# Non-Invasive Oxygen Determination in Intelligent Packaging Using a Smartphone

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***Abstract*— Here, we present a technique for the determination of the gaseous oxygen concentration inside packed food. It is based on the use of a luminescent membrane sensitive to $O_2$ that is optically excited and read by a smartphone. The flash of the smartphone along with an optical filter is used as the light source for the optical stimulation of the membrane. The luminescence generated, which is quenched by the surrounding gaseous oxygen, is registered by the rear camera of the same device. The response parameter is defined by combining the registered intensities at two different wavelength ranges corresponding to the emission and the absorption peaks of the sensitive membrane. Thanks to this novel response parameter, the sensitivity is increased and, more importantly, the thermal dependence of the membrane is significantly reduced. This approach allows the use of a luminescent $O_2$-sensitive membrane for intelligent packaging with no need of any associated electronics for its excitation and reading. This means that an oxygen sensor is developed, where a luminescent compound acts as an indicator, therefore combining the advantages of both schemes, that is, the simplicity and reduced cost of indicators with the high sensitivity and accuracy of selective sensors.**

***Index Terms*— Intelligent packaging, oxygen sensing, optical sensor, luminescent membrane, smartphone, Android$^{\text{TM}}$ application.**

## I. INTRODUCTION

ALONG with its primary objective of containment and transportability, the main aim of packaging is the protection and preservation of food and beverage from external contamination. Thanks to the presence of an appropriate packaging, the content can be prevented from deterioration, its shelf life is extended and its quality and safety preserved. In the last decades there has been an increasing interest regarding research and development of new technology in food and beverage packaging, due to the growing requirement from the final consumers to receive a guarantee of freshness, quality and safety of the product.

Three major strategies have been followed in order to add new functionalities to packaging systems: active and intelligent packaging (AP and IP), edible films/coatings and modified atmosphere packaging (MAP) [1]. Active packaging allows incorporation of additives, such as gas scavengers or temperature and moisture controllers among others to enhance the quality and sensory aspects of packaged foods [2]. Intelligent packaging is oriented to gather information related to the status of the content, and transmit it to the consumer [3]. Edible films are defined as thin layers of materials that can be consumed and provides barriers to moisture, oxygen and solute movement for the food [4]. MAP may be defined as an active packaging method in which an altered atmosphere is created in the headspace, which retards chemical deterioration while simultaneously delays the growth of spoilage organisms [5].

One of the most important parameters to sense in intelligent packaging and MAP is the concentration of gaseous oxygen within the package, since oxygen is the main cause of food spoilage [6], [7]. The presence of an atmosphere with some elevated concentration of $O_2$ (above 2%) facilitates processes such as promotion of microbial growth, lipid oxidation, protein decomposition, and discoloration [8].

In IP and MAP there are two main technologies for monitoring the analytes of interest: indicators and sensors [9]. Indicators are molecules that displays an optical effect with a specific analyte such as oxygen [10]. They can be disposed as two-dimensional membranes, aimed to provide direct information about the presence and/or concentration range of a substance. For oxygen determination, many indicators have been developed following this principle. Some of them are designed to react to the mere presence of oxygen, showing a color drift in this case [11], while others present a wide color displacement related to the oxygen concentration, thus allowing a qualitative detection of the oxygen [8], [12]. In this last case, the color difference should be significant for different concentrations to prevent the misreading of the consumer. Another type of optical oxygen indicators are those based on a luminescent response. In such systems, the luminescence generated by an optically excited luminophore is related, in terms of intensity or lifetime, to the concentration of the surrounding molecular oxygen [13]. These indicators also

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AQ:4

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provide qualitative information about the oxygen concentration within the package [14], [15]. The drawback of luminescent indicators is that they require an external light source. Sensors are more complex systems or devices that include control and processing electronics, interconnection network and software. A sensor contains one or more detectors that generate a signal proportional to the monitored analyte or magnitude. This signal is processed and transmitted as information to a user or consumer, thus providing quantitative data. Oxygen sensors are able to provide the value of the oxygen concentration with very high resolution [16]–[18].

In the last two decades there has been a great effort to adapt or create oxygen sensors that are suitable to be included in intelligent packaging [8], [19]–[21]. The most promising oxygen sensors are based on luminescent detectors, following the same operation principle than the luminescent-based indicators. They offer fast responses, do not consume any analyte and present high sensitivity and accuracy. Many examples of oxygen sensors for intelligent packaging have been proposed based on this type of sensors [22]–[27]. The main drawback in all cases is that they require the presence of a light source, a light detector, processing and communication electronics and powering. This leads to a whole circuitry that must be included in the package in a process that becomes very expensive and complex. As a consequence, the inclusion of full sensing capabilities in intelligent packaging may result prohibitive.

