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Fermentation endpoint detection of Soybean using specially designed *Pd*-coated FBG stress sensor

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Determining the fermentation endpoint of organic compounds is critical for optimizing yield, ensuring the product consistency, and minimizing byproducts. However, conventional detection methods are slow, labor-intensive, and lack real-time monitoring, limiting their suitability for industrial automation. We propose a novel, non-destructive method for real-time detection of fermentation endpoint of Soybean using Palladium (*Pd*)-coated fiber Bragg grating (FBG) stress sensor. The fermentation endpoint can be detected by monitoring the shift in Bragg wavelength caused by the stress in *Pd*-coated FBG sensor due to the volume expansion of the *Pd* coating upon the formation of Palladium Hydride (*PdH_x*) after Hydrogen (*H₂*) gas absorption, which is released as a byproduct during Soybean fermentation. The *Pd*-coated FBG stress sensor is analytically designed and validated using OptiSystem simulation tool, achieving a high sensitivity of 61.6 p.m./MPa. Our findings confirm that this method provides a simple, efficient, and real-time solution for monitoring the fermentation process of organic compounds that produce *H₂* offering significant advantages over traditional techniques.

KEYWORDS

fermentation endpoint, Soybean, Palladium, Hydrogen sensing, fiber Bragg grating, wavelength sensitivity, induced stress

1 Introduction

Fermentation of organic compounds is a vital biochemical process with significant applications in food industry, biofuel generation, and biotechnology [1]. It involves the microbial metabolism of organic substrates such as carbohydrates into simpler compounds like alcohols, organic acids, and gases facilitated by bacteria, yeast, and fungi [1]. Detecting the fermentation endpoint is essential to ensure optimal yield, quality, and process efficiency [1]. For example, in the fermentation of Soybean into acetic acid, the process occurs in two stages [1, 2]: the first is the hydrolysis of Soybean proteins and carbohydrates into simpler sugars and amino acids, followed by the oxidation of ethanol to acetic acid by acetic acid bacteria (AAB) such as

acetobacter species. Accurate endpoint detection is crucial to prevent over-oxidation which could degrade acetic acid into carbon dioxide and water, compromising both quality and yield [2]. Advanced analytical techniques, including pH monitoring, gas chromatography, and high-performance liquid chromatography are commonly used to determine the fermentation endpoint and maintain the desired acetic acid concentration. Research emphasizes the importance of controlling factors like temperature, oxygen levels, and microbial activity to optimize the process [2, 3]. Beyond enhancing the nutritional profile of Soy-based products, Soybean fermentation for acetic acid production also yields valuable ingredients for the food industry such as vinegar and condiments, showcasing the broader economic and nutritional significance of fermentation [2, 3].

From an economic perspective, the production of acetic acid from Soybean's fermentation is highly valuable due to its applications in food, pharmaceuticals, and chemicals. This process is cost-effective compared to synthetic production as it utilizes natural source, thus reducing the energy consumption and production costs. The growing demand of acetic acid produced through natural sources has further boosted its market value. The global acetic acid market, valued at over \$10 billion in 2022, is projected to grow, driven by its use in food processing, textiles, and biodegradable plastics [4]. Fermentation supports sustainability and creates economic opportunities for agricultural communities by utilizing local raw materials.

2 Related works

Determining the fermentation endpoint in Soybean processing is crucial for ensuring the product quality, maximizing yield, and avoiding over-fermentation which can result in unwanted byproducts. A range of traditional analytical techniques are utilized to identify when fermentation is complete. These methods include pH monitoring [5], gas chromatography (GC) [6], high-performance liquid chromatography (HPLC) [7], and Fourier transform infrared (FTIR) spectroscopy [8]. Analytical methods for fermentation endpoint detection often suffer from limitations such as time consuming sample preparation, high operational costs, potential contamination risks, and need for specialized equipment and expertise. Non-destructive, real-time, and online techniques for fermentation endpoint detection are essential to overcome the limitations of traditional analytical methods ensuring continuous monitoring and improved process efficiency. Various real-time techniques have been proposed to monitor the fermentation endpoint in different organic compounds. For example, monitoring the fermentation in dairy products using fluorescence spectroscopy [5], ultrasonic sensor [9], infrared light backscatter sensor [10], near-infrared (NIR) spectroscopy [11], monitoring the fermentation process in ethanol using viable cell sensor [12], monitoring the fermentation in yeast using software controlled automatic real-time biosensor [13], monitoring the fermentation process in Soybean using miniature fiber NIR spectrometer [14], monitoring the fermentation process in wine with benchtop 1H NMR spectroscopy [15] and NIR spectroscopy [16], and monitoring the fermentation process in black tea using an electronic tongue [17]. The literature review has been further elaborated in Table 1

by comparing the important achievements of past studies with proposed work.

