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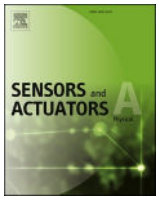

Paper-based metal-air battery electrochemical sensor for smartphone-assisted oxygen monitoring in food packaging

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ABSTRACT

The monitoring of oxygen in food packaging during storage and transportation is crucial in food quality surveillance, warning users regarding food spoiling, happening through compound oxidation and aerobic microorganism proliferation. In this overall scenario, we report the development of a flexible, cost-effective, and Bluetooth-assisted electrochemical sensor for oxygen detection in food packaging. The device encompasses three layers, namely a zinc sheet as an anode, a conductive silver ink printed on an oriented polypropylene sheet serving as a cathode, and a deep eutectic solvent deposited on a paper-based substrate sandwiched between both electrodes. The sensing tool provided a wide linear range for oxygen detection up to 20.9 O_2% v/v with good intra-electrode repeatability (RSD % = 0.02 %). Finally, the developed device was integrated with a 3D printed holder and tested for oxygen detection in packages containing mushrooms, tomatoes, and broccoli samples, obtaining a good correlation with the reference method. This study opens noticeable possibilities for employing paper-based metal-air batteries in the detection of specific target analytes, by integrating paper substrate and metal-based batteries delivering smart and self-powered instruments as reliable and accurate analytical tools.

1. Introduction

Food packaging is a common practice to maintain nutrient quality over time, preventing the degradation induced by environmental factors such as microorganism presence, prolonged light exposure, and high-temperature storage. The resulting gaseous emission and the presence of moisture can easily compromise the quality of the nutrients [1,2]. To overcome these drawbacks, gases are usually used as preserving agents in modified atmosphere packaging, including carbon dioxide, nitrogen, and oxygen, used singly or combined to balance safe shelf-life extension [3].

Recently, advanced technologies have boosted the fabrication of smart packaging using biodegradable materials that can release functional or bioactive components. Additionally, the embedding of sensors has a crucial function for assessing the quality of the product directly within the package [4,5]. By communicating real-time information about food quality, the information related to the integrity of food during storage and transportation is monitored in a timely manner [6]. Research activities have recently started for the development of analytical tools to address this requirement with successive industrialization. For instance, Ozbek designed a potentiometric sensor to detect potassium ions in fruits-containing packages [7], while Magnaghi et al. produced a smart dye-based device to assess poultry meat spoilage identified with naked eyes [8], and a bromocresol purple/ethylene-vinyl alcohol copolymer optode for milk freshness measurement [9].

Among the analytes useful for assessing food safety and quality, oxygen can be a crucial marker in providing this information to producers and customers. Its presence could lead to food spoiling through the oxidation of ingredients and the proliferation of aerobic microorganisms, resulting in rapid ripening and browning. The conservation is generally enhanced when oxygen concentration is reduced, affecting microbial metabolism and growth capacity. On the other hand, depending on the food, high oxygen concentration can offer certain benefits, mainly organoleptic, such as maintaining the colour of red

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meat [10]. Up to now, spectroscopic techniques have been mainly used for oxygen monitoring in food packages [11–13]. Additionally, these techniques require laboratory-based equipment and skilled personnel, avoiding the application at the point of need, i.e., inside the packaging. To overcome these challenges, current research trends are focusing on the development of miniaturized, easy-to-use, and sustainable devices, with the overriding goal of providing rapid-response, user-friendly analytical tools, and cost-effective measures to perform in situ oxygen monitoring, with easily accessible data acquisition [14,15]

As booming analytical platforms, electrochemical sensors fulfil these requests, as they are characterized by a quantitative response, cost-effective fabrication, miniaturized dimensions, and reliable measurements [16–18]. These features enable the production of smart food packaging with analytical devices for in-situ and user-friendly oxygen monitoring.

The application of metal-air battery-based electrochemical oxygen sensors for food monitoring was first reported by Won et al. in 2021 [19]. The authors designed a miniaturized potentiometric sensor for oxygen detection in food packaging that exploited oxygen molecules from the air to oxidize metals localized in the zinc anode. In detail, the whole device is based on three commercially available components, namely a cathode and an anode separated by an adhesive electrolyte, enabling the electrochemical process. This sensor produced an output potential that was proportional to the oxygen gas concentration within the range of 0–21 % (v/v) with a good linearity ($R^2 = 0.999$) and a sensitivity equal to 18 mV/O_2.

Despite the significant evolution in food packaging, there are still key challenges to be addressed in delivering reliable sensors for oxygen monitoring, characterized by i) portability and in-situ detection, and ii) the use of biodegradable and sustainable materials, to finally achieve safe and cost-effective daily monitoring of food during storage and transportation.

In this context, the introduction of paper-based sustainable platforms represents a promising approach in the field of flexible, environmentally friendly, and cost-effective metal-air battery-based devices, thanks to the features of the paper, namely abundance, lightweight, flexibility, thinness, cost-effectiveness, biocompatibility, and biodegradability. Up to now, several types of paper-based energy storage systems have been produced for various applications, including paper-based laser induced graphene for the fabrication of sustainable high-energy density supercapacitors [20], flexible ceramic-based paper separators for high-performance lithium-ion batteries [21], and paper-based hybrid battery for powering flexible electronics devices [22].

An additional key application of paper is its use as a promising analytical sensing platform for developing electrochemical sensors and biosensors. To this stage, several studies developed by our group demonstrated the exploitation of paper and its application as a portable and sustainable analytical sensing platform for the detection of target analytes in solution e.g. glucose in tears [23], on the surface e.g. chloride on the concrete [22], and in the aerosol phase e.g. SARS-CoV-2 [24] in breath, demonstrating its versatility [25–27].

As a specific application for food freshness monitoring, Naik et al. [28] exploited paper as a substrate for screen-printing interdigitated electrodes, delivering a paper-based electric gas sensor (PEGS) for real-time monitoring of spinach freshness in food packaging. By assessing the conductance change in response to gas presence (ammonia in controlled situation, and multiple gas in real samples as a total response) the PEGS was used to evaluate the freshness of spinaches, by the integration with a NFC-powered device, and encapsulation in polyurethane-based tattoo film.

To our knowledge, the only work that proposed a paper-based battery for sensing applications was reported by Sabate group [29]. In detail, they developed a battery based on magnesium, serving as an anode and silver/silver chloride electrode, serving as a cathode, placed side-by-side and covered by a piece of hydrophilic paper strip, for the measurement of ionic conductivity in liquid samples such as milk, juice, artificial eccrine perspiration, urine, lake water, and phosphate butter.

