_12	BOPP/CPP_50	PE/EVOH/PP_50
ΔE calculated between fresh and stored biscuits						
30 d	L	0.63	0.34	0.33	0.54	0.38
60 d		0.68	0.60	1.89	0.88	0.51
90 d		1.16	1.47	3.24	1.7	0.68
30 d	D	0.23	0.22	0.57	0.09	0.19
60 d		0.64	0.28	0.51	0.33	0.20
90 d		0.72	0.82	0.92	0.65	0.92
ΔE calculated between biscuits stored under light and dark conditions						
30 d		0.54	0.33	0.41	0.63	0.32
60 d		0.88	0.34	1.50	0.79	0.49
90 d		0.64	0.66	2.39	0.90	0.56

PET/CPP_34: polyethylene terephthalate/casting polypropylene

BOPP_40: biaxially oriented polypropylene

PVC_12: polyvinylchloride

BOPP/CPP_50: biaxially oriented polypropylene/casting polypropylene

PE/EVOH/PP_50: polyethylene/ethylene vinyl alcohol/polypropylene

30 d: day 30; 60 d: day 60; 90 d: day 90

20.3 ± 5.8 N (n = 8). For all packaging materials considered in this study, the hardness of gluten-free oat biscuits decreased ($p > 0.05$) more in the dark as compared to light conditions (Table 5). However, the decrease was not statistically significant. Unlike the results from this study, Piga et al. (2005) observed that cookies wrapped in aluminium foil significantly hardened to a lesser extent than PVC ones because of the occurrence of the water migration from the surface to the interior of the cookie.

Packed biscuits stored under light conditions, up to 60 d, showed similar values for hardness as that of fresh ones. After 90 d of storage under light conditions, the hardness significantly decreased ($p < 0.05$) for biscuits packed in BOPP_40 and BOPP/CPP_50. For the others packages, the decrease of hardness was not significatly different

Table 5 Hardness of gluten-free oat biscuits packed in different films and stored in light and dark conditions up to 90 days

Storage Period	Condition	PET/CPP_34	BOPP_40	PVC_12	BOPP/CPP_50	PE/EVOH/PP_50
Fresh samples		20.3 ± 5.8				
30 d	L	20.9 ± 4.5^{a}	21.6 ± 4.2^{a}	20.3 ± 5.5^{a}	22.6 ± 3.9^{a}	23.4 ± 7.2^{a}
60 d		17.4 ± 8.1^{abc}	16.5 ± 8.3^{ac}	13.5 ± 4.5^{ab}	19.0 ± 7.6^{ac}	16.6 ± 5.8^{ab}
90 d		19.6 ± 3.3^{ac}	11.1 ± 2.8^{bc}	14.2 ± 2.7^{ab}	12.0 ± 1.9^{b}	16.3 ± 3.5^{ab}
30 d	D	15.1 ± 3.2^{ae}	20.7 ± 3.6^{a}	17.3 ± 3.1^{ab}	20.2 ± 5.5^{ac}	20.6 ± 5.0^{ab}
60 d		12.5 ± 3.6^{be}	17.5 ± 2.7^{abe}	14.4 ± 5.2^{ab}	15.4 ± 2.1^{bc}	15.9 ± 4.7^{ab}
90 d		13.7 ± 3.9^{ce}	11.9 ± 2.9^{ce}	12.2 ± 6.8^{b}	13.2 ± 4.4^{bc}	15.3 ± 2.5^{b}

The values are expressed as mean ± SD, n = 8

A–e: Values followed by the same superscript letter in a column are not significantly different ($p > 0.05$) between different storage time and conditions

PET/CPP_34: polyethylene terephthalate/casting polypropylene

BOPP_40: biaxially oriented polypropylene

PVC_12: polyvinylchloride

BOPP/CPP_50: biaxially oriented polypropylene/casting polypropylene

PE/EVOH/PP_50: polyethylene/ethylene vinyl alcohol/polypropylene

30 d: day 30; 60 d: day 60; 90 d: day 90

L light, *D* dark

($p > 0.05$) between the storage period. The biscuits packed in BOPP_40 were the softest after 90 d under darkness conditions. However, as a general trend, as the storage period increased, the hardness value decreased ($p > 0.05$).

