

Article

A Novel Optical Instrument for On-Line Measurement of Particle Size Distribution—Application to Clean Coal Technologies

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Abstract: A flow cell is a critical measurement interface for many optical instruments. However, the flows are often sampled under harsh conditions, such as under high pressure and/or high temperature, in the presence of particles, moisture, vapors with high dew points or corrosive gases. Therefore, obtaining a high-optical-quality flow cell that does not perturb the measurement is a significant challenge. To address this challenge, we proposed a new flow cell that employs a unique laminar coaxial flow field (for the purge and sample flows). A test system was built to conduct particle size distribution (PSD) measurements with no sampling bias using a state-of-the-art analyzer (Malvern Panalytical Insittec). The results revealed that the measurement zone is well defined solely by the sample flow, and the optical windows are well protected by the purge flow, with minimal risk of any depositions from the sample flow. Using this flow cell, the Insittec can successfully measure PSD under high pressure and temperature under moist, corrosive conditions without generating any sampling bias. Importantly, we successfully applied this flow cell for on-line PSD measurement for the flue gas of a 100 kW_{th} pressurized oxy-coal combustor operating at 15 bara.

Keywords: flow cell; laminar; coaxial flow field; sample flow; purge flow; particle size distribution; optical instrument; on-line measurement; high pressure; pressurized oxy-combustion



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1. Introduction

Optical instruments have been widely used for measurements associated with molecules, particles, and droplets. For example, Fourier-Transform Infrared Spectroscopy (FTIR) can be used to measure the composition and concentrations of gases that absorb in the infrared [1], and light scattering can be used to measure the size distributions and concentrations of particles and/or droplets [2]. Optical instruments typically consist of an emitter, one or more detectors, and often employ a flow cell. The flow cell isolates the sample flow from the emitter and detectors and incorporates windows to allow for an optical beam path between them [3,4]. A well-designed flow cell minimizes sampling bias and enables continuous, accurate on-line measurements.

In this work, we develop an approach to on-line measurement of particle size distribution (PSD) utilizing a state-of-the-art Malvern Panalytical Insittec optical instrument (Malvern, UK). The instrument employs light scattering from small particles and Mie theory to determine the PSD across a wide range (0.1–1000 µm) [5]. Accurate measurement of PSD in flue gas is essential for the development of clean coal technologies. These data are crucial to enable precise estimation of radiation heat transfer in the boiler sections and

for addressing ash deposition and the design of particle removal systems. Furthermore, on-line PSD measurements can aid in diagnosing the combustion system.

Washington University in St. Louis has been developing a Staged, Pressurized Oxy-Combustion (SPOC) technology, which can achieve near-zero or carbon-negative emissions for next-generation power plants [6,7]. A process flow diagram for SPOC is shown in Figure 1. The process can operate with gaseous, liquid or solid fuels and, unlike traditional oxy-combustion technologies, the system operates under pressure and with low quantities of flue-gas recycle. Unique to the SPOC process, the boilers are arranged in a series-parallel configuration. This innovation allows for a substantial reduction in flue gas recirculation (FGR) which increases the plant efficiency. Downstream of the pressurized boilers, the flue gas stream is fed into a high-pressure heat recovery unit (Figure 1), where heat is extracted and integrated into the steam cycle. Fly ash particles in the flue gas are removed by a particulate filter. The flue gas is further cooled in a direct contact cooler (DCC) column, where the flue gas flows against a stream of water. The flue gas moisture condenses and, since the pressure is elevated, the condensed water leaving the bottom of the DCC is at a sufficiently high temperature that the heat can be integrated into the steam cycle to preheat the boiler feedwater (i.e., the latent heat is recovered). This step, which can only be accomplished at elevated pressure, is a key to improving boiler thermal efficiency. An additional advantage of pressure is that sulfur- and nitrogen-containing species and hydrogen chloride in the flue gas are dissolved in the water and thereby removed in the DCC, dramatically simplifying flue gas cleanup and reducing capital costs [8]. The water leaving the DCC is neutralized by, for example, adding caustic, therefore minimizing acid corrosion. The process of simultaneous latent heat recovery and emission removal, both of which are only effective under pressure, are key advantages of the SPOC process.

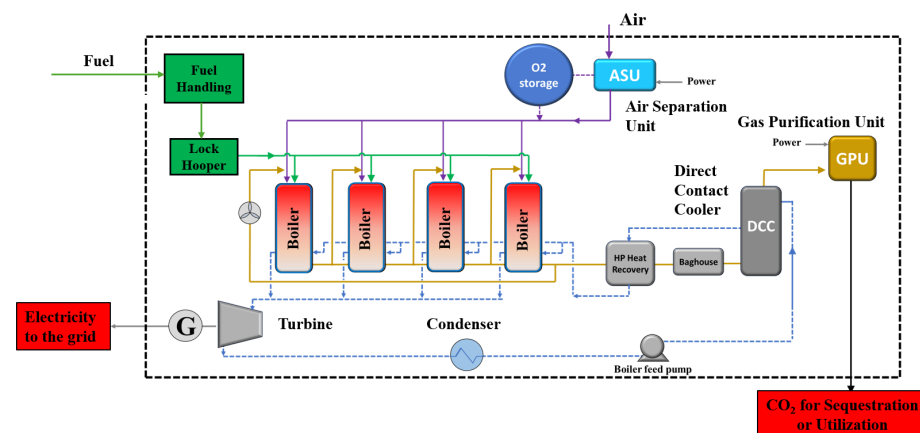


Figure 1. SPOC process flow diagram.