Herein, in this work, we explore for the first time the combination of a paper substrate with a metal-air battery as an electrochemical sensor for the detection of a specific analyte, namely oxygen.

Specifically, we developed an oxygen sensor based on a battery, leveraging the oxygen reaction with a zinc anode and a silver-ink cathode. The so-conceived battery uses a synthesized green electrolyte and office paper layer, where the electrolyte is loaded. In detail, the miniaturized electrochemical sensor (2 cm×1 cm) is based on a metal anode made of zinc and a cathode made of silver ink loaded on an oxygen-permeable polypropylene substrate. Additionally, a custom-synthesized electrolyte based on deep eutectic solvent (DES), polyvinyl alcohol, and potassium hydroxide was loaded on office paper support and sandwiched between the anode and the cathode, ensuring closure of the circuit and delivering a cost-effective and sustainable detection system, by replacing the use of toxic liquid electrolytes.

Besides acting as an efficient DES electrolyte support, ensuring uniform electrolyte distribution, paper was used as a separator to prevent short-circuiting between the zinc and silver OPP electrodes, which are highly conductive materials.

The electrochemical sensors were morphologically and electrochemically characterized by using scanning electron microscopy (SEM) and electrochemical impedance spectroscopy (EIS)/open circuit potentiometry (OCP), respectively.

After the sensor was optimized, its suitability for analytical applications was demonstrated by testing the concentration of oxygen in food packaging containing mushrooms, broccoli, and tomatoes. The sensor was integrated with a 3D printed holder and connected with a portable and miniaturized potentiostat, exploiting Bluetooth technology for data transmission to an external smartphone. The results were compared with the reference method, namely a commercial oxygen gas analyzer, demonstrating the accuracy of the metal-air battery-based sensor for oxygen monitoring in food packaging. This work highlighted the suitability of a flexible paper substrate for metal-air batteries, creating a self-sustained analytical platform that effectively operates as an oxygen sensor able to continuously monitor oxygen levels in food packaging during both storage and transportation.

2. Materials and methods

2.1. Reagents and equipment

Polyvinyl alcohol (PVA), lithium perchlorate ($LiClO_4$), and urea were purchased from Sigma Aldrich, while potassium hydroxide (KOH) from Fluka. Silver ink (Electrodag 477 SS) from Acheson (Milan, Italy), oriented polypropylene (OPP), and pure Zinc (Zn) sheets were bought from Thermo Fisher Scientific.

Open circuit potentiometry (OCP) was conducted using a miniaturized and portable potentiostat (EmStatBlue) instrument (PalmSens, Netherlands) connected to a smartphone via Bluetooth using PStouch application developed by PalmSens.

SEM micrographs of zinc, silver ink, and oriented polypropylene were characterized with field emission scanning electron microscopy (FEG-SEM, Leo Supra 35, UK). A 3D Printer (Cetus) MK2 (Printing temperature: 250 °C. temperature of the plate: 20 °C. Extruder diameter: 0.04 cm. Layer thickness: 0.02 cm) was used to print the support structure based on Polylactic acid (PLA).

Mushrooms, broccoli, tomatoes, and polypropylene bags (23 cm×32 cm) were purchased from a local shop.

2.2. Fabrication of the metal-air battery-based electrochemical sensor

The flexible electrochemical sensor comprises three components (Fig. 3A), namely an anode based on zinc, a cathode based on silver ink printed via screen-printing on oriented polypropylene (1 ×2 cm) substrate, and an office paper substrate (Fabriano 80 g/m^2) loaded with gel

electrolyte.

2.2.1. Electrodes fabrication

For the fabrication of the cathode, the zinc sheet was cleaned with acetone to remove any dust on the surface and cut to the final size of 1 cm× 2 cm. For the fabrication of the anode electrode, silver ink was screen-printed onto oriented polypropylene using a 245 DEK (Weymouth, UK) screen-printer. After, the ink was dried in oven at 70 °C for 20 min. Finally, the OPP was cut to the final size of 1 cm× 2 cm.

2.2.2. Electrolyte preparation and deposition onto paper

The DES was prepared by mixing urea and lithium perchlorate in a molar ratio of 4.1:1 [30], and stirring until a clear solution was obtained. The PVA-KOH gel was prepared by mixing 4 g of PVA and 2.4 g of KOH in 40 mL of distilled water at 90 °C and stirring until a clear solution was obtained (Fig. S1). Subsequently, the DES-KOH-PVA gel electrolyte was obtained by merging PVA-KOH (3g) and DES (3g) and stirring for several hours, until obtaining a homogeneous solution. To remove the formation of oxygen bubbles, the electrolyte solution was sonicated in a bath at 40 °C for 30 min [31]. Finally, the electrolyte solution was dropped onto a glass petri dish to obtain the final gel electrolyte, by a heating process at 110 °C. After that, the gel electrolyte was loaded onto A4 office paper by immersing the paper into the gel for 5 s.

Finally, the paper-loaded electrolyte was sandwiched between both electrodes (Zn and Ag-OPP) and pressed all together, giving rise to the sensor. Therefore, the device was kept in the fume hood overnight for drying, as depicted in Fig. 1A.

2.3. Setup and electrochemical sensing platform

The operational characteristic of the developed sensor for oxygen detection was evaluated by measuring the open circuit potential signals as a function of oxygen concentration. For this, the sensor was inserted into an airtight container (Fig. 1), which remained consistently supplied with nitrogen to reach the final oxygen concentration of 0 %, 5 %, 10 %, 13 %, 16 %, and 20.9 % v/v. Nitrogen was supplied into the airtight container using a nitrogen gas system (Rivoira, Milan-Italy). The response of the sensor was recorded using a portable potentiostat EmStat Blue (PalmSens, The Nederland, https://www.palmsens.com/product/emstat-blue/), inserted in the airtight container, and connected to the sensor by two crocodile wires, one to the zinc sheet and one to the OPP sheet, respectively.

Additionally, the miniaturized EmStat Blue potentiostat was wireless connected via Bluetooth to a smartphone, placed outside the airtight container. The smartphone is equipped with the PSTouch application, developed by PalmSens company, enabling communication with the potentiostat and data retrieval. Using the app, the end-user can i) start and stop the measurements performed by the EmStat Blue potentiostat inside the airtight container, ii) visualize the recorded data, export, and save the analytical output.