We introduce a novel non-destructive approach for real-time detection of the Soybean fermentation endpoint using a *Pd*-coated FBG stress sensor. This method detects fermentation completion by tracking the Bragg wavelength shift caused by stress in the *Pd*-coated FBG sensor due to *Pd* coating expansion upon *PdH_x* formation after *H₂* absorption which is a key fermentation byproduct. The sensor is analytically designed and validated using OptiSystem simulation tool having a wavelength sensitivity of 61.6 p.m./MPa. This pioneering work establishes a new pathway for real-time monitoring of fermentation processes involving *H₂* production.

3 Modelling the fermentation process and working principle

To accurately model the fermentation process of Soybean and explain the working principle of the proposed method, it is important to estimate the total yield of *H₂* released as byproduct during fermentation process and the amount of stress induced on the *Pd*-coated FBG sensor by volume expansion of the *Pd* coating due to the formation of *PdH_x* after absorption of *H₂*, where *x* is the ratio of Hydrogen to Palladium. The fermentation of Soybean involves microbial activity that degrades organic compounds, resulting in the production of *H₂* alongside other byproducts such as organic acids and carbon dioxide *CO₂*. The fermentation process of soybean can be expressed by following chemical equation [18].



The Equation 1 illustrates the fermentation process of Glucose (*C₆H₁₂O₆*) which is considered as a primary component in Soybean, into acetic acid (*CH₃COOH*), *CO₂*, and *H₂*. From this equation, it is evident that 1 mol of *C₆H₁₂O₆* produces 4 mol of *H₂*. Therefore, the total mass of *H₂* produced per kg of *C₆H₁₂O₆* can be calculated by following steps.

Molecular weight of *C₆H₁₂O₆* = 80 g/mol

Molecular weight of *H₂* = 2 g/mol

Mass of 4*H₂* = 4 × 2 = 8 g

Thus, 180 g of *C₆H₁₂O₆* (1 mol) yields 8 g of *H₂*. The mass of *H₂* produced per kg of *C₆H₁₂O₆* is calculated as.

$$\frac{8 \text{ g}}{180 \text{ g}} \times 1000 = 44.49 \text{ g}$$

Therefore, 1 kg of *C₆H₁₂O₆* produces approximately 44.49 g of *H₂*. As we have considered 5 kg Soybean in this research, therefore 222.5 g ($\approx 2.5 \text{ m}^3$) of *H₂* gas is produced as byproduct during the fermentation process. To calculate the volume of *H₂* generated during fermentation at STP, we use the ideal gas law. At STP, the molar volume of an ideal gas is 22 L/mol. The molar mass of *H₂* is 2 g/mol, so for 222.5 g of *H₂*, the number of moles is calculated as:

$$\text{Number of moles} = \frac{\text{Mass}}{\text{Molar mass}} = \frac{222.5 \text{ g}}{2 \text{ g/mol}} = 111.25 \text{ mol.}$$

TABLE 1 Elaboration of the literature survey and comparison with proposed work.

Study	Organic compound	Technique	Sensing type
[5]	Yogurt	Offline and destructive	pH monitoring
[6]	Milk	Offline and destructive	Gas chromatography
[7]	Grapes	Offline and destructive	Liquid chromatography
[8]	Oat and pea	Offline and destructive	FTIR spectroscopy
[9]	Yogurt	Online and non-destructive	Ultrasonic measurement
[10]	Milk	Online and non-destructive	Light scattering
[11]	Yogurt	Online and non-destructive	NIR spectroscopy
[12]	Ethanol	Online and non-destructive	Cell sensor
[13]	Yeast	Online and non-destructive	Biosensor
[14]	Soybean	Online and non-destructive	NIR spectroscopy
[15]	Wine	Online and non-destructive	1H NMR spectroscopy
[16]	Wine	Online and non-destructive	NIR spectroscopy
[17]	Black tea	Online and non-destructive	Electronic tongue
Proposed	Soybean	Online and non-destructive	FBG sensing

Using the molar volume of an ideal gas at STP, the volume of H_2 gas is:

Volume = Number of moles
× Molar volume
= 111.25 mol × 22.4L/mol
= 2492 L.