In previous works the authors proposed some designs for gas sensing including oxygen [23], [28], [29]. These prototypes are based on passive flexible tags that generate power supply from energy harvesting, therefore they are suitable to be used as sensors in intelligent packaging and MAPs. Nevertheless, they are still complex and expensive. In this work, we present a new approach in which the luminescent oxygen sensor is simplified by removing the whole electronics, thus reducing it to a single oxygen sensitive membrane. The optical excitation of this membrane is carried out by the application of the flash light of a smartphone along with an optical filter at a fixed distance, and the emitted luminescence is registered in a photography taken with the rear camera of the smartphone. Similar schemes can be found in the literature but using optical fibers as an alternative to the free-space orientation of the proposed sensor [30], [31]. The processing capabilities of this device allow the evaluation of the intensity of this luminescence, thus generating an accurate prediction of the inner oxygen concentration. In this way, the sensitive membrane is treated as an indicator since no electronics are used, but it maintains its sensor characteristics. The system offers the advantages of both schemes: the simplicity and very low cost of an indicator together with the high sensibility and accuracy of a sensor. Moreover, the scan of the luminescent membrane using the camera of the smartphone removes the subjectivity of the consumer in the reading of the provided information.

## II. MATERIALS AND METHODS

### A. Reagents and Materials

Platinum octaethylporphyrin complex (PtOEP), 1,4-diazabicyclo[2.2.2] octane (DABCO, 98%), tetrahydrofuran (THF) and polystyrene (PS, average MW 280,000, Tg: 100 °C, GPC grade) were all supplied by Sigma–Aldrich Química S.A. (Madrid, Spain). The gases $O_2$ and $N_2$ ($>99\%$) were supplied in gas cylinders by Air Liquide S.A. (Madrid, Spain).

### B. 2.2 Instruments and Software

In order to calibrate and characterise the proposed system, standard gas mixtures were prepared using nitrogen as inert gas and oxygen, controlling the different percentages using mass flow controllers (Iberfluid, Barcelona, Spain), work pressure 1 atm and flow rate 5 Nl $\bullet\text{min}^{-1}$.

For the monitoring of freshness in pork meat, a thermostatic chamber, with a lateral hole for the connection to a computer and gas tubing entrance, made possible to maintain a controlled temperature between $-50$ °C and $+50$ °C with an accuracy of $\pm 0.1$ °C for thermal characterization. A heat sealer PFS-300MM Electric Impulse Sealing Machine C. (Media w.s. trade S.L. Barcelona, Spain), high barrier bag material BB3055 specific for meat products were obtained from Sealed Air (Seville, Spain) and a CheckPoint – Handheld Gas Ana-lyzer ($O_2/CO_2$) Dansensor A/S (Rønnedevej 18, DK-4100 Ringsted, Denmark) was used as reference method for $O_2$ measurement inside meat packages.

The smartphone used in this work was the Samsung Galaxy S7. This smartphone features a 12-megapixel rear camera with an f/1.6 aperture, focal length of 26mm, optical image stabilization and autofocus. The AndroidTM version running on the device was 7.0 Nougat, which corresponds to Application Programming Interface (API) level 24..

### C. Sensing Membrane Preparation

The cocktail for the preparation of the oxygen-sensitive membrane was made by dissolving 0.5 mg of PtOEP and 12 mg of DABCO in 1 ml of a solution of 5% (w/v) of PS in freshly distilled THF. The sensitive membrane was cast by placing a volume of 20 $\mu$L of the cocktail on an inert support using a spin-coater at 180 rpm under ambient atmospheric conditions. After the deposition, the sensing membranes were left to dry in darkness in a THF atmosphere for at least 1 h. The obtained membranes were homogeneous, transparent and pink colored. When they are not in use, they must be kept in darkness to extend their lifetime.

### D. System Description

The proposed system is composed of two elements: the oxygen sensitive membrane that is attached to the inner surface of the food package, in contact with the inner atmosphere; and a smartphone used to excite the membrane and register the emitted optical signal generated through a programmed application, as it is schematised in Figure 1.