During the measurements, the concentration of oxygen was confirmed with a commercial oxygen analyzer (Goyojo, 133 ×67 x

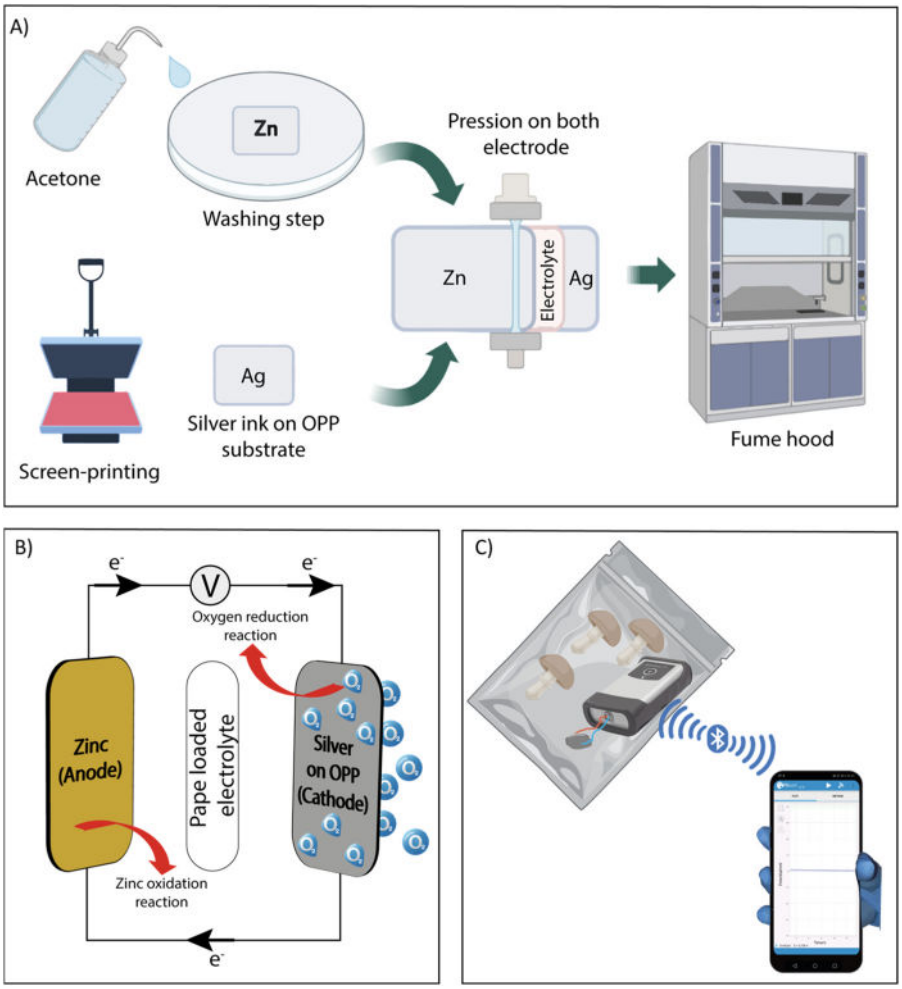

Fig. 1. A) Metal-air battery electrochemical sensor fabrication, B) Working mechanism of the metal-air battery electrochemical sensor, C) Electrochemical sensing platform for oxygen monitoring in food packaging.

30 mm, linear range [O_2] 0–20.9 % v/v, with a response time in less than 30 s). All the measurements were conducted at room temperature.

2.4. 3D printed holder

To hold the developed sensor for the real sample analysis, a plastic holder was in-house fabricated using the 3D Printer (Cetus-MK2). The Computer Aided Design (CAD) model was designed with Autodesk Fusion 360, and the final hoder was fabri. The dimensions of the final holder were tailored to achieve an exact match with the device, ensuring a customized fit as shown in the Fig.S2.

2.5. Real sample measurements

Mushrooms, broccoli, and tomatoes of marketable quality and similar size (weighted 300 g ± 14 g) without any treatment were packed into transparent food bags. The developed sensor was inserted into the 3D printed holder, and toghether with the miniaturized potentiostat and the commercial oxygen analyzer, they were fixed into the bag. The oxygen content inside the bag was measured by means of open-circuit potentiometry, and the data were transferred via Bluetooth to a smartphone equipped with PStouch App.

3. Results and discussion

3.1. Operational mechanism

The working configuration of the developed device relies on the concept of rechargeable metal-air batteries (RMAB), comprising a zinc anode, a silver OPP cathode, and a gel electrolyte. Zinc is usually employed in RMAB for energy conversion and storage device fabrication [32]. Indeed, compared to other metals such as lithium, magnesium, and aluminum, zinc shows relevant features, including highly efficient charging in aqueous electrolytes, low cost, and low toxicity [33].

Oriented polypropylene was selected as the substrate for the silver screen printing, thanks to its oxygen permeability ($2.6 \sim 6.0 \times 10^{-12}$ $cm^3 \cdot m/m^2 \cdot s \cdot Pa$) [34] and its wide use in food packaging.

Indeed, as a gas diffusion layer, the OPP enables the oxygen to diffuse from the air to the device, where it is reduced at the cathode. Here, silver acts as an effective catalyst for oxygen reduction [35], and it is also suitable in food packaging frameworks thanks to its non-toxicity and antimicrobial activity [36].

Finally, the gel electrolyte was used as an essential element for closing the circuit and achieving the mobility of anions and cations between both electrodes. Notably, the fabricated gel electrolyte features a large operating potential window, low viscosity to access small pores, good chemical and thermal stability, and high sustainability [37].

In detail, the in-lab-produced gel electrolyte used in this work relies on a deep eutectic solvent merged with potassium hydroxide mixed in a polyvinyl alcohol matrix.

While the deep eutectic solvent is recognized as an environmentally friendly solvent, characterized by several features, including good ionic conductivity, thermal/chemical stability, and non-flammability properties [38], the use of potassium hydroxide and a polyvinyl alcohol matrix (as a plasticizer) serve to boost the ionic conductivity and to enhance the flexibility of the developed sensor, respectively.

The fabricated electrolyte was then loaded on office paper. In this context, using paper is essential to obtain an optimal electrolyte loading without any leakage, avoid the potential short circuit of the system, in the presence of cathode-anode contact, and deliver a flexible sensing platform able to hold on various angles deformation in food packaging applications. Furthermore, using paper as electrolyte support coupled with non-toxic electrolytes allows for a sustainable and food-compatible sensor.