Discrimination of oat biscuits' samples by electronic nose

Electronic noses found a very good applicability in food control, food safety and food shelf-life investigations in different storage conditions (temperature, humidity, lighting, packages) (Peris and Escuder-Gilabert 2009). Different types of electronic noses were used previously to estimate the rancidity and assessment of shelf-life of cookies formulated with eugenol-rich clove extracts or to compare the flavour qualities of mushrooms packed with different packaging materials at 6 days, 12 days and 21 days of storage time (Donglu et al. 2017).

In this study, the electronic nose system was used to study the evolution of the odour profile for the same packed product, stored in two illumination conditions during 90 d. Thus, for each type of packaging (PET/CPP_34, BOPP_40, PVC_12, BOPP/CPP_50 and PE/EVOH/PP_50), the odour of fresh (day 0) and stored (30 d, 60 d and 90 d for both conditions) gluten-free oat biscuits were considered (Fig. 1). Biscuits samples were well distinguished with high discrimination index: 91 (PET/CPP_34), 94 (BOPP_40), 88 (BOPP/CPP_50) and 86 (PE/EVOH/PP_50).

For PET/CPP_34 and BOPP_40, the samples stored for 90 d under light conditions were positioned on the bottom left of the plot far distant to the others, suggesting the distinct odour profile. This is correlated with the sensory evaluation done by panelists who evaluated these samples with higher scores for off-flavour (3.1–4.3) and for the peroxide value also. On the other side, samples stored under dark were grouped which means that the changes in the odour profile of the products packed in PET/CPP_34 and BOPP_40 and maintained in darkness were not so significative in time. This is correlated with the scores of the panelists in the sensory evaluation also.

For samples packed in PVC_12, PCA analysis showed that biscuits stored for 30 d under light and dark conditions were overlapped showing that these samples were similar in the volatile composition, with no differentiation.

For BOPP/CPP_50, a very good discrimination was observed for the samples stored in darkness which were placed on the upper part of PCA graph.

In the case of PE/EVOH/PP_50, samples stored in the darkness for 30 d and 60 d had a close odour profile with the fresh samples while the other samples were placed in the opposite side of the PCA plot.

Sensorial evaluation

The effect of the package properties, especially the oxygen transmission rate, and the light exposure on different sensory attributes is showed in Fig. 2. The sensory quality of gluten-free oat biscuits was highly influenced by the package and the light.

The surface colour of biscuits decreased in time from a nice light brown to a paler colour. This is one of the physical changes in biscuits' deterioration during storage which was previously mentioned in the literature and it is explained by the fat blooming phenomena: fat from biscuit migrates on the outer layers and it crystallizes on the surface forming a white layer which determines the lightening of biscuits' colour (Manley 2011; Swapna and Jayaraj Rao 2016). The changes in the colour were more pronounced under the light exposure and, especially, for PVC_12, the package with the highest OTR. In dark conditions, the colour changes during biscuits' storage did not significantly differ ($p > 0.05$) from the control (Fig. 2). However, the values of the surface colour attribute were smaller. Package BOPP/CPP_50, with high OTR and high thickness (50 μm), had the most positive effect on maintaining the bright brown colour of the products in both conditions: light and dark, followed by PE/EVOH/PP_50 (the package with the lowest OTR), BOPP_40 and PVC_12. The decrease in the colour intensity varied between samples and it was not uniform during storage. Comparing with the fresh biscuit, the colour of biscuits stored in the light conditions became lighter with 11.63–18.60% after 30 d, with 16.28–37.21% after 60 d and with 18.60–55.81% after 90 d. In dark conditions, the decrease of the colour scores was up to 13.95% after 90 d of storage.