The flash light is turned on and the device is placed at a fixed distance of the membrane. This light contains the wavelength required for the optical excitation of the sensitive membrane. To reduce optical interference with the rest of wavelengths, an optical filter is attached to the flash light of the smartphone. The filter is a non-commercial 50$\mu$ m-thick

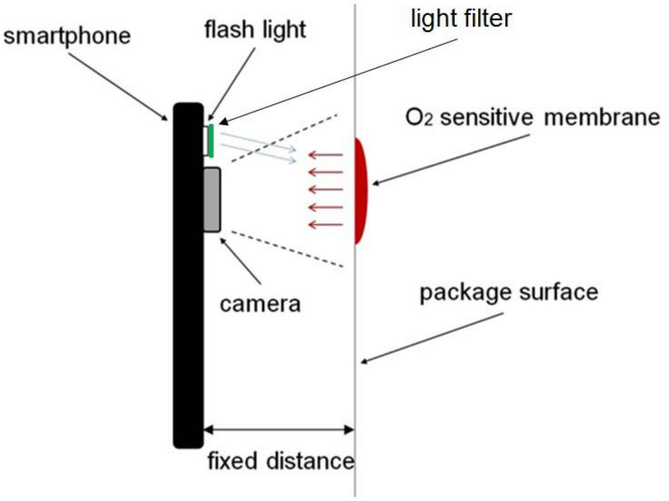

Fig. 1. Scheme of the system used to sense $O_2$.

green plastic film. Several films with different green tones have been tested until achieving the maximum transmittance at the desired wavelengh. Under stable excitation, the generated luminescence presents an intensity that is proportional to the concentration of the surrounding oxygen. In this situation, the smartphone takes a photograph of the excited membrane and processes it to obtain a value of the emitted intensity. An accurate prediction of the oxygen concentration inside the package is computed from this emitted intensity value [17].

### *E. Android Application*

A custom developed Android application allows the user to obtain the oxygen concentration by simply approaching the phone to the oxygen sensitive membrane. The application user interface consists of a single white reference circle that is superimposed onto the camera preview. Firstly, the application automatically turns the camera flash light on to excite the membrane. When the camera is aimed at the circular membrane, the application is able to detect it and distinguish it from the background. In order to take the photograph, the user must match the reference on-screen circle with the detected membrane. When both shapes match, a photograph is automatically taken and saved. In this way, the distance between the phone camera and the sensitive membrane is always identical and fixed, so that the same conditions from calibration are achieved. The next screen of the application shows the taken photograph and computes the average red (R), green (G) and blue (B) components inside the detected membrane. After the image processing, which takes less than two seconds, the oxygen concentration computed from these RGB components according to the previous calibration is displayed on the screen, as shown in Figure 2. Finally, the application saves the processed photograph along with the obtained results to the phone internal memory.

The application has been designed using the integrated development environment (IDE) Android Studio 2.3.3. It has been developed and tested against API 24 (Android 7.0), although it is compatible with different Android versions as the lowest supported API level is API 18 (Android 4.3). The algorithms developed to accomplish the detection and processing tasks are based on computer vision OpenCV 3.1.0 Android library. This application is extendable to any Android-based device (smartphone and tablet). For other mobile operating systems, the application should be translated into their appropiate programming language.

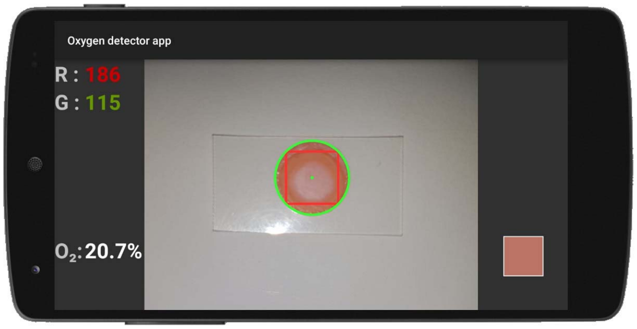

Fig. 2. Screenshot of the developed Android application.