As mentioned above, the proposed device is based on metal-air batteries that produce the signal by oxidizing metals with oxygen from the air (Fig. 1B, and Fig. 1C). Specifically, pure zinc at the anode is oxidized to metal ions by releasing electrons. These electrons are then accepted by oxygen, which diffuses through a gas diffusion layer from the air at the cathode (silver electrode), and no pollution is produced during either the charging or discharging processes [39].

The electrochemical reactions occurring in zinc-air batteries using neutral and acidic electrolytes can be represented by the following equations:

Zn anode: $Zn \rightarrow Zn^{2+} + 2e^-$

Air cathode: $½\ O_2 + 2H^+ + 2e^- \rightarrow H_2O$

3.2. Morphological characterization

A morphological investigation of the sensor electrode components is reported in Fig. 2, while Fig. S3 shows the relative elemental analysis. The surface of the single layers was observed at different magnifications. The oriented polypropylene in Fig. 2A shows a smooth even morphology. After silver ink printing and drying steps, the polymer surface was covered with interconnected silver metal flakes, observed in detail at higher magnification in Fig. 2B. The silver ink forms a well-distributed and even conductive layer, well adhered to the oriented polypropylene substrate. Ultimately, the clean anodic zinc sheet displays the typical roughness of a metallic surface (Fig. 2C).

3.3. Ionic conductivity determination of the DES electrolyte

In recent years, deep eutectic solvents have gained significant attention as promising alternative electrolytes for electrochemical devices [40,41]. A DES typically consists of at least two components: a hydrogen bond acceptor (HBA), such as choline chloride, N, N-dimethylacetamide, or tetramethylurea, and a hydrogen bond donor (HBD), like glycerol, orcinol, or lactic acid. When combined, these components form a mixture with a melting point significantly lower than that of the individual substances [42].

The choice of DES-based electrolyte relies on multiple features (Fig. S4), enabling the developed sensor to reach relevant performances in terms of reliable measurements and sustainable attributes. These features include i) excellent thermal and chemical stability, ii) non-volatility and no flammability, iii) a wide operating temperature range, iv) a broad electrochemical window (up to 2.2 V), and vi) good ionic conductivity. Additionally, to further increase the ionic conductivity, KOH was incorporated into the solution of DES [30]. Finally, to increase the flexibility of the solid-state device, DES-KOH was mixed with a bio-compatible polymer, namely PVA, acting as a plasticizer, facilitating the flexibility of the device and avoiding any leakage of the electrolyte.

In order to measure the ionic conductivity obtained by the home-fabricated DES electrolyte, electrochemical impedance spectroscopy was conducted by inserting the crocodile clamps at the ends of the zinc anode and silver cathode. Impedance spectroscopy measurements were carried out in a frequency range of 100 kHz to 0.1 Hz, with a perturbation voltage of 10 mV. In detail, the obtained data could be visualized as a Nyquist plot, by the imaginary part Z" versus the real part Z' (Fig. 3C).

To determine the ionic conductivity, the Nyquist plot was used applying an electrical equivalent circuit [43]. Typically, a single semicircle in the high-frequency region is mainly related to the charge transfer resistance at the electrode-electrolyte interface, and a straight line in the low-frequency region is attributed to the diffusion. The intersection where the real axis meets the Nyquist plot at high frequency is ascribed to R_s (electrolyte resistance), which is equal to 7 Ω. Therefore, the ionic conductivity was calculated by using the following equation [44]:

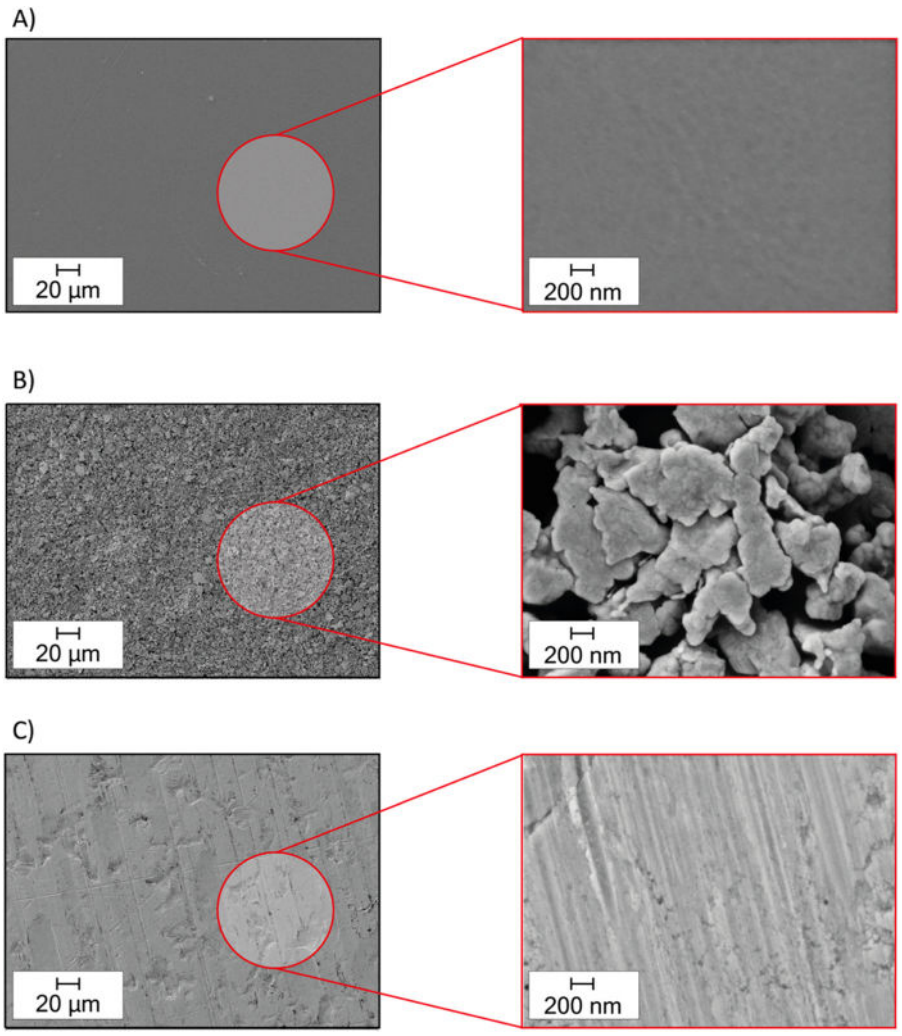

Fig. 2. Micrograph of (A) OPP sheet, (B) Silver ink printed on OPP, (C) Zinc sheet.

