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Development of a Real-Time Respirable Coal Dust and Silica Dust Monitoring Instrument Based on Photoacoustic Spectroscopy

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Abstract
We report the first spectral photoacoustic measurements of silica, coal, and kaolinite dust absorption coefficients from 11 to 13 µm at 5 nm resolution made with a tunable quantum cascade laser. This is important because airborne silica dust and coal dust within mining environments continue to be a problem for mine workers and staff due to their severe health effects on the respiratory system, while other dust types are potentially interferents to their detection. Our real-time spectra compare favorably with the non-real-time filter-based Fourier transform infrared spectrometer (FTIR) spectra obtained using the sampling system developed by NIOSH for their end-of-shift method to determine silica dust concentrations using a portable FTIR. We discuss our new dust generation system and instrument testing chamber. We also show that our PM4 silica mass concentration measurements by a low-cost air quality sensor (SPS30) are in good agreement with the TSI Aerosol Particle Sizer instrument, the NIOSH end of shift method, gravimetric mass, and correlate well with photoacoustic light absorption measurements at a wavelength where silica absorbs strongly.

1 Introduction

Silica is a common mineral in the Earth's crust and can occur naturally in the amorphous or crystalline forms [1]. Most crystalline silica appears in the form of alpha quartz [2]. In the mining industry, the presence of silica is associated with different mineralogy, being present either in the ore or in the surrounding rock formations. The different unit operations in a mine cycle, such as drilling, blasting, and scaling, will generate small particle sizes in the range of $\lessapprox 4.0$ µm in addition to larger particles. Small particles are easily inhaled and directly deposited into the lung structures, causing serious damage to the lung alveoli [3]. Respirable crystalline silica (RCS) is associated with several lung diseases, including silicosis [1] and lung cancer [4]. The long-term exposure to RCS and the short-term exposure to high concentrations represent a serious problem in the mining industry, which can lead to incurable diseases and even death [5]. However, illnesses caused by exposure to RCS can be prevented by applying engineering controls to reduce the concentration of respirable particles in the mine air [6]. Nevertheless, the continuous monitoring of exposure to RCS