The sample flow from the SPOC system is moist flue gas at high pressure and elevated temperature, and the flow contains particles and corrosive acid gases with a high dew point. The sampling locations can be at the bottom of the combustor, the outlet of the boiler, the outlet of the heat recovery unit, or the outlet of the DCC. The high pressure and high sulfur and nitrogen content in the sample flows result in harsh conditions for the on-line measurements. It is noted that many other industrial applications also face similar harsh sampling conditions. Most aerosol instruments such as the scanning mobility particle sizer (SMPS), electric low-pressure impactor (ELPI) or optical particle sizer (OPS) are designed to work under atmospheric conditions, making real-time aerosol measurements under pressure quite challenging [9]. Most measurements of particles and gases from pressurized systems are made by reducing the sampling pressure to atmospheric pressure so that the sample gas is compatible with the analyzer [9,10]. However, depressurizing the sample

can cause sample bias due to selective particle loss during the gas expansion process and condensation. Additionally, sample dilution is sometimes employed to avoid condensation, but this decreases the signal-to-noise ratio of the instrument. Furthermore, decreasing pressure may change the particle agglomeration dynamics of an aerosol.

Ideally, the sampling pressure would be maintained when conducting particle measurements. However, at high pressure, the moist and corrosive flue gas can condense at a high temperature. Thus, it is important to control the flow cell temperature to avoid condensates. Achieving a flow cell with high optical quality that does not perturb the measurement under these conditions is a challenge, and this is the goal of this study.

There are a limited number of commercially available flow cells that provide optical access [11]. One example is Malvern's commercial flow cell for the Insittec analyzer. However, this cell is not designed for high temperature and/or high-pressure conditions [12]. Additionally, this flow cell uses a large flowrate of purge gas to protect the optical windows, shielding them from the sample flow to avoid particle deposition or condensation [12]. The large purge flow results in strong mixing with the sample flow, creating a complex flow field in the flow cell. This design can lead to (1) significant dilution of the sample flow, which may bias the sample and/or limit the measurement at low particle concentrations, (2) a risk of particle deposition on the window [11], necessitating regular cleaning and (3) significant uncertainty in measuring particle concentration. To prevent condensation and temperature gradients that disturb the optical beams, heating the purge flow may be necessary, which is difficult to achieve with Malvern's commercial flow cell which is connected to the Insittec. To address these challenges, a new design for an optical flow cell is proposed in this work, introduced in Section 2. A test system was built to validate this flow cell design with the Insittec analyzer, as described in Section 3. The testing results and discussions are also presented in Section 3. In Section 4, we utilize the flow cell for on-line PSD measurements of flue gas at 15 bara from a 100 kW_{th} pressurized oxy-coal combustor.

2. Optical Flow Cell Design

2.1. Light Beam Steering (Schlieren Effect) Analysis

One of the biggest challenges in designing and operating an optical flow cell is minimizing beam steering (Schlieren effect) caused by refractive index gradients. Figure 2 illustrates beam steering due to a temperature gradient, where the refractive index of the cold zone is higher. The direction of the refractive index gradient that causes beam steering is perpendicular to the optical axis. The deflection of the beam away from the optical axis (δ) is proportional to the gradient in the refractive index (dn/dx) and the span of the disturbance in the direction of the optical axis (L) [13], as shown in Equation (1).

$$\delta = L \cdot dn/dx \quad (1)$$

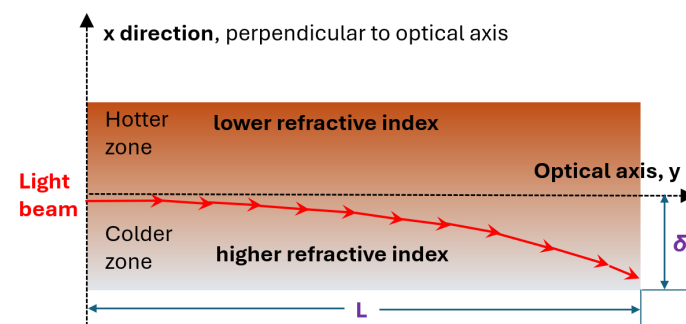


Figure 2. Beam steering due to a temperature gradient.