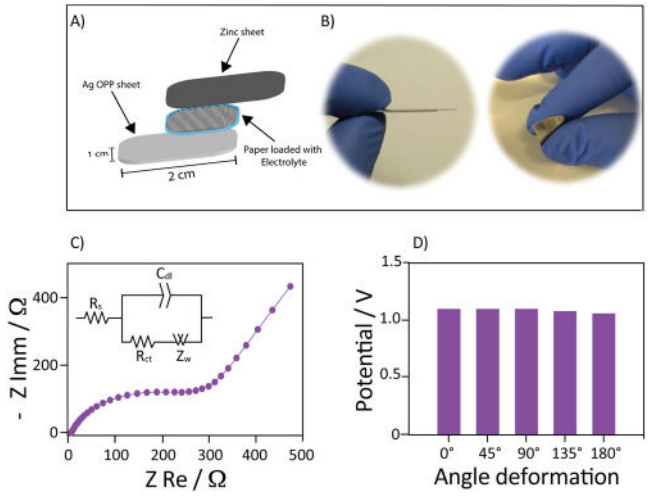

Fig. 3. (A) Schematic representation of the developed sensor; (B) Picture showing the developed sensor (C); Electrochemical impedance spectroscopy measurements obtained using the developed sensor, inset: Randles equivalent circuit; (D) Open circuit potential recorded during the bending test, bending the sensor by angle deformation of 45°, 90°, 135°, and 180°.

$$\sigma = d/(R_s.S)$$

where σ is the ionic conductivity (S/cm), d is the thickness of the developed sensor (cm), R_s is the electrolyte resistance (Ω), and S is the surface area (cm^2). Using the thickness value of 210 µm, the ionic conductivity was found to be equal to 1.5 mS/cm^2. This value is higher than the typical ionic conductivity of perovskite materials (1. 10^{-10} mS/cm^2) for the case of solid-state batteries [45], and it is comparable to the value reported by Biancolli et al. [46] when using solid polymer electrolyte powders for energy storage application.

3.4. Flexibility of the printed electrochemical sensor

To assess the resilience during bending tests, the sensor underwent different angles deformation (namely, 45°, 90°, 135°, and 180°) as shown in Fig. 3B, and the open circuit potential of the sensor was monitored at each deformation using a digital multimeter (IDM91E) as depicted in Fig. 3D. The initial potential dropped to 1.07 V following the deformation test (at 180°) with a loss of 4 % compared to the initial condition. In summary, the sensor exhibits sufficient flexibility and robustness to be exploited in food packaging under various angles and positions.

3.5. Operation of the printed electrochemical sensor

A preliminary test was conducted to investigate the proper

functioning of the sensor. The study was focused on monitoring the output potential variations of the sensor, in the presence and in the absence of oxygen (Fig. 4A). The developed sensor was connected to the portable potentiostat and inserted inside the airtight container.

The measured output potential of the sensor with the open lid of the airtight container, i.e., with 20.9 % v/v oxygen concentration, was equal to 1.124 V. After closing the airtight container lid and introducing pure nitrogen gas, i.e., removing the oxygen, the potential swiftly to 1.143 V.

Finally, upon opening the airtight container lid, the oxygen levels returned to the atmospheric concentration of 20.9 % v/v, and the sensor recorded the initial value after 51 s, as shown in Fig. 4A, demonstrating the reliability of the developed sensor.

3.6. Analytical features

The electrochemical performances of the sensor in response to increasing oxygen concentrations were investigated. Nitrogen was sequentially introduced into a closed airtight container to reach the final oxygen concentration equal to 0 % v/v, 5 % v/v, 10 % v/v, 13 % v/v, 16 % v/v, and 20.9 % v/v, while the developed sensor was connected to the portable potentiostat, and both were inserted in the airtight container. The response was reported in Fig. 4B, showing a decrease of the potential with increasing oxygen concentration, as reported in the literature [47].

The detection of oxygen showed a linear correlation in the concentration range of 0–20.9 % v/v expressed by the calibration curve equal to $y = -(0.9 \pm 0.1)\,x + (1143 \pm 1)$, ($R^2 = 0.984$) (Fig. 4C). The Limit of detection, calculated as ($3 \times \sigma_b$) / S (where "σ_b" is the standard deviation of 10 blank measurements, and "S" is the slope of the calibration curve) was equal to 1.47 % v/v.

The experiment was repeated five times using the same sensor, and the relative standard deviation % (RSD %) was equal to 0.02 %, for 20.9 % v/v oxygen concentration.

3.7. Investigations for application in real-sample monitoring

Environmental changes are one of the main factors that can affect gas detection. Indeed, the involved electron transfer reaction is sensitive to different parameters, such as the number of electron transfers and the diffusion coefficient, which are both temperature-dependent [48]. Therefore, the effect of temperature on the sensor output potential was investigated. As illustrated in Fig. 4D, testing the oxygen concentration of 20.9 % v/v and raising the temperature from 1 °C to 60 °C, the potential increased from 1.094 V to 1.159 V. This phenomenon can be ascribed to the electrochemical oxygen reduction rate and oxygen transfer rate increasing with the temperature [49]. Therefore, like other gas oxygen sensors, the sensor should be paired with other developed or commercial temperature sensors to adjust its measurements accordingly.

To evaluate the selectivity of the device, acetone and ethanol were tested as representative interfering gases. Indeed, these volatile organic compounds (VOCs) are commonly associated with food spoilage, making them relevant interference species as reported in the literature [50, 51].

In detail, ethanol was initially introduced into the airtight container, followed by the potential measurements. The experiment was repeated with the same process using acetone. In both cases, the presence of the tested VOCs did not show any significant potential variation, indicating the selectivity of the developed sensor Fig. 5A.

Additionally, the electrochemical sensor was characterized under both dark and light conditions, exhibiting consistent behavior without any significant variations, as shown in Fig. 5B. This demonstrates the device's robustness and suitability for use inside food packaging, where products are often stored and transported under varying light conditions.

Moreover, the effect of humidity on the sensor response was investigated at different humidity percentage ranging from 20 % to 60 %. As illustrated in Fig. 5C the potential shifted from (1.1231 ± 0.0006) V to (1.1254 ± 0.0002) V, demonstrating a limited impact of humidity on the electrochemical sensor response.