Comparing all the types of package used, the biscuits stored at 30 d, 60 d and 90 d, under dark conditions and 30 d under light exposure did not significantly affect ($p > 0.05$) the surface colour of the biscuit compared with the control. On the other side, storage in light conditions for 60 d and 90 d produced a significant decrease ($p < 0.05$) in the surface colour attribute. However, BOPP/CPP_50 did not significantly differ ($p > 0.05$) from the control at 60 d and 90 d, indicating that BOPP/CPP_50 could preserve the colour of biscuits even under light conditions for a longer time.

Flavour migration during storage may determine a dilution of the smell and taste sensation (Manley 2011).

In terms of protecting the smell, the samples packed in PE/EVOH/PP_50 and BOPP/CPP_50 were scored with the most pleasant smell after 90 d. The initial score for smell was marked by the panelists to 4.9 and it decreased with 30.61–38.77% after 30 d, less in the case of samples PE/EVOH/PP_50 and BOPP/CPP_50, under light conditions. In dark, the smell was maintained better. However, biscuits

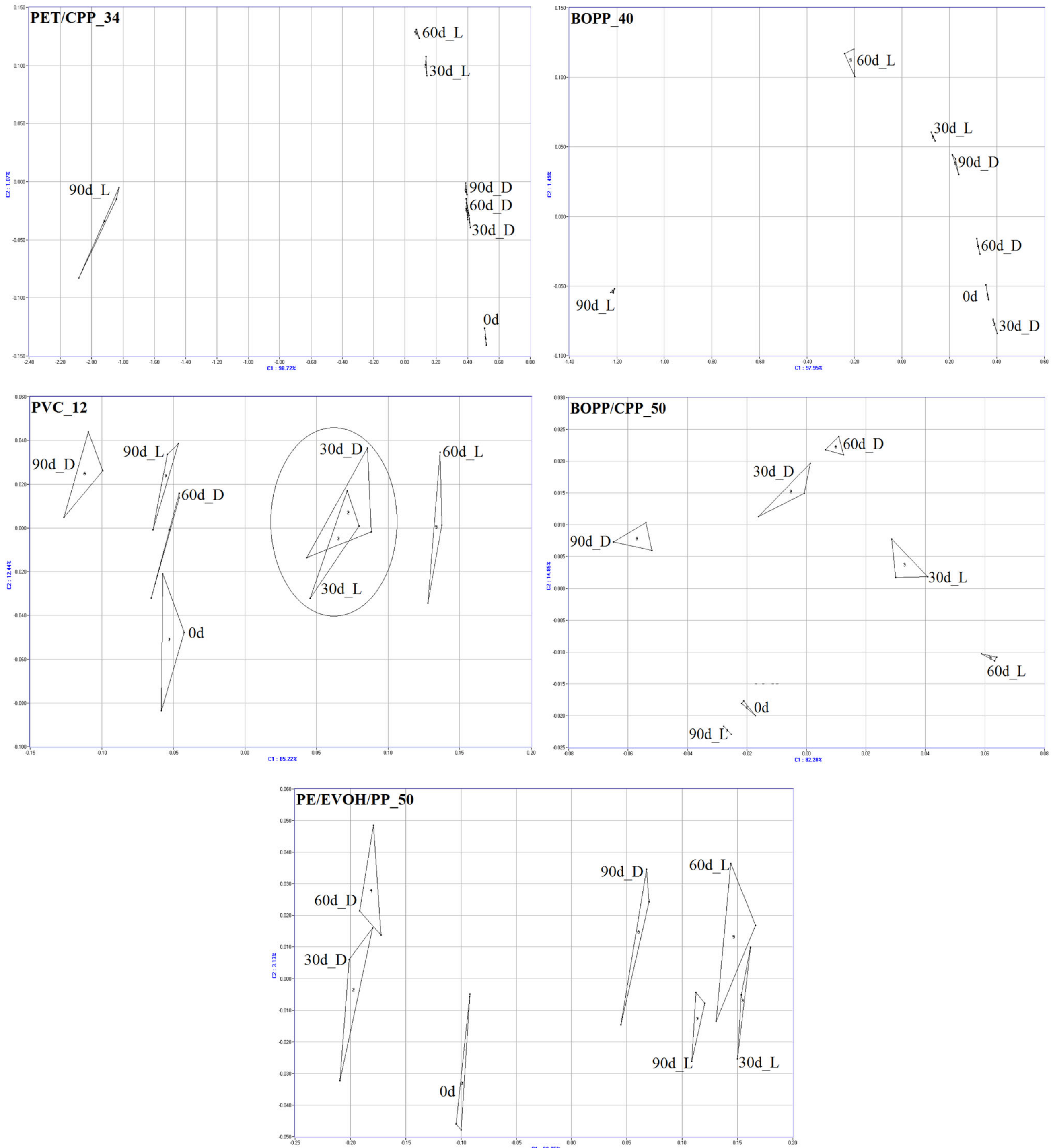

Fig. 1 Discrimination of gluten-free oat biscuits' samples at day 0 (0 d) and during storage: 30 d, 60 d and 90 d under light (L) and dark (D) conditions for each type of packaging based on PCA. Legend: PET/CPP_34: polyethylene terephthalate/casting polypropylene, BOPP_40: biaxially oriented polypropylene, PVC_12: polyvinylchloride, BOPP/CPP_50: biaxially oriented polypropylene/casting polypropylene, PE/EVOH/PP_50: polyethylene/ethylene vinyl alcohol/polypropylene, 0 d: day 0 (fresh biscuits); 30 d: day 30; 60 d: day 60; 90 d: day 90; L: light; D: dark

stored in dark conditions in BOPP/CPP_50 and PE/EVOH/PP_50 did not produce a significant decrease ($p > 0.05$) in the score of the smell attribute (Fig. 2), suggesting that in the case of BOPP/CPP_50 and PE/EVOH/PP_50, the smell was close to the control.