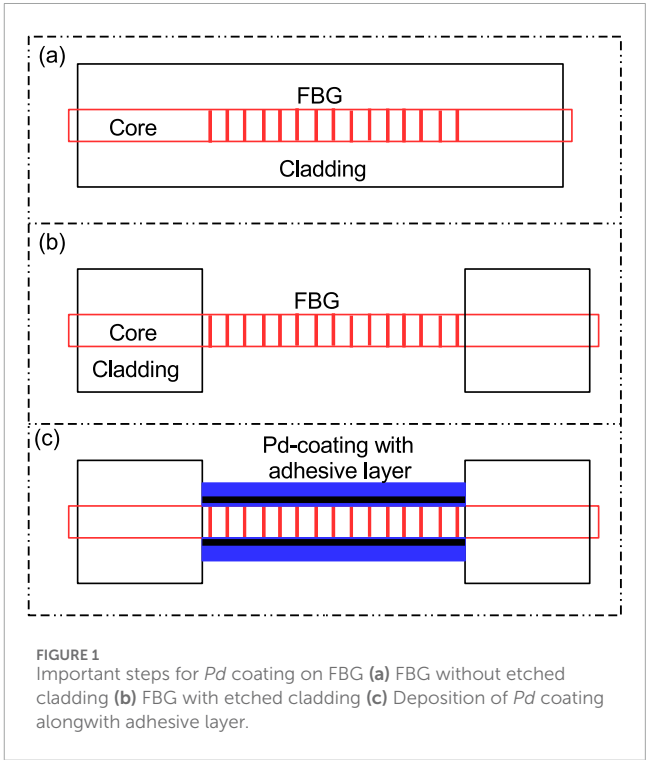
Converting liters to cubic meters (since $1\text{ m}^3 = 1000\text{ L}$), the volume of H_2 generated at STP is:

Volume = $\frac{2492\text{ L}}{1000} = 2.492\text{ m}^3$.

To determine the stress induced in *Pd*-coated FBG sensor when exposed to 222.5 g of H_2 , first we need to consider the H_2 absorption in *Pd* coating, saturation limit of PdH_x formation, and stress-strain relationship. Then we shall be able to calculate the shift in Bragg wavelength of FBG stress sensor.