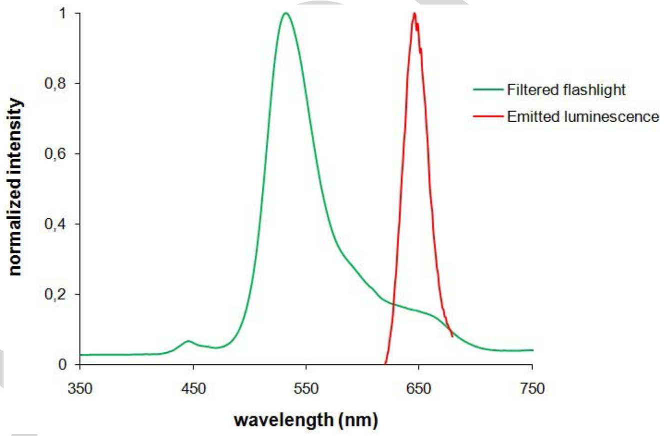

Fig. 3. Normalized emission spectra of the luminophore and emitted light of the smartphone flashlight with external filter.

## III. RESULTS AND DISCUSSION

For the determination of $O_2$, a chemical sensor based on luminescence quenching of the complex PtOEP is used. When this luminophore is optically excited at the wavelengths of 380 and 532 nm, it produces a luminescent emission in the red region of the spectrum with a peak at 645 nm [17]. Figure 3 presents the normalized emission spectrum of this luminophore together with the spectrum of the filtered flashlight of the smartphone.

As it can be seen, the filtered excitation light presents a maximum at the wavelength of 529 nm, which fits almost perfectly with the absorbance peak of the luminophore. Therefore, assuming that the system is optically isolated, only the green light of the filtered source and the red luminescence emitted by the oxygen sensitive membrane have influence on the registered photograph.

Figure 4 shows an example of the response of the system. Here the original and false-color images of an oxygen sensitive

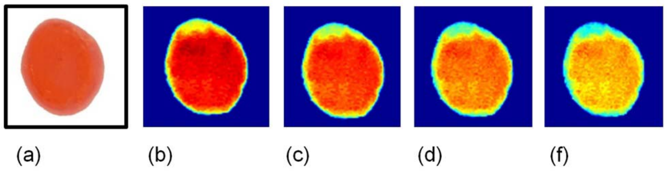

Fig. 4. Oxygen sensitive membrane (a) and false-color processed images under growing oxygen concentrations 0%, 30%, 50% and 100% (b-f).

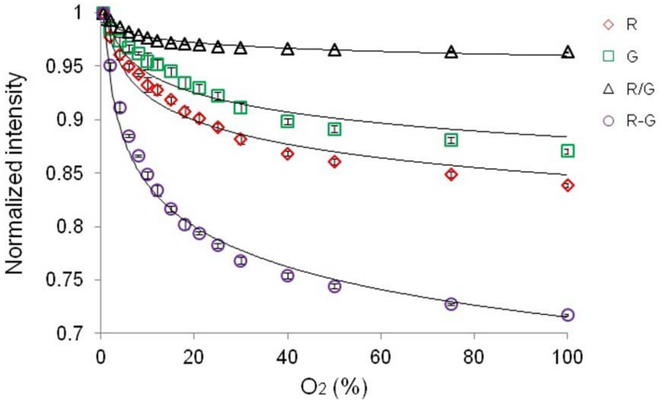

Fig. 5. Calibration and fitting curves for $O_2$ at room temperature using different intensity quantification parameters.

membrane immersed in atmospheres with different oxygen concentrations are depicted. As it is expected, the intensity of the red luminescence decays when the oxygen concentration is increased [32].

### *A. System Characterization*

The sensing membrane based on PtOEP(PS) has been widely used for oxygen detection, and it is well known that it shows no cross-sensitivity to carbon dioxide or humidity; nevertheless, the temperature has a strong influence on its response. Therefore, this membrane has to be fully characterized by analysing the emitted luminescence at different oxygen concentrations and temperatures. With this aim, two independent calibrations have been carried out. On the one hand, a calibration of this membrane in the full oxygen range 1-100% at room temperature (20°C). On the other hand, a calibration over the temperature range from 0 to 40°C at open air oxygen concentration (20.9%). In both cases 6 replicas for each concentration are taken. The obtained results are presented in Figures 5 and 6, respectively.

In previous works, the intensity of the luminescence generated by the oxygen sensitive membrane was quantified by means of the red component of the image [17], [28] from the red-green-blue (RGB) space, since it corresponds purely to this luminescence assuming that the system is optically isolated from external light. In other cases, a combination of this R component and the corresponding to the wavelength of the optical source for the excitation has been proposed [23], since it can reduce the influence of small fluctuations of the source. In this work, other parameters for the quantification of the image intensity are evaluated and shown in the Figures 5 and 6. The considered parameters are the R component, thus only including the intensity of the emitted luminescence; the G component, which provides information about the absorbance of the membrane, is also affected by the concentration of the surrounding oxygen; and two relationships derived from these components: the R/G ratio and the R-G difference, where the information about the emitted and absorbed intensities are combined.