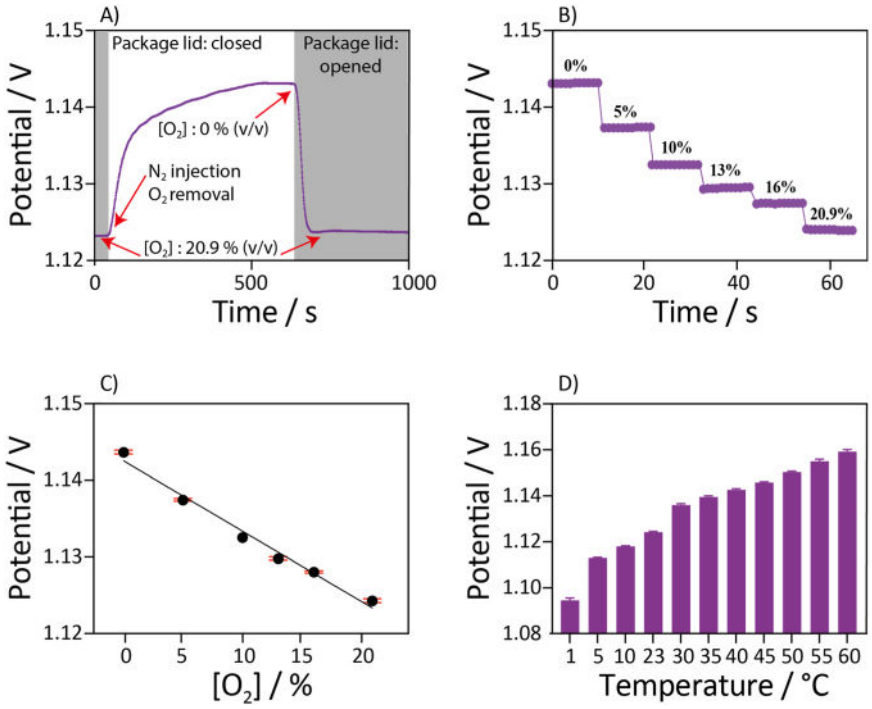

Fig. 4. (A) Electrochemical sensor response to oxygen concentration changes from 0 % v/v to 20.9 % v/v; (B) OCP of the sensor at different oxygen concentrations, (C) Calibration curve of the potential versus oxygen concentration (number of measurements using the same sensor for each oxygen concentration = 5); (D) Dependence of the sensor potential on temperature (number of measurements using the same sensor for each temperature = 5).

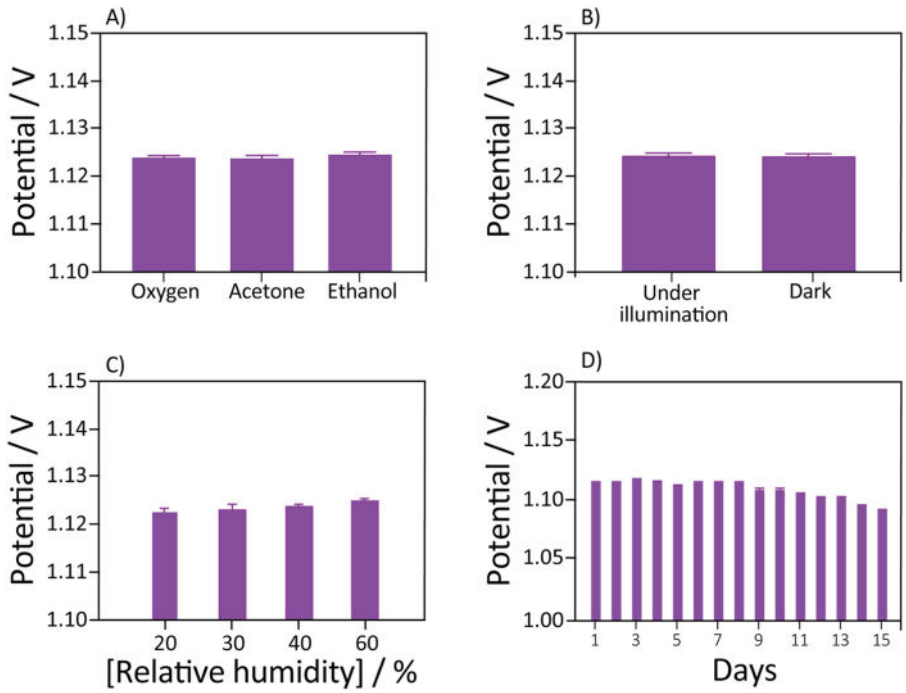

Fig. 5. Histogram bars depicting: (A) the effect of volatile organic compounds presence on sensor response; (B) the sensor response under illuminated and dark conditions; (C) the dependence of the sensor voltage on relative humidity; (D) the storage stability test up to 15 days.

Finally, the stability of the paper-based metal-air battery electrochemical sensor designed for oxygen gas detection was evaluated. Initial tests showed that the electrode maintained a stable potential over two weeks, as illustrated in Fig. 5D. From the first day to day 15, the device exhibited a 2 % potential loss, indicating the need of a pre-calibration procedure before the analysis of the target analyte when used after 9 days from the fabrication. To overcome this drawback, strictly related to applications requiring prolonged monitoring, the use of peptide-enhanced eutectic gels or cross-linkers with tailored features (i.e., resilience, anti-freezing, and anti-drying properties) can be a solution to improve sensor durability and reduce signal loss or potential drift during prolonged use [52].

Table 1
Comparison of the developed device with oxygen sensors reported in the literature.

Sensing Platform	*Detection technique*	*Fabrication method*	*Limit Of Detection*	*Linear range*	*Flexibility*	*Dimensions (cm)*	*Real sample*	*Ref.*
Photoelectrochemical system based on TiO_2/GaN Nanowires on sapphire surface	Electrical resistance induced by UV-light	Nanowires deposited by metal organic chemical vapor deposition	~ 0.45 % v/v	1 – 25 % v/v	No	–	–	[53]
Photoelectrochemical system based on TiO_2/Graphene on silicon surface	Electrical resistance induced by UV-light	Graphene grown by chemical vapor deposition /thermal process	–	0.0134 – 100 % v/v	No	–	–	[54]
TiO_2 on alumina surface	Electrical resistance	Molecular layering deposition	–	0.2 – 10 % v/v	No	–	–	[55]
Photoelectrochemical system based on LaOCl–SnO_2 hollow spheres on alumina surface	Electrical resistance induced by UV-light	Hollow spheres synthesized by hydrothermal process	–	0 – 5000 ppm	No	–	–	[56]
Photoelectrochemical system based on ZnO nanorods on SiO_2/Si surface	Electrical resistance induced by UV-light	Nanorods deposited by spin coating	–	0 – 20 % v/v	No	–	–	[57]
Stretchable hydrogel electrolyte sandwiched between free standing elastomer film based active carbon cloth platinum nanoparticles	Electrochemical	Spin coating	0.003 % v/v	0.003 – 100 % v/v	Yes	–	–	[58]
Stretchable hydrogel electrolyte based on polyacrylamide, encapsulated by ecoflex elastomer film	Electrochemical	Spin coating	–	0.0005 – 90 % v/v	Yes	3.0 × 0.5 × 0.5	Breath	[59]
Metal-air battery based on deep eutectic solvent loaded into paper sandwiched between Zn anode and polypropylene Ag cathode	Electrochemical	1) Deep eutectic solvent synthesized from urea and lithium perchlorate 2) Ag ink screen printed on polypropylene	1.47 % v/v	0 – 20.9 % v/v	Yes	20 × 10 x 0.4	Mushrooms, broccoli, and tomatoes	**This work**