3.1 H_2 absorption capacity of *Pd* and saturation limit of PdH_x

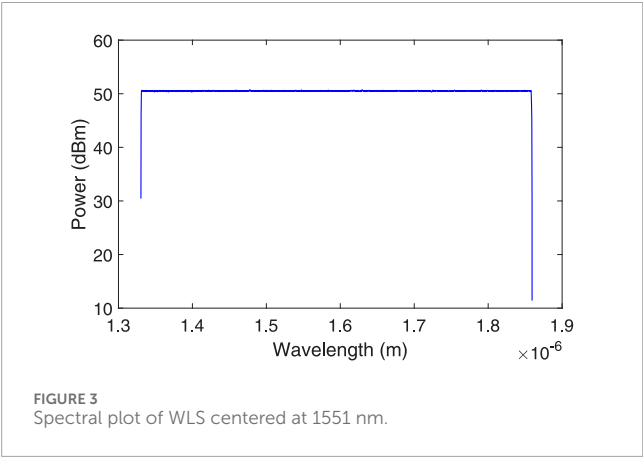
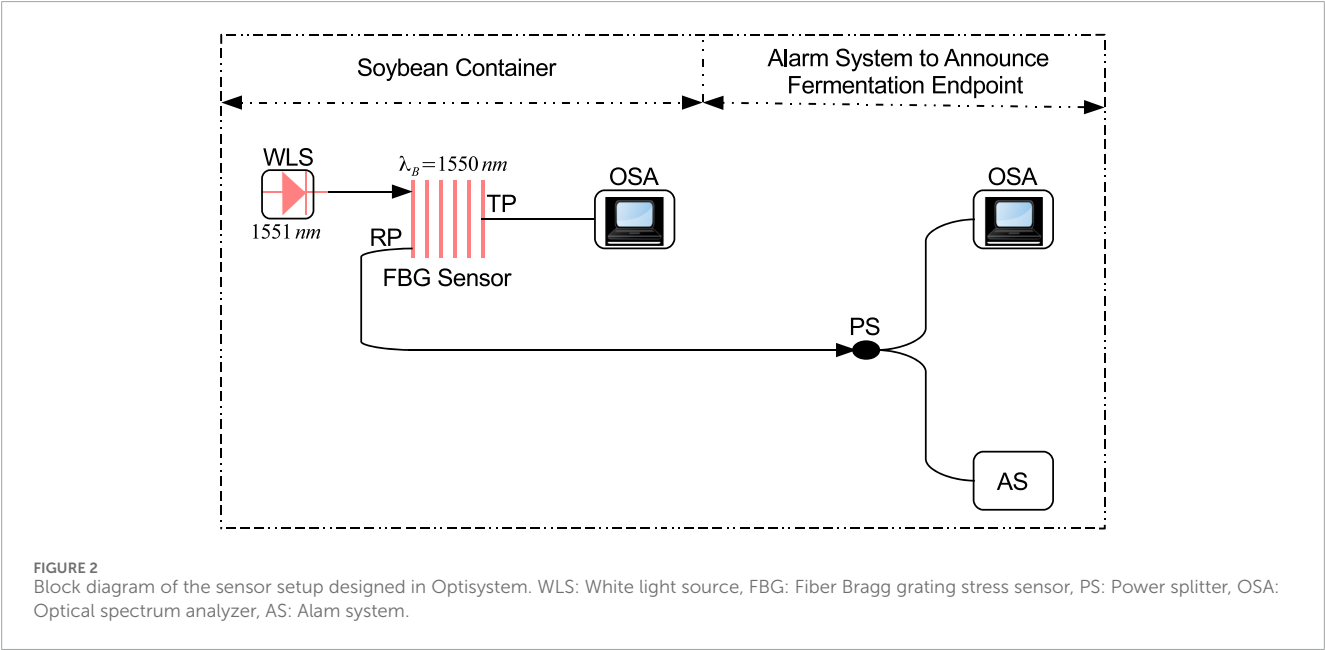
Figure 1 illustrates the fabrication of *Pd*-coated FBG sensors which involves three critical steps. First, the FBG is inscribed in the core of single-mode fiber (SMF) using a UV laser as shown in Figure 1a to create the periodic refractive index variation. Second, the fiber cladding is selectively etched using hydrofluoric acid to reduce the diameter exposing the core as shown in Figure 1b.



Finally, a uniform *Pd* layer of 50–200 nm thickness is deposited either through sputtering or electroless plating as illustrated in Figure 1c.

TABLE 2 Comparison of parameters for the analytical model and OptiSystem analysis of the Pd-coated FBG stress sensor.

Sr. No	Parameters	Numerical model	OptiSystem model
1	Initial Bragg wavelength	1,550 nm	1,550 nm
2	Core radius	4.6 μm	4.6 μm
3	Cladding radius	62.5 μm	62.5 μm
4	Thickness of Pd layer	100 nm	100 nm
5	Young's modulus of Pd	1,200 MPa	1,200 MPa
6	Poisson's ratio of Pd	0.39	0.2
7	Photoelastic constant of the Silica fiber	0.22	0.9



First of all, the reaction between Pd and H₂ to form PdH_x is represented by Equation 2 which is reversible equilibrium Equation 19.