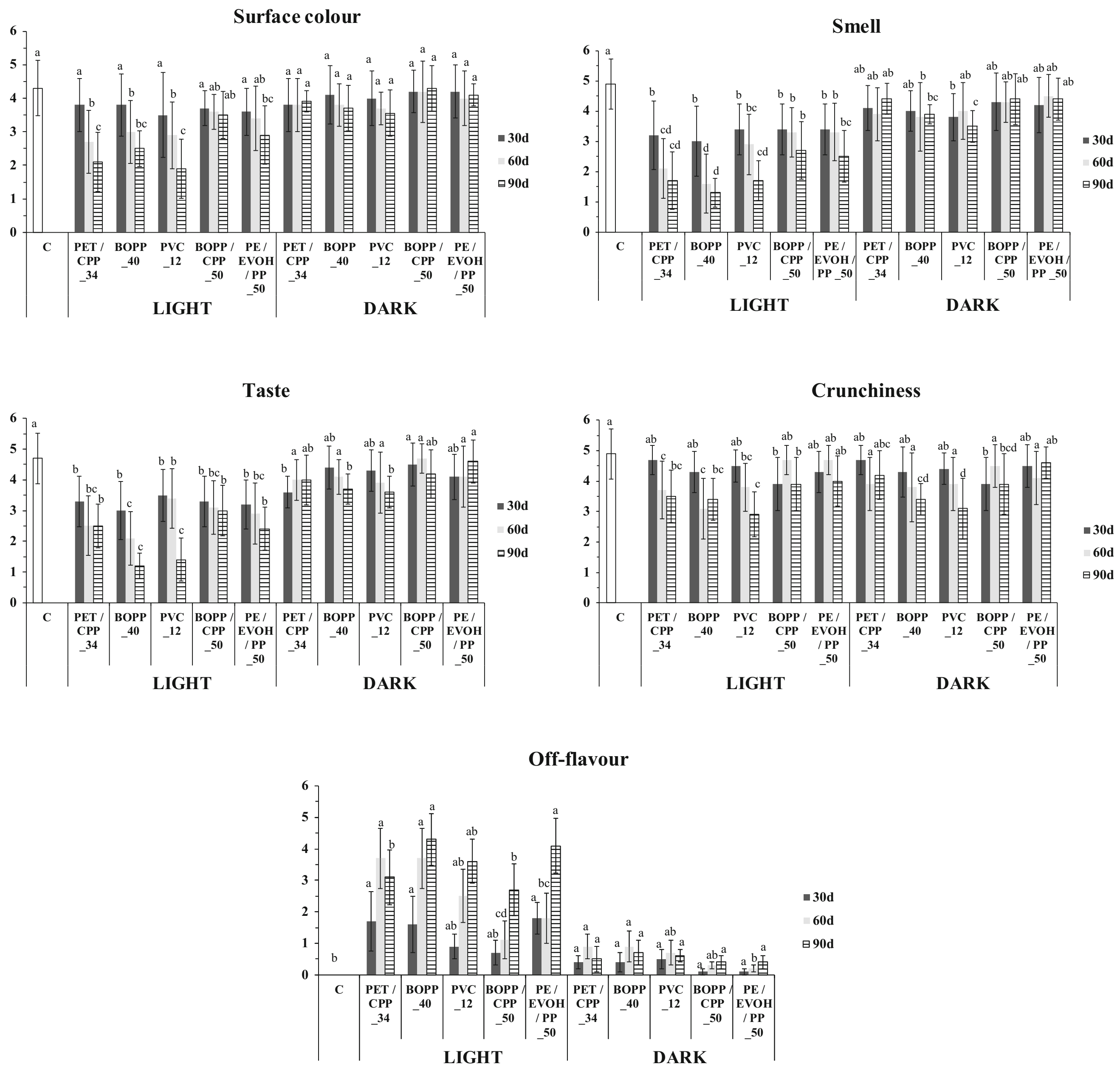

Fig. 2 Scores for surface colour, smell, taste, crunchiness and off-flavour for gluten-free oat biscuits stored at room temperature for 30 d, 60 d and 90 d under light and dark conditions in different packages with different oxygen transmission rates (OTR). Values labeled with a different lowercase letter are significantly different ($p < 0.05$) within samples (calculated between the control and the group of samples packed in the same type of package after 30 d, 60 d and 90 d). Legend: PET/CPP_34: polyethylene terephthalate/casting polypropylene, BOPP_40: biaxially oriented polypropylene, PVC_12: polyvinylchloride, BOPP/CPP_50: biaxially oriented polypropylene/casting polypropylene, PE/EVOH/PP_50: polyethylene/ethylene vinyl alcohol/polypropylene, 30 d: day 30; 60 d: day 60; 90 d: day 90

But, under light exposure, the score attributes for smell were significantly lower ($p < 0.05$) with respect to the control, for all the types of package at the same point investigated (30 d, 60 d and 90 d, respectively). This may be due to the fast changes in the smell that took place under light exposure.

In terms of taste, in dark conditions the samples were better maintained. Even after 90 d, the scores were more than 3.60 on a scale from 1 to 5. Under the light storage, after 30 d the taste decreased 25.53–36.17%. After 60 d, the taste was still appreciated as acceptable, with lowest scores for BOPP_40 (2.1). BOPP/CPP_50 had the best effect on maintaining a pleasant taste under light conditions, while under dark, BOPP/CPP_50 and PE/EVOH/PP_50 were the best. After 90 d of storage under dark conditions, there was no statistical difference ($p > 0.05$)

between PET/CPP_34, BOPP/CPP_50 and PE/EVOH/PP_50 in terms of taste as compared with the control. On the other hand, under light exposure biscuits packed in PET/CPP_34, BOPP/CPP_50 and PE/EVOH/PP_50 were scored lower than the control, but higher than the other types of package investigated.

The crunchiness evaluated by the sensory panel was well correlated with the hardness values instrumentally measured: both decreased during storage. Loss of crunchiness is the most likely sensory attribute perceived by consumers and one of the main aspects of shelf life (Manley 2011).