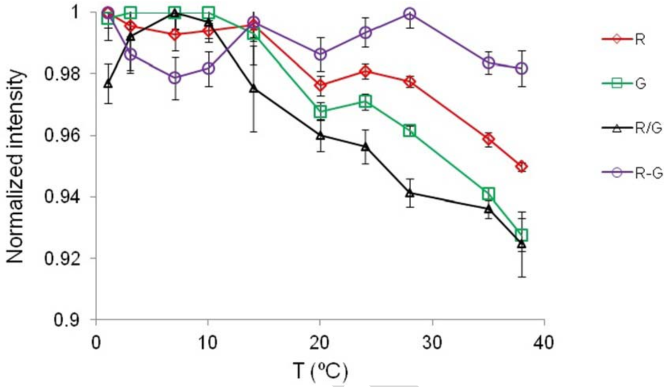

Fig. 6. Calibration curves of temperature at open air using different intensity quantification parameters.

From the results shown in Figure 5, it can be observed that the fitting curve of the normalized intensity to the oxygen concentration, whichever parameter is selected to be representative of the intensity of the image, responds to a potential equation of the form $I = \alpha \bullet [O_2]^{\beta}$. Nevertheless, the wider signal variation in the full range of $O_2$ is obtained for the parameter R-G, which means that improved sensitivity is obtained if this parameter is used to quantify the intensity of the image. For this case, the fitting curve expression is:

$$I_n = \frac{I}{I_0} = 1.0057[O_2]^{-0.076} \qquad (1)$$

where $I$ is the intensity given by R-G in the processed image, $I_0$ the intensity at the minimum oxygen concentration and $I_n$ the normalized intensity. The coefficient of correlation of this fitting curve is $R^2 = 0.9947$.

From the curves presented in Figure 6, it can be concluded that the intensity of the image calculated as intensity of the luminescence (R), absorbance (G) or a combination of these two magnitudes (R/G, R-G) decays with temperature. This is a common effect in luminescence sensors that has been widely explained in the existing literature [17], [33], [34]. Nevertheless, the published studies show that the influence of the temperature on the response of the luminescent membrane is not well-defined but it shows a behaviour that is not monotonically decreasing and therefore it cannot be fitted to a simple function [35], as it is confirmed by the curves depicted in Figure 6. Therefore, it is difficult to carry out a thermal compensation of the response of the sensitive membrane since there is no accurate fitting expression for this dependence. In addition, a thermal compensation requires the presence of a temperature sensor. In this work, this sensor is not available since our aim is to avoid any electronics from the envelope, and the current smartphones do not include it. In view of this situation and in the light of the curves of Figure 5, the R-G parameter is the most suitable to represent the image intensity since it has the lowest variation with temperature, which is limited to 2.1%, while the variation of the rest of parameters is above 5%. Consequently, we can assume that the error in the determination of the R-G parameter is 2.1% in the worst case and no further thermal compensation is required.

In summary, from the calibrations presented in Figures 5 and 6, the novel intensity parameter defined as R-G, where information about luminescence and absorbance is combined, is used to relate the image of the sensitive membrane to the oxygen concentration. This parameter offers advantages such as increased sensitivity and less temperature dependence.

The resolution of the system can be obtained from Equation (1) taking derivatives in both sides of the equation and approximating them to increments [23]. By doing so, the obtained expression for the resolution is:

$$\Delta O_2 = \frac{\left(\frac{I_n}{\alpha}\right)^{\frac{1}{\beta}}}{\beta I_n} \Delta I_n \tag{2}$$

where $\Delta I_n$ is the error in the determination of the normalized intensity $I_n$, which is taken as 2.1% because of the temperature influence as explained above. In this case, the average obtained resolution for the prediction of the oxygen concentration is 30%. If the package is kept at a constant temperature as it happens in the storage of foods, the thermal drift is minimized, and the error in its determination is given by the accuracy of the system. This parameter is taken as the standard deviation of the replicas taken for each measurement of Figure 5, obtaining a value of 0.31%, which leads to a resolution of 4.4% of the predicted oxygen concentration.