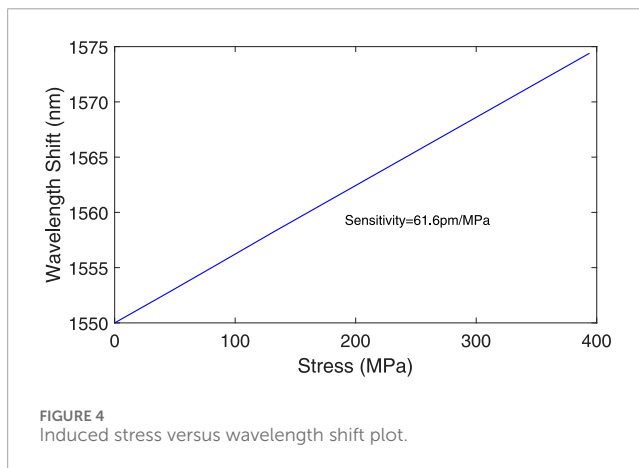
The absorption of H₂ in Pd occurs up to a maximum atomic ratio of $\frac{H}{Pd} = x = 0.65$ which represents the maximum saturation limit. This implies that while the total available amount of H₂ may be around 222.5 g, Pd can only absorb H₂ up to its saturation. Consequently, stress development in Pd layer is confined to the period during which it becomes fully saturated with H₂. Once saturation is achieved, any excess H₂ does not contribute to further stress development. It is also pertinent to mention that H₂ absorption can be controlled to obtain the required value of x either by reducing the H₂ exposure time or using the Pd alloy with lower absorption capacity or using a buffer layer to limit the Pd expansion.

3.2 Calculation of stress developed in Pd coating

To calculate the stress induced in Pd-coated FBG sensor, the following assumptions are crucial to consider.

TABLE 3 List of simulation parameters.

Sr. No	Parameters	Values
1	Operating wavelength of WLS	1,551 nm
2	Initial Bragg wavelength of FBG	1,550 nm
3	Power spectral density of WLS	−60 dBm/Hz
4	Effective index of FBG	1.45
5	Grating length	10 mm
6	Resolution bandwidth of OSA	0.1 nm
7	Sequence length	1,024 bits
8	Samples per bit	512



- Radii of the core and cladding are 4.6 μm and 62.5 μm , respectively.
- Thickness of *Pd* layer over FBG sensor is 100 nm.
- Thickness of adhesive layer of Titanium is 20 nm.
- Saturation limit of 0.1 is considered.
- The effect of temperature on Bragg wavelength shift is not considered in this research.

The strain induced in the *Pd* coating due to H_2 absorption is given by the relation.

$$\varepsilon_{PdH} = 0.2 \times x \quad (3)$$

In Equation 3, x is the atomic ratio of H to *Pd* and 0.2 is the empirical coefficient of expansion. At $x = 0.1$, the value of induced strain is $\varepsilon_{PdH} = 0.2 \times 0.1 = 0.02$. Therefore, the stress in *Pd*-coated FBG sensor due to H_2 absorption is given by the following equation.

$$\sigma_{PdH} = \frac{E_{Pd}}{1 - \nu_{Pd}} \times \varepsilon_{PdH} \quad (4)$$

In Equation 4, σ_{PdH} is the stress induced in *Pd* due to H_2 absorption, $E_{Pd} = 12 \times 10^9$ Pa is the Young's modulus of *Pd*, and $\nu_{Pd} = 0.39$ is the Poisson's ratio of *Pd*. Therefore, the value of stress at $x = 0.1$ is around 393.44 MPa.

3.3 Effect of stress on Bragg wavelength shift

The shift in the Bragg wavelength (λ_B) of *Pd*-coated FBG sensor due to axial strain after absorbing H_2 is given by the equation [20].

$$\Delta\lambda_B = \lambda_B (1 - P_e) \cdot \varepsilon \quad (5)$$

where P_e is the effective photoelastic constant of the fiber (≈ 0.22 for silica fibers), ε is the strain in the FBG sensor (≈ 0.02 at saturation), and λ_B is the initial Bragg wavelength (typically $\approx 1,550$ nm). Applying these values in Equation 5, the shift in Bragg wavelength ($\Delta\lambda_B$) is around 24.2 nm. This is the maximum shift in the Bragg wavelength, which corresponds to a stress of 393.44 MPa induced in FBG sensor when exposed to 222.5 g of H_2 that is released during the fermentation process.

The Bragg wavelength shift of 24.2 nm serves as a key indicator for detecting the fermentation endpoint in Soybean processing. This shift results from the stress-induced expansion of the *Pd* coating on the FBG sensor due to H_2 absorption and the subsequent formation of PdH_x . Since H_2 is a byproduct of soybean fermentation, the observed wavelength shift directly correlates with the completion of the fermentation process.