The relationship between refractive index (n) and gas density (ρ) is given by

$$n = k\rho + 1 \quad (2)$$

where k is the Gladstone–Dale coefficient and is nearly constant over most of the visible spectrum [13]. Using Equations (1) and (2) and the ideal gas equation, δ can be obtained from

$$\delta = -kL \cdot (\rho/T) \cdot (dT/dx) \quad (3)$$

Equation (3) shows that temperature gradients (dT/dx) can cause beam steering, but more importantly, that high pressure and a long disturbance span can exacerbate beam steering. Therefore, it is critical to minimize temperature gradients across the optical axis and the disturbance span under high-pressure conditions.

2.2. Design

The flow cell that was designed to address these issues is shown in Figure 3. This design creates a steady, smooth coaxial flow field, particularly in the optical beam zone. The flow field consists of steady, coaxial laminar flows of purge gas and sampling gas, both having similar velocities. Since there is minimal mixing between the two streams, the measurement zone is the intersection between the sample flow and the optical beam. By using the laminar coaxial flow field, the measurement zone is well defined, without any influence from or mixing with the purge flow. At the same time, the optical windows, which are mounted on the wall of the middle pipe, are well protected because of the purge flow, mitigating any risk of deposition or condensation from the sample flow. The supply tube that contains the sample flow guides the sample flow to a point just above the measurement zone, at which point the flow merges with the purge flow. Moreover, by preheating the purge flow and heat tracing the flow cell and sample flow, this design minimizes temperature gradients inside of the flow cell and avoids condensation. The only temperature gradients that exist are at the inner window and these are in the direction of the beam, so they do not cause beam steering. By using this unique design, the flow cell can handle high pressure, moist sampling conditions with corrosive gas, and/or vapors with high dew point. The smooth coaxial flow field also enables a short length of the optical beam zone inside the flow cell, which minimizes the disturbance span if there were beam steering. It is noted that in addition to applications for particle analyzers, this flow cell can also be applied to optical gas analyzers, such as FTIR.

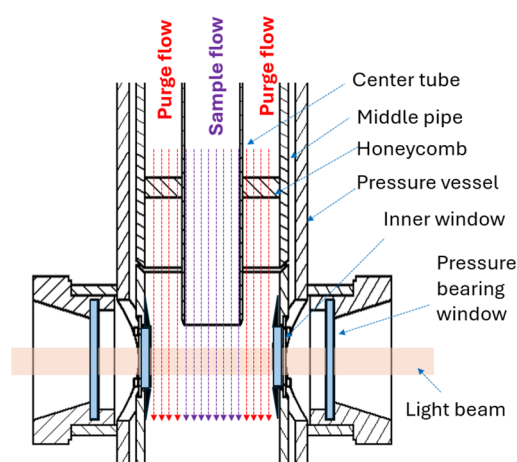


Figure 3. Basic design of the optical flow cell for use with an optical instrument.

To enable the flow cell to work under high pressure, the coaxial vertical flow channels of the sampling and purge flows are contained within a pressure vessel, allowing measurements to be conducted under high pressure and temperature. The outer optical windows are mounted on the wall of the pressure vessel and are rated for operating pressure and temperature. The inner windows, while not pressure sealed, are able to perform at the operating temperature of the flow cell. Both the inner windows and outer windows are aligned with the optical beam zone. The clear aperture sizes of the inner windows and outer windows are sufficiently large to allow all the signals from the measurement zone that need to be measured by the optical detector to pass through. The selected optical windows minimize signal loss and noise. The light beam is aligned with the cross-section zone of the sample flow, making sure the beam is located in the center of the zone, by simply using a fixture with a hole having the same diameter as the light beam, which is attached to the inner windows. It should be noted that while the design of the flow cell targets pressurized conditions, the flow cell can also be employed in less harsh conditions, such as normal pressure, at a wide range of temperatures. Depending on the operational conditions and the laser beam wavelength, a wide range of optical windows such as N-BK7, sapphire, quartz, or borosilicate can be used for the inner and outer windows.

The manufacturing cost of this flow cell is low due to its simple design. The main material of construction of the flow cell is stainless steel. For a high-pressure and high-temperature design, the most expensive component is the outer windows, which must handle the pressure and temperature and ensure high optical quality. The cost of each outer window will decrease as demand increases.

3. Performance Test

3.1. Malvern Panalytical Insitac Analyzer

The Insitac analyzer has been widely used for on-line measurements of PSD in the range of 0.1–1000 μm [12]. As particles pass through the laser beam, light that is scattered in the forward direction is collected by a Fourier lens and focused onto a detector array located at the focus plane of the lens, as shown in Figure 4a. The Fourier lens is a standard converging lens, which allows optical Fourier transformation. Based on Mie theory, each particle size has its own characteristic scattering pattern (Figure 4b) [5]. To measure the scattering pattern, the analyzer employs a detector array consisting of 32 individual co-annular ring detectors, each of which measures the light scattered in a defined range of forward angles [14,15]. From the smallest to the largest angle, the area of a ring detector increases exponentially by about 3 orders of magnitude to capture the weak signals at larger angles [14,15]. From the raw scattering data (excluding the data of the two innermost detectors, Detector-1 and -2), the particle size distribution can be derived [12,14]. Light that is not scattered is focused so that it passes through a 200-micron diameter pinhole in the center of the detector array and is measured by the beam power detector (Detector-0) to provide a light transmission reading [12]. The RTSizer 7.40 software, which serves as the interface for the Insitac analyzer, allows the refractive index of the particles to be input, which is particularly important for small particles, whose scattering is more strongly affected by refractive index [15].