### *B. Case Study*

The proposed system has been applied to the monitoring of freshness in pork meat. It is known that the concentration of oxygen in a sealed package containing the meat is related to the bacterial activity [36]. Therefore, a measurement of the inner $O_2$ concentration provides direct information about the state of the content. For this experiment raw pork was purchased fresh, 500 g packaged inside an $O_2$ impermeable bag, and sealed using the impulse bag sealer. Two identical oxygen membranes have been used, one inside the packed fresh pork and the other one as reference outside the package. The objective of the reference membrane is to show that the changes in the response of the internal membrane are not produced by external variations but only by the modification of the internal atmosphere. Figure 7 shows the analysed package.

This package has been stored in a controlled-temperature environment at 4°C for a week. During this time, measurements of the internal oxygen concentration have been carried out by using the gas analyser as well as the proposed system. 6 replicas for each measurement were taken. Figure 8 presents the obtained results. In Figure 8-A the evolution of the normalized intensity parameter R-G in both external and internal sensitive membranes is depicted. As it can be seen, the response of the reference membrane remains constant, while the corresponding to the sensing membrane grows over time. The variation of the signal generated by the internal membrane is processed and translated into variation in the predicted oxygen concentration within the package, as it is shown in Figure 8-B, where it is compared to the direct oxygen measurements obtained with the gas analyser. As it was expected, the oxygen concentration decays over time as a result of the bacterial activity. The predicted values of oxygen

Fig. 7. Pork meat in a sealed package where internal and external sensitive membranes have been attached.

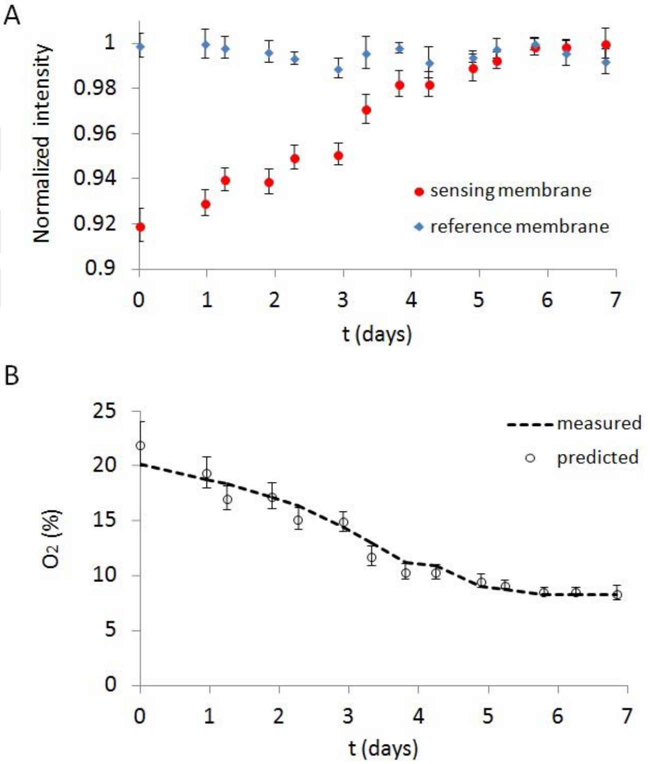

Fig. 8. Response of the internal and external membranes (A) and oxygen concentration within the package (B).

obtained with the novel system are in good concordance with the direct measurements taken using the reference method.

## IV. CONCLUSION

In this work a novel approach for oxygen determination in intelligent packaging is proposed. In the presented scheme, an oxygen sensitive membrane with luminescent response is attached to the inner surface of the packed food. A smartphone is used for simultaneous excitation and reading of the membrane, thus avoiding the necessity of any additional electronics integrated in the envelope to complete the oxygen sensor. This leads to a system where the sensitive membrane acts as an indicator since no other elements in the package are required to provide information about the oxygen concentration. Moreover, the system also has the advantage of providing qualitative information, that is, accurate predictions of the oxygen concentration inside the package, as a full sensor. An easy-to-use Android app has been developed to take a photograph of the membrane at a fixed distance. The application also processes it in order to obtain the colorimetric information about the intensity of the luminescence and the absorbance of the membrane. These magnitudes are combined to define a new intensity parameter that is related to the oxygen concentration. This parameter shows improved sensitivity and immunity to thermal drift. The system has been applied to the monitoring of pork meat freshness sealed in a package and stored into a temperature-controlled environment. The results show that the developed system is able to provide accurate information about the oxygen concentration inside the package, information that is directly related to the bacterial activity and therefore to the state of meat.

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