4 Design validation of *Pd*-coated FBG stress sensor using OptiSystem

The analytical model of the *Pd*-coated FBG stress sensor that is developed in the last section produces a Bragg wavelength shift of 24.2 nm is analyzed using OptiSystem simulation tool. Table 2 compares the parameters used in the analytical model with those employed in OptiSystem for design analysis. Using the parameters of the OptiSystem model, a shift of 24.5 nm in Bragg wavelength is achieved which is comparable to the analytical model making the assumption reasonable.

5 Simulation setup

Figure 2 shows the block diagram of the simulation setup designed in OptiSystem to detect the fermentation endpoint of Soybean. A white light source (WLS) with a power spectral density (PSD) and center wavelength of -60 dBm/Hz and 1,551 nm, respectively is used to illuminate the FBG stress sensor. Figure 3 illustrates the spectrum of WLS. The WLS model used in OptiSystem generates noise bins or sampled signals at the output according to the following mathematical expression.

$$\begin{bmatrix} E_x(t) \\ E_y(t) \end{bmatrix} = \begin{bmatrix} x_x(t) + jy_x(t) \\ x_y(t) + jy_y(t) \end{bmatrix} \cdot \sqrt{\frac{P}{4}} \quad (6)$$

In Equation 6, E is the optical field and P is average power. In the above equation, a Gaussian distribution has been assumed to describe the probability density function (PDF) for the real and imaginary parts of the optical field components E_x and E_y . A broadband light source is essential for FBG sensors because it provides a wide spectral range to accurately track the shifts in

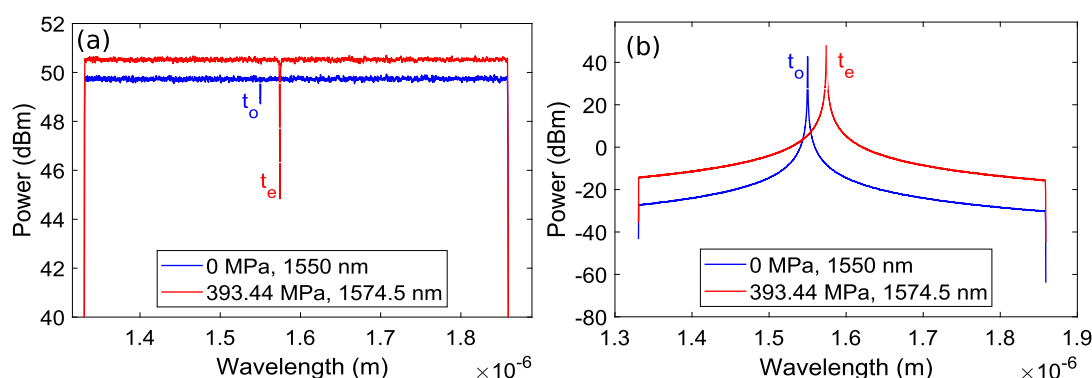


FIGURE 5
(a) Transmission spectra of FBG stress sensor (b) Reflection spectra of FBG stress sensor.

Bragg wavelength caused by external perturbations [21]. Unlike narrowband lasers that require fast scanning to avoid missing signal detection, WLS enables high-resolution detection of small wavelength changes which are critical for real-time monitoring in applications like *Pd*-coated FBG stress sensor [21]. Additionally, broadband light sources support multiplexing of multiple FBGs on a single fiber, making it ideal for scalable industrial systems. Its stable and noise-resistant output ensures reliable measurement of stress-induced shifts which are vital for precise fermentation endpoint detection [21]. In practical scenario, the *Pd*-coated FBG stress sensor will be placed inside the fermentation container holding 5 kg of Soybean with a suitable microbial culture to initiate the fermentation process. The temperature, humidity, and pressure inside the container is controlled to ensure optimal fermentation conditions. The *Pd* layer serves as the active sensing material, absorbing H_2 molecules released during fermentation. Upon absorption, *Pd* undergoes a phase transition to PdH_x , leading to volumetric expansion which induces mechanical strain inside the FBG sensor. This strain modifies the grating period of the FBG, causing a Bragg wavelength shift which serves as an indicator of the fermentation endpoint. In OptiSystem, this process is realized using the numerical values of stress calculated in Section 3 to create the corresponding shift in Bragg wavelength of stress sensor. The optical signal reflected from the FBG stress sensor at a shifted Bragg wavelength corresponding to the applied stress, is split into two parts using a 20:80 power splitter (PS) attached to the sensor's reflection port (RP). The 20% output of the PS is sent to an optical spectrum analyzer (OSA) for analysis of the results while the 80% output is connected to the alarm system (AS) for annunciation of the fermentation endpoint. Similarly, the transmitted spectrum is directed to another OSA via the transmission port (TP) for analysis. The important simulation parameters used in this work are described in Table 3.