Particles may pass anywhere along the length of the exposed laser beam [11]. Due to the Fourier lens, the collected scattered beams for a given angle can be focused on the same annular detector ring, regardless of the positions of the particles in the laser beam zone. However, it is necessary to observe certain limits on the maximum distance between the light-scattering particles and the Fourier lens [15]. This maximum distance is a function of the focal distance and lens diameter. Exceeding this distance results in vignetting of the

signal on the outer detector rings, skewing the calculated size distribution towards larger particles [15].

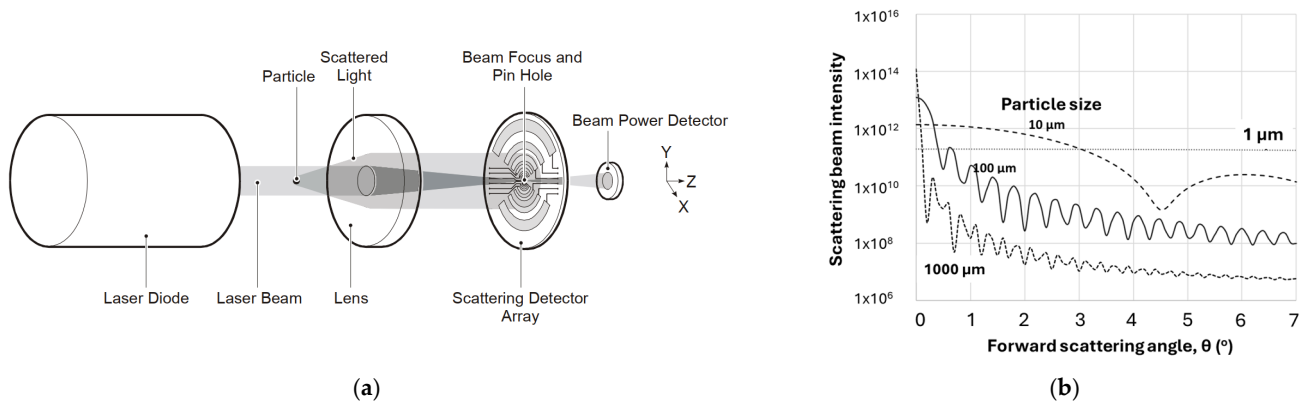


Figure 4. (a) The basic design of Malvern Insittec [12] and (b) the light scattering distribution at different particle sizes based on Mie theory [5].

3.2. Test System

Figure 5 shows the test system for the flow cell. The Insittec emitter module (right) and receiver module (left) are firmly attached to the Insittec open frame which aligns the two modules. There are also fine adjustment screws inside the receiver module to ensure that the laser beam passes through the pinhole in the center of the detector array [12]. The Insittec open frame and the flow cell are mounted on a metal stand, as shown in Figure 5, with several adjustments to allow for alignment of the Insittec and flow cell.

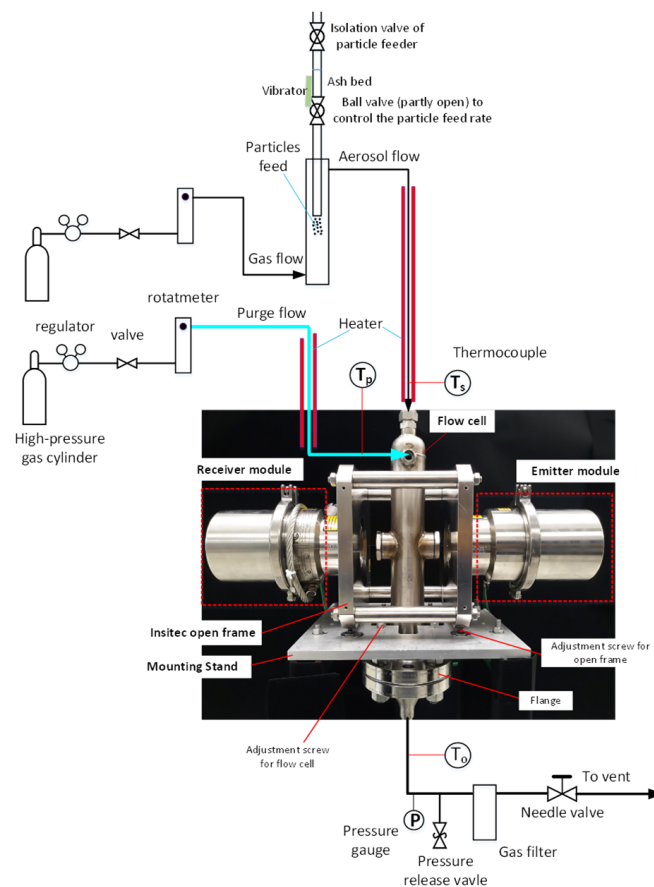


Figure 5. Test system for the flow cell with the Insittec.

