In-situ temperature monitorization in oscillatory flow crystallizer using optical fiber sensors with a Bragg grating inscribed at the fiber tips ends

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Abstract. Optical fiber sensors were implemented to measure *in-situ* temperature variations in an oscillatory flow crystallizer operating in continuous. The sensors were fabricated by cleaved in the middle 8 mm-length fiber Bragg gratings, forming tips with a Bragg grating of 4 mm inscribed at the fiber ends. The geometry of the sensors fabricated, with a diameter of 125 µm, allowed the temperature monitorization of the process flow, inside the crystallizer, at four different points: input, two intermediate points, and output. The results revealed that the proposed technology allows to perform an *in-situ* and in line temperature monitorization, during all the crystallization process, as an alternative to more expensive and complex technology.

1 Introduction

Crystallization is a very important process in the pharmaceutical industry because it is responsible for the manufacturing of 90% of its products, the Active Pharmaceutical Ingredients (APIs) [1]. This process can occur in crystallizers with different geometries and sizes, such as the Oscillatory Flow Crystallizer (OFC).

The OFC is a tubular crystallizer containing spaced orifice baffles, which are transversely assembled to a periodically oscillating flow. In the past decades, the development of this type of OFC has played a significant role in the pharmaceutical industry because it can operate in a continuous way, allowing the manufacturing of APIs with higher purity. However, due to the diameter of the OFC tubes/channels, the monitorization of the crystallization process is hampered. Typically, the probes used in other geometries of crystallizers exceed the OFC channels diameter. One of the most important process parameters that need to be control during the crystallization is the temperature of the process flow.

In view of the need to monitor the temperature *in-situ* and in crystallizers such as OFC, in this work, a technology compatible with the dimensions of the OFC channels was developed. Four optical fiber sensors, based on Bragg gratings (FBGs) inscribed at the fiber tips end, were applied to measure the temperature of the OFC process flow in four different sections of the crystallizer.

2 Results

2.1. Interrogation system and sensing probes

The sensing probes proposed consists of 8 mm-length FBG sections that were cleaved in the middle, forming fiber tips with FBGs of 4 mm-length inscribed at its ends. The sensing probes were interrogated in reflection through a portable interrogation system and its responses were recorded by a data acquisition software (BraggMONITOR SI) – Figure 1.

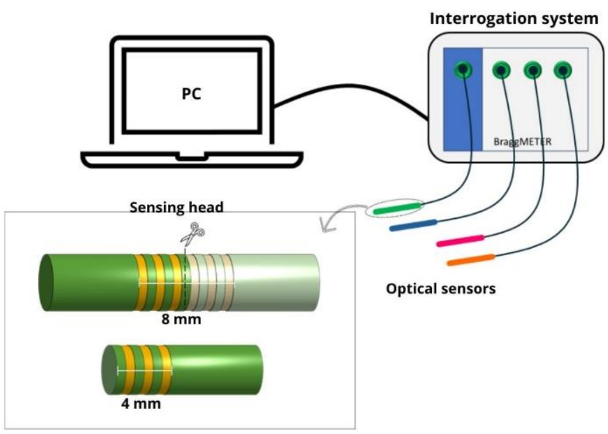

Fig. 1. Interrogation system and the detail of the sensing heads.

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As previous referred, the sensing heads was fabricated using FBGs, three of them were centered at 1554 nm and the other one was centered at 1562 nm.

After the cleavage, the output spectrum of each sensor proposed maintains a maximum output power level near of these values (1554 nm and 1562 nm). Regarding to the characteristics of FBGs, it is expected that, during variations of temperature, the response of each proposed sensor shows a wavelength dependence on temperature, which is caused by the thermo-optical effect that changes the material effective refractive index [2].

2.2. Calibration curves

The influence of temperature in the output spectrum of each sensor was analysed. For this, each sensing head were immersed in a temperature-controlled water bath (~10°C to 60°C) and the wavelength dependence on temperature was determined, through the sensor's response. As expected, for all the sensing heads, a linear approximation of the obtained calibration was carried out, with correlations factors equals to 0.999. The data of this approximation are given in Table 1.

Table 1. Wavelength dependence with temperature variations. Sensor's calibration: parameters obtained by a linear approximation ($y = mx + b$).

Sensing head	m	b	R^2
(1)	9.20×10^{-3}	1554.35	0.999
(2)	9.22×10^{-3}	1662.01	0.999
(3)	9.09×10^{-3}	1554.32	0.999
(4)	9.12×10^{-3}	1554.42	0.999

2.3 In situ temperature monitoring

The temperature monitorization was performed in four different channels of the OFC, as it is showed in Figure 2: (1) input, (2) e (3) intermediates and (4) output channels. The sensing heads were vertically immersed in each channel, according to its numeration (see Table 1).

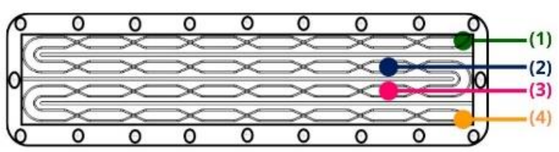

Fig. 2. Experimental setup for *in-situ* temperature monitorization in oscillatory flow crystallizer.

The OFC was filled with water at 15ºC and thermostated at 35ºC. The water was fed into the OFC at 58 mL/min and the frequency, and the amplitude of oscillation were set at 4 Hz and 9 mm. During the monitorization in *in-situ*, the sensor response was acquired and then, through the previous calibration performed, the temperature in each OFC channel was determined – Figure 3.

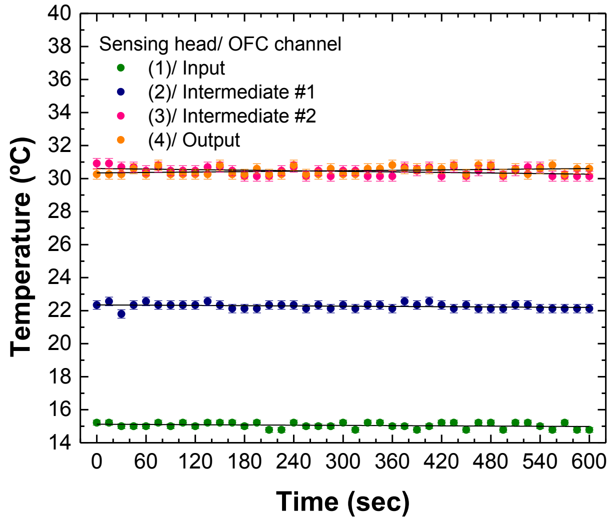

Fig. 3. *In-situ* temperature monitorization in oscillatory flow crystallizer.

The results obtained (Figure 3) were the expected. Considering that the OFC was operating in continuous, and the water was fed into the OFC at 15ºC, and it was thermostated at 35ºC, it was expected at the input channel a temperature higher than 15ºC and lower than 35ºC. Also, it was expected a higher temperature variation between the input channel and the first intermediate channel, and a lower temperature variation between the second intermediate channel and the output channel, as it was obtained.

3 Conclusions

In this work a portable interrogation system was used to monitor *in-situ* the temperature of process flow in an OFC. It was used optical fiber sensors, based on FBGs inscribed at the fiber tips ends. The gratings wavelength dependence on temperature allow to measure the temperature in specific points/channels of the OFC, without disturbing the process flow. In this way, the technology proposed can be applied in OFC systems and other crystallizers with different types of geometries due to the reduced dimensions of the fiber, suppressing the need pointed out in this sense.

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