6 Results and discussion

Figure 4 illustrates the induced stress due to formation of PdH_x after absorbing H_2 by *Pd*-coated FBG sensor versus wavelength shift. It is clear that wavelength sensitivity of 61.6 p.m./MPa

has been achieved. A linear relationship between the wavelength shift and induced stress can be observed. The reason of linear relationship between the wavelength shift and the stress shown in Figure 4 is attributed to the fundamental principle of opto-mechanical coupling as expressed in Equation 5. We acknowledge that practical scenarios may introduce noise and nonlinearities due to thermal fluctuations, mechanical vibrations, material hysteresis, and deformations in the *Pd* coating which can affect wavelength shift and resolution. However, the reported sensitivity threshold of 61.6 p.m./MPa sets a floor for the FBG interrogators. Figure 5a shows the transmission spectra of the FBG sensor for stress values of 0 MPa and 393.44 MPa obtained by connecting the OSA to TP of FBG stress sensor as illustrated in Figure 2. Similarly, Figure 5b shows the reflection spectra of FBG the sensor for stress values of 0 MPa and 393.44 MPa indicating the onset and endpoint of fermentation process, respectively. Reflection spectra is obtained by connecting the OSA with 20% output of the PS, that is connected with RP of the FBG stress sensor as shown in Figure 2. Assuming the fermentation starts at time t_o , the transmission dip and the reflection peak of the transmitted and reflected optical signals, respectively equal to the initial Bragg wavelength of *Pd*-coated FBG sensor which is 1,550 nm, as shown in Figure 5 corresponding to the absence of detectable H_2 . As fermentation process progresses, microbial activity produces H_2 which is absorbed by the *Pd* coating forming PdH_x . This induces stress in the *Pd*-coated FBG stress sensor, linearly shifting the Bragg wavelength of reflected optical signal. Similarly, the fermentation endpoint is achieved at time t_e . Consequently, the absorption of H_2 in *Pd* coating saturates and any excess H_2 will not contribute to further stress development. The transmission dip and reflection peak of transmitted and reflected optical signals equal to 1,574.5 nm. A maximum shift of 24.5 nm in Bragg wavelength corresponding to a stress of 393.44 MPa is induced in FBG sensor indicating the fermentation endpoint.

7 Conclusion

In this study, we demonstrated a non-destructive and real-time method for detecting the fermentation endpoint of Soybeans using a *Pd*-coated fiber Bragg grating stress sensor. The method

relies on monitoring the shift in the Bragg wavelength, which is caused by the stress induced in the *Pd*-coated FBG sensor due to the volume expansion of the Palladium layer upon the formation of Palladium Hydride after absorbing Hydrogen gas released as a byproduct during the fermentation of Soybeans. The *Pd*-coated fiber Bragg grating sensor was analytically designed and its performance was analyzed using the OptiSystem simulation tool, achieving a wavelength sensitivity of 61.6 p.m./MPa. The results demonstrate that the proposed method provides a reliable, straightforward, and efficient solution for real-time monitoring and detection of the fermentation endpoint of various organic compounds that release Hydrogen gas as a byproduct. This approach holds significant potential for applications in food processing, biotechnology, and industrial fermentation processes.

Data availability statement

The raw data supporting the conclusions of this article will be made available by the authors, without undue reservation.

Author contributions

JM: Conceptualization, Project administration, Software, Validation, Writing – original draft, Writing – review and editing. AA: Methodology, Writing – review and editing. BK: Conceptualization, Investigation, Writing – original draft. FK: Investigation, Methodology, Software, Writing – original draft. IA: Funding acquisition, Resources, Software, Writing – original draft, Writing – review and editing. AA: Resources, Software, Writing – original draft.

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Conflict of interest

Author AA was employed by Optiwave Systems Inc.

The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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