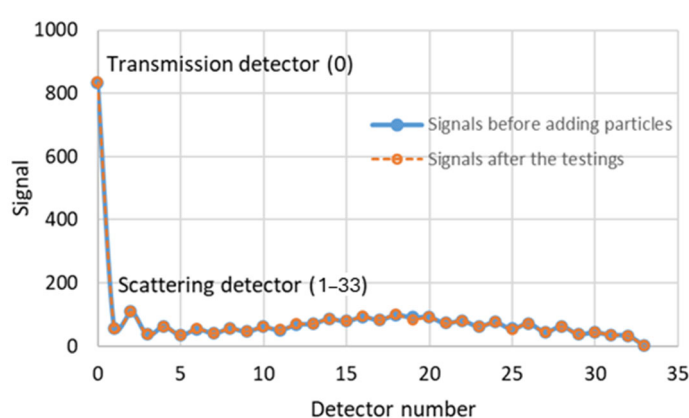
A particle feeding system supplies an aerosol stream for testing the flow cell at high pressure (15 bara) and temperature (200 °C). By using a partially open ball valve with a vibrator, a small number of particles can be continuously added to the sampling gas line and carried by the gas flow, and the flowrate of the sampling gas is controlled to uniformly transport particles into the flow cell. A similar gas control system is used to supply the purge flow. The temperatures of both the sample flow and the purge flow are controlled by heating tapes on the lines connected to the flow cell, and the lines are insulated. The space between the middle pipe and the pressure vessel of the flow cell has no flow through it, and acts to reduce heat loss. Therefore, we can assume that the temperatures in the measurement zone will be nearly equal to the temperatures of inlet gas flows. The pressure in the flow cell is controlled by a needle valve downstream. Table 1 shows the main operating conditions of the test system. The particles used were sieved solid particles, with a median sieve size of about 54 μm . It should be noted that based on the simple fundamentals of this flow cell, the flow cell is not restricted to the range of parameter space given in Table 1. This should only be considered as an example.

Table 1. Operating conditions of the test system for the flow cell.

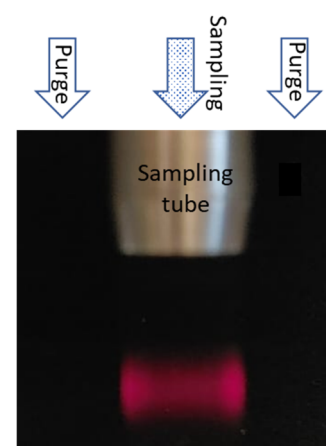
Sampling gas flowrate (SLPM)	3
Purge gas flowrate (SLPM)	12
Cross-section area of sample flow (m^2)	0.00023
Cross-section area of purge flow (m^2)	0.00104
Sample velocity in the center tube (m/s)	0.023
Purge velocity in the middle pipe (m/s)	0.021
Sampling gas temperature at the inlet of the flow cell, T_s (°C)	200
Purge gas temperature at the inlet of the flow cell, T_p (°C)	200
Flow cell operation pressure (bara)	15

3.3. Test Results and Discussions

The background scattering and transmission are measured with the Insittec before introducing particles and then again after running with particles for more than 100 h (Figure 6a). No change was observed, verifying that no particle deposition occurred on the windows when running with particles.



(a)



(b)

Figure 6. (a) Typical background scattering and transmission signals from the Insittec before and after particle testing for more than 100 h, and (b) the clearly defined particle measurement zone (red scattered light) below the outlet of the sampling tube in the flow cell.

The measurement zone is defined by the sample flow in the flow cell, as seen in Figure 6b. The pathlength of the observed measurement zone is equal to the inner diameter of the sampling tube, and there is no light scattering outside of this measurement zone. Based on this, the pathlength can be easily changed by replacing the sampling tube with one of a different inner diameter, which would enable the Insittec to measure particles over a wide range of concentrations. For a short pathlength, the Insittec can measure particles at high concentrations, while for a long pathlength, it can measure particles at low concentrations. Furthermore, the clearly defined pathlength of the measurement zone is important to accurately measure the particle concentration. The calculated particle concentration delivered by the particle feeder is around 93 ppmv based on the particle mass feed rate and the sampling gas flow rate, and the measured results from the Insittec is 94 ppmv. This indicates that there is no dilution of purge gas in the measurement zone. Thus, this flow cell design addresses the critical problems of window fouling and sampling bias.