A techno-economic investigation was performed by comparing the developed sensor with commercially available oxygen sensors and other electrochemical sensors reported in the literature (Table 1), and commercially oxygen sensor reported in (Table S1).

The metal-air battery electrochemical sensor is based on office paper, a low-cost, flexible, biodegradable, and easily functionalized analytical platform. Compatible with simple techniques like printing and wax patterning, office paper allows scalable and eco-friendly production. Using screen-printing technique offers a cost-effective, reliable, and scalable alternative to complex methods like MOCVD, CVD-grown graphene, and molecular layering for sensing production [53–55], which require expensive equipment and high temperatures.

Compared to the market devices and literature sensors, the reported metal-air battery-based electrochemical sensor boasts a faster response time, i.e., around 51 s, with a compact size and lightweight of approximately 0.37 g. As a result, the developed sensing device can be easily encapsulated in food packaging, providing a rapid and easy-to-manage response.

Additionally, the fabrication cost of the whole device (including the used seven chemical compounds: Zn sheet, silver ink, OPP substrate, PVA, $LiClO_4$, KOH, and urea) is estimated at 1 €.

Finally, taking into account the fabrication process of the electrodes, the use of the screen-printing technology is a well-established, scalable method. This technique has indeed inherent mass-production capabilities, gaining success over time in various fields, including textiles, electronics, glass, and ceramics. Additionally, the use of easily available and cost-effective materials such as zinc sheet and oriented polypropylene (OPP) fosters the production of this sustainable device.

Furthermore, the use of a deep eutectic solvent (DES) as a green alternative electrolyte offers significant advantages due to its physicochemical properties, low vapor pressure, non-flammability, and good ionic conductivity

3.8. Real samples

To perform measurements directly into food packaging for real sample measurements, a 3D printed holder was fabricated to accommodate the designed sensor and to protect it when inserted in the packaging (Fig. 6).

Once demonstrated the suitability of the sensor for oxygen gas detection, the developed analytical system was applied to monitor oxygen concentration over time in a package containing mushrooms, broccoli, and tomatoes. Furthermore, the electrochemical performances of the sensor were compared to a commercial oxygen analyzer, serving as a reference method.

Fig. 7(A, C, E) shows a decrease in oxygen concentration over time, correlating with the respiration rate of vegetables. This decrease in oxygen concentration is attributed to the natural metabolic processes of the vegetables, which consume oxygen and produce carbon dioxide as they respire [60].

Fig. 7B, D, and F compare the results obtained by the developed sensor vs the commercial sensor for the detection of oxygen in the case of mushrooms, broccoli, and tomatoes, respectively. In detail, a good agreement has been obtained with the commercial sensor, with a correlation factor equal to 0.987, 0.993, and 0.944 in the case of mushrooms, broccoli, and tomatoes, respectively.

In detail, using the commercial analyzer a decrease in oxygen concentration in the package containing mushrooms was recorded from 20.9 % to 9.2 % (Fig. 7 (A, B)), while for a package containing broccoli, a decrease from 20.9 % to 8.3 % (Fig. 7 (C, D)) was registered.

Accordingly, the developed sensor recorded a decrease in oxygen concentration from (20.9 ± 0.2) % to (11.1 ± 0.6) % (Fig. 7 (A, B)), and from (20.9 ± 0.1) % to (10.1 ± 0.1) % (Fig. 7 (C, D)), for mushrooms and broccoli, respectively.

Taking into account the package containing tomatoes, a negligible decrease was observed after 3 h. For this reason, we opted to monitor the oxygen value in the tomato sample after one day of storage, when more significant modifications are observed. As shown in Fig. 7 (E, F), the developed sensor recorded a slight decrease in oxygen levels in tomatoes from (20.9 ± 0.1) % to (17.4 ± 0.3) %, indicating a lower oxygen respiration rate compared to mushrooms and broccoli. This was confirmed by the commercial gas analyzer analyses, with a decrease from 20.9 % to 19.1 %

4. Conclusions

The use of paper in the sensing field has demonstrated over the years the capability of this low-cost and eco-designed material to confer additional features to the devices, such as reagent-free, multi-analyses, and unconventional sample measures without any treatment.

Herein, we investigated for the first time the use of office paper to deliver a flexible metal-air battery for analyte detection. In detail, the paper has been used to obtain a solid-state electrolyte substrate in which the deep eutectic solvent was incorporated into a biocompatible matrix-based polyvinyl alcohol on paper. The office paper worked not only as a sustainable and cost-effective substrate for electrolyte loading but also significantly enhanced the durability and flexibility of the system under diverse deformation angles.

To demonstrate the reliability of this novel concept, oxygen was selected, considering its role as a reagent in the redox reaction of this battery and for application in food packaging, where cost-effectiveness, flexibility, and miniaturization are required features.