Crunchiness significantly decreased ($p < 0.05$) during the storage up to 90 d with 18.36–20.40% under light for samples BOPP/CPP_50 and PE/EVOH/PP_50 and to 40.81% for PVC_12 compared to fresh biscuits. Under dark, the crunchiness was better maintained. However, crunchiness of the biscuits packed in PVC_12 and BOPP/CPP_50, and stored in dark was similar to that of the biscuits stored under light conditions, for the different storage period considered. Comparing all the packages and regardless the type of storage conditions, after 90 d of storage, PE/EVOH/PP_50 did not significantly differ ($p > 0.05$) from the control, suggesting that PE/EVOH/PP_50 managed to keep the initial crunchiness of the biscuit. Swapna and Jayaraj Rao (2016) also found a decrease in the textural characteristics of the biscuits during storage.

The development of off-flavour was significantly influenced ($p < 0.05$) by the storage conditions. The samples stored under light conditions were scored with higher off-flavour starting at 30 d. BOPP/CPP_50 and PE/EVOH/PP_50 had a good effect in keeping a good flavour of the products under light, with scores acceptable until 60 d. All the samples presented high off-flavour scores after 90 d of storage under light. Interestingly, even if BOPP/CPP_50 caused an increase in the off-flavour attribute, this increase was not significantly different ($p > 0.05$) from the control.

Under dark exposure, the off-flavours were mostly unnoticed after 30 d. Samples PET/CPP_34 and BOPP_40 presented a higher unpleasant taste after 60 d of storage in the dark. All the samples had a modified taste after 90 d in the dark, but the scores for off-flavour were below 1. The results are in accordance with the study of Larsen et al. (2005). They showed that the intensity of the rancid odors and flavours is delayed in the extruded oat stored in darkness as compared to light.

Microbiological analysis

Microbiological analysis was performed during the biscuits' storage up to 90 d. Packaging materials and storage conditions up to 60 d had no significant effect on TPC. After 60 d, the count increased in the biscuits stored in PET/CPP_34 ($1.8 \cdot 10^2$ CFU/g), BOPP_40 ($1.5 \cdot 10^2$ CFU/g) and PVC_12 ($2.7 \cdot 10^2$ CFU/g) in comparison to BOPP/CPP_50 and PE/EVOH/PP_50 with TPC below 10 CFU/g. However, the counts were higher in the biscuits sample stored in light than darkness conditions for the same type of packaging. Jan et al. (2017) also showed the significant effect of packaging material and storage period on the growth of microorganisms of biscuits. *Enterobacteriaceae* and yeasts' and moulds' counts were below 10 CFU/g in any sample studied during the storage period. The values obtained for the microbiological stability are in line with the a_w parameters. The lower a_w values prevented the microbial growth. Even if the oat biscuits are microbiologically stable, their shelf-life is limited by changes in sensory characteristics.

Conclusion

This study showed that the physicochemical parameters and sensory attributes of gluten-free oat biscuits were influenced by the storage conditions (light and dark) and type of packaging material used. Moisture and water activity of oat biscuits decreased during storage in all packages. In both storage conditions, the highest peroxide value was obtained for biscuits packed in PVC_12, while the lowest was for the PE/EVOH/PP_50. The change in biscuits' colour from yellow–brown to light yellow was more pronounced in the light as compared to the dark storage conditions. Regarding the texture of the biscuits, the hardness value decreased as the storage period increased; the effect was more obvious in the dark as compared to light conditions. The e-nose system through PCA analysis, showed that the distinct volatile composition of the biscuits stored in the light was correlated with the higher scores of the off-flavour attribute and with the peroxide values. The sensory scores revealed that BOPP/CPP_50 preserved the colour of the biscuits, while PE/EVOH/PP_50 kept the initial crunchiness of the biscuits up to 90 d of storage in both the light and the dark conditions. Under dark up to 90 d, the smell and taste attributes for the biscuits packed in BOPP/CPP_50 and PE/EVOH/PP_50 were not significantly different compared with control. Moreover, under dark conditions, the off-flavour was lower scored. Overall, storage in dark gave more acceptable sensory attributes than light exposure.

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