The design of the flow cell is also important to accurately measure the PSD. The aperture size of the windows must be sufficiently large to allow the passage of all of the relevant scattered light, and beam distortions due to the windows must be minimized. Figure 7a,b shows the PSDs obtained with and without the flow cell for a wide range of particle sizes (1–300 μm for sieved solid particles and 0.1–100 μm for water droplets via a nebulizer). The results demonstrate that the flow cell does not introduce measurement bias. Furthermore, the measured median size of the sieved solid particles (54 μm) from the Insittec agrees with a median size based on sieve size. It is noted that the purge and sample flows do not influence the measurement accuracy of the PSD, based on Section 3.1, and thus an analysis of optimized flow is not included here.

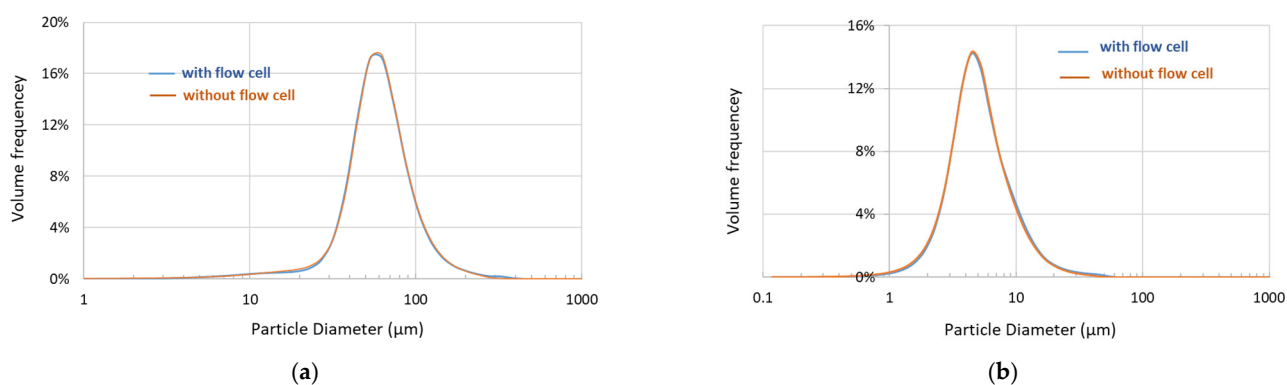


Figure 7. Particle size distributions of (a) sieved solid particles (sieve medium size is 54 μm), and (b) the droplets from a nebulizer measured by a Malvern Insittec with and without a flow cell under normal pressure and temperature.

One of the greatest challenges for optical measurements under high pressure and temperature is the Schlieren effect. As shown in Figure 8, with this flow cell the Insittec can successfully measure the PSD under a wide range of pressures and temperatures. The measured PSD under 200 $^{\circ}\text{C}$ and 15 bara is shifted slightly to the right, which is likely due to particle agglomeration under high pressure. Thus, the Schlieren effect, which would typically distort the scattering angle and thus the measurement accuracy, is minimized with no detectable measurement bias.

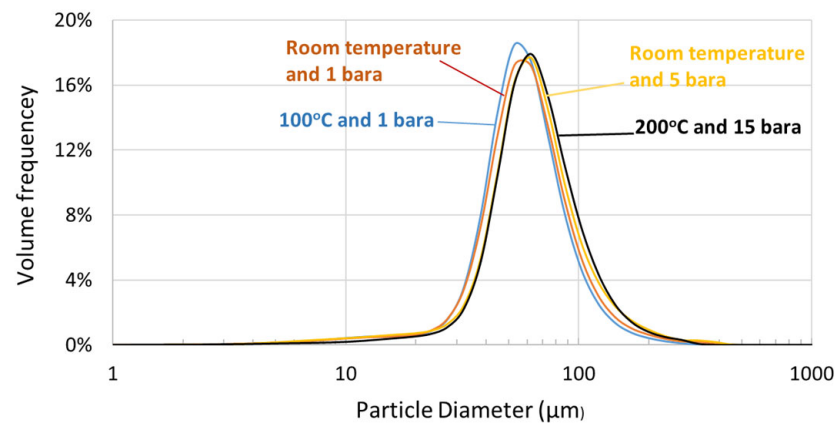


Figure 8. The measured particle size distribution in the flow cell under various conditions by the Insitec.

4. Application: On-Line PSD Measurements in Flue Gas Under High Pressure from a Pressurized Oxy-Coal Combustor

4.1. On-Line PSD Measurement System

Figure 9 shows the on-line PSD measurement system used for a 100 kW_{th} pressurized oxy-coal combustor. A detailed introduction to this combustor system can be found in [7]. The mounting and alignment of the flow cell and the Insitec are identical to the test system shown in Figure 5. However, in this setup, the flow cell purge is regulated by a mass flow controller to ensure a more stable flow, typically ranging from 5–10 SLPM, during measurement. The purge gas velocity is varied between 0.0086 and 0.017 m/s. The isokinetic flowrate of sampling, originating from a sampling probe located at the combustor bottom, is adjusted using a needle valve downstream of the flow cell. To prevent any vapor condensation in the flow cell and on the tube walls, the temperatures of the purge line, sampling line, flow cell body, and exhausted line are controlled within the range of 200–300 °C. The flow cell pressure is maintained equal to the sampling pressure (15 bara). By maintaining stable flows for both purge and sample, along with stable temperatures and pressure, a uniform temperature profile is established within the beam zone inside the flow cell. This enables the acquisition of a constant scattering background with minimal Schlieren effect, which is critical for obtaining accurate on-line PSD measurements.

A high-pressure, high-temperature filter is installed in the exhaust line to capture the particles and ensure proper functioning of the needle valve. Additionally, a rotameter is incorporated into the exhaust line to monitor the total flowrate from the flow cell. By subtracting the purge flowrate from the total flowrate, the sample flowrate can be determined. A cooler and a desiccant dryer are employed to remove the water vapor, ensuring the rotameter functions properly without interference from the condensate.

When sampling and measurement are not taking place, a sampling line purge flow consisting of CO₂, typically ranging from 10 to 20 SLPM, is directed into the sampling line. This purge flow prevents flow from the combustor from entering the flow cell. A split stream derived from the sampling line purge flow, and equal to the sample flowrate during measurement, flows into the flow cell. This arrangement maintains a smooth coaxial flow field and a uniform temperature profile within the flow cell when sampling and measurement are not occurring, thereby ensuring the stability and consistency of the performance of the flow cell.

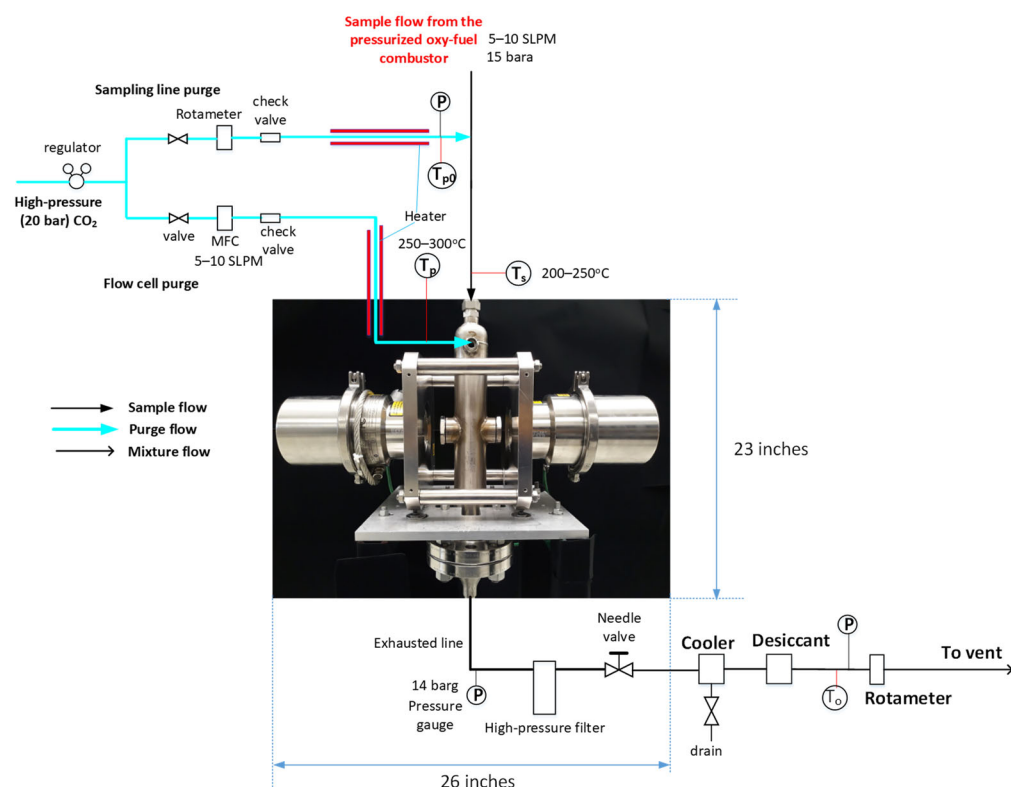


Figure 9. The on-line PSD measurement system for a pressurized oxy-coal combustor.

4.2. Results and Discussion

The PSD in the sampled flue gas of the pressurized oxy-coal combustor is presented in Figure 10. The PSD of PRB (Powder River Basin) coal, used as the fuel, is also measured by the Insitex and presented in Figure 10 as a reference. The PSD in the flue gas exhibits a bimodal pattern, characterized by two prominent volume frequency peaks observed at approximately 14 and 74 μm . The small particles correspond to the fly ash that is generated from coal combustion. These particles are typically lighter and more easily transported by the flue gas. On the other hand, the large particles could be attributed to bottom ash or to the agglomeration of smaller particles under conditions of high pressure and high temperature. The medium size of the PRB coal is about 70 μm , while the median size of particles in flue gas is only around 15 μm .

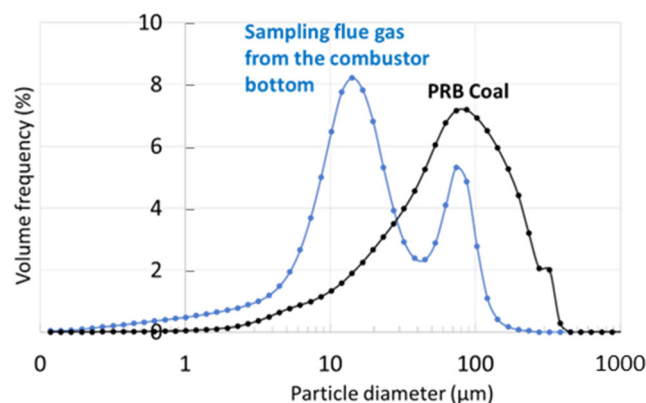


Figure 10. The PSD in the sampled flue gas under 15 bara from the outlet of a 100 kW_{th} pressurized oxy-coal combustor and the PSD of the PRB coal used as the fuel.

During this on-line measurement, the beam transmission was found to be around 97–99%, indicating a low particle concentration in the sampling gas. This low particle concentration can be attributed to the ash particles adhering to the walls and slagging in the combustor. As a result, the intensity of scattering pattern signals is limited to the range of 0–66 in this measurement, whereas the maximum scattering intensity measurement limit of the Insitac can reach up to 2000. Low scattering signals are common in various industrial processes where the particle concentrations in the flow stream are low. It is important to avoid dilution of the sample gas to maintain a sufficiently high signal-to-noise ratio for accurate PSD measurements and this flow cell accomplishes that. In comparison, to protect the flow cell windows in the commercial Malvern flow cell, a purge flow is used which is at least 47 SLPM higher flow than the sample flow. This can potentially dilute the sample gas, reduce the signal-to-noise ratio, and make measurements impossible in low-signal scenarios. In contrast, the smooth coaxial flow field provided by this new flow cell avoids dilution from the purge flow. Furthermore, the smooth coaxial flow field is less prone to enhancing the Schlieren effect under high pressure and temperature conditions. This allows the Insitac with the new flow cell to be applicable across a wide range of conditions. It can be used for on-line PSD measurements in various applications, including conventional coal power plants, next-generation carbon capture processes, gas–solid catalytic processes, and particle materials production processes under high pressure and temperature. Therefore, it will be important to conduct testing of this flow cell for measurement of on-line PSD by using the flow cell in different applications, which may have different particle types and PSDs and more aggressive operating conditions. We intend to conduct testing on the durability of the flow cell, the wear resistance of the optical windows and the stability of its operation under long-term industrial conditions.

5. Conclusions

In this work, we present a novel optical flow cell design that can be effectively utilized with advanced optical instruments. To showcase its capabilities, we implemented the flow cell in conjunction with the state-of-the-art Malvern Insitac particle analyzer under challenging sampling conditions involving high pressure, high temperature, and corrosive moist gases with a high dew point. The experimental results demonstrate that the flow cell provides a well-defined measurement zone achieved through a smooth coaxial flow field. This setup offers several advantages:

- (1) The optical windows of the flow cell are effectively protected by the purge flow, minimizing the risk of any deposition from the sample flows. This ensures the integrity and longevity of the optical components.
- (2) The sample flow within the measurement zone remains undiluted by the purge flow, allowing for accurate PSD measurements without significant interference.
- (3) The flow cell design promotes a stable scattering background even under high pressure and temperature conditions, resulting in minimal beam steering and ensuring reliable measurement results.

The testing results reveal that the Insitac Analyzer, equipped with this flow cell, successfully measures PSD under high pressure and high temperature without introducing measurement bias. Specifically, the Insitac Analyzer, paired with the flow cell, achieved on-line PSD measurements in the sample flow under 15 bara extracted from a 100 kW_{th} pressurized oxy-coal combustor. The results show a bimodal distribution of particle sizes, which holds significance for the design and operation of power plant boilers.

6. Patents

A patent, resulting from the work reported in this manuscript, has been applied for. The international application number is PCT/US2023/070693.

Author Contributions: Conceptualization, R.A. and M.C.; methodology, R.A., M.C. and Z.W.; software, Z.W.; validation, M.C., Z.W. and D.M.; formal analysis, M.C. and Z.W.; investigation, M.C. and Z.W.; resources, R.A.; data curation, M.C.; writing—original draft preparation, M.C.; writing—review and editing, M.C., Z.W. and R.A.; visualization, M.C.; supervision, R.A.; project administration, R.A.; funding acquisition, R.A. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflicts of interest.

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