The developed sensor exhibited a sensitivity of 0.9 mV/ % v/v O_2, a fast response (51 s), and a good linearity (R^2=0.984) in the linear range

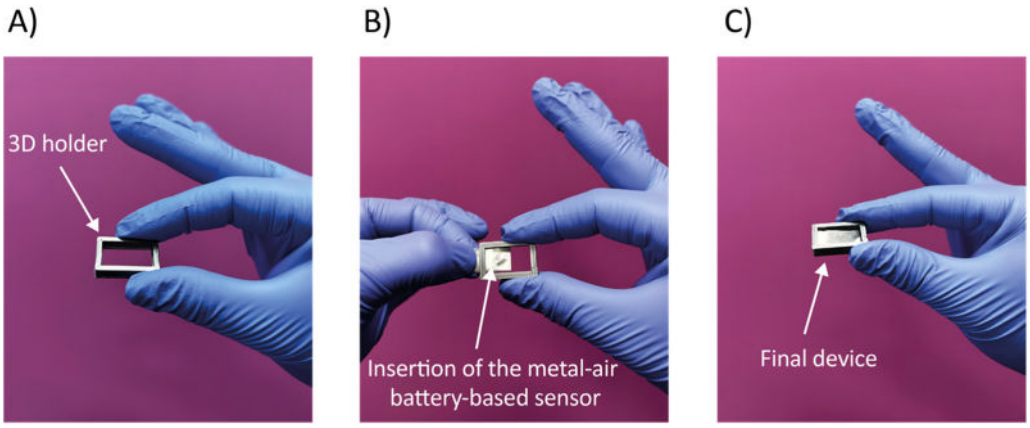

Fig. 6. (A) 3D printed holder; (B) Inserting the metal-air battery-based sensor into the 3D printed holder; (C) Metal-air battery-based sensor fully incorporated into the 3D printed holder.

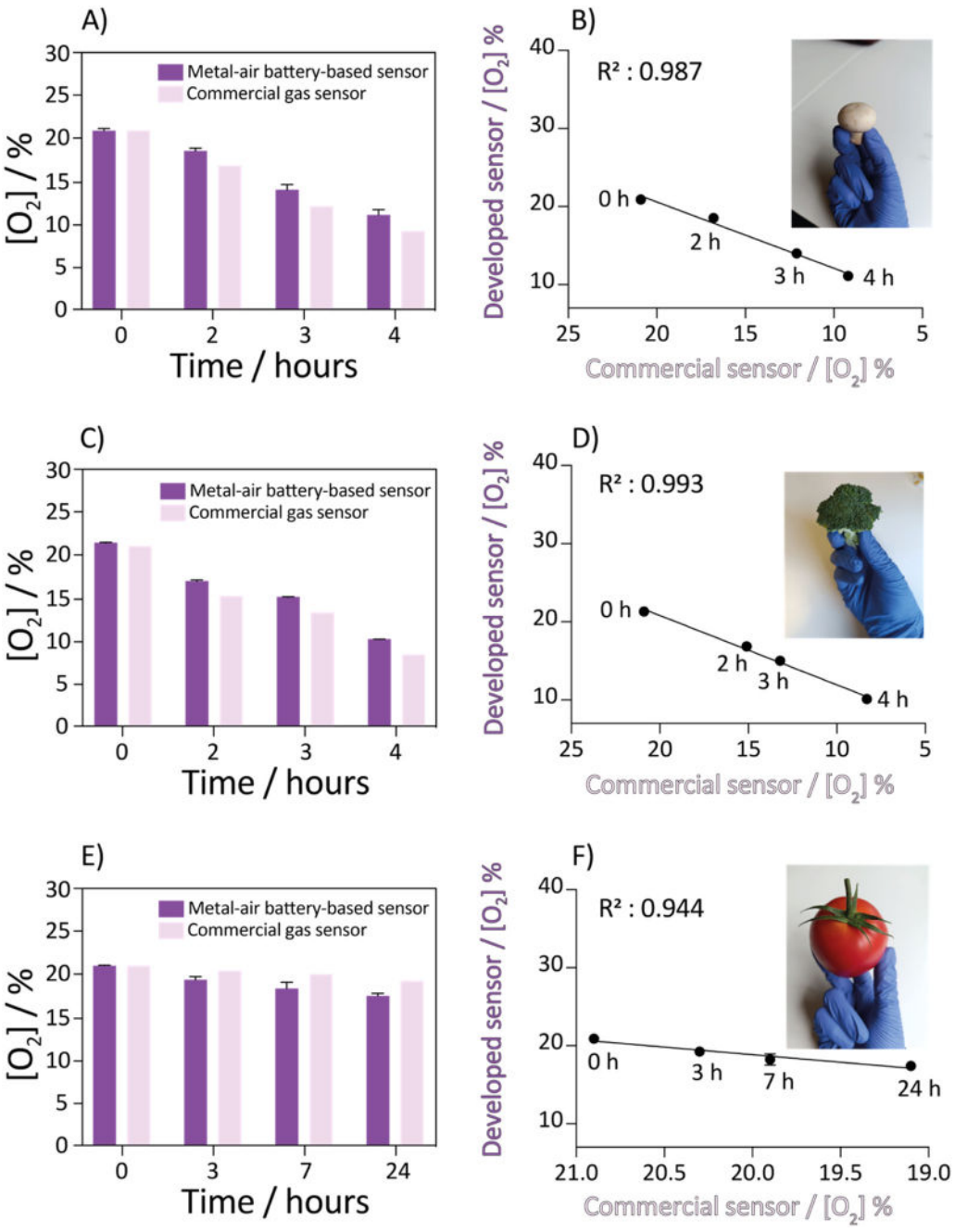

Fig. 7. Oxygen concentration monitoring during storage: (A, B) mushrooms; (C, D) broccoli; (E, F) tomatoes.

up to 20.9 % v/v O_2. The applicability in food packaging was carried out by analysing O_2 content during the time in packaging containing mushrooms, broccoli, and tomatoes, furnishing results in agreement with the reference method, namely a commercial oxygen analyzer.

In summary, we demonstrated the additional application of paper for sensing purposes, beyond the use of paper for reagent-free, multi-analyses, lab on a chip, aerosol and surface monitoring without any sample treatment, by simply embedding a loaded office paper in a flexible metal-air battery.

Following the application explored in this study, the developed sensor can be potentially applied in sectors where monitoring of the oxygen atmosphere is strictly necessary. These include pharmaceutical industries, where oxygen levels are critical for product quality, and industrial factories to control oxygen concentration in particular working environments.

CRediT authorship contribution statement

Achref Chebil: Writing – review & editing, Writing – original draft, Methodology, Formal analysis, Data curation, Conceptualization. **Fabiana Arduini:** Writing – review & editing, Project administration, Funding acquisition, Conceptualization. **Ludovica Gullo:** Investigation. **Leonardo Duranti:** Writing – review & editing, Investigation. **Vincenzo Mazzaracchio:** Writing – review & editing, Writing – original draft, Supervision, Methodology, Conceptualization.

Declaration of Competing Interest

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

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.sna.2025.116922.

Data availability

Data will be made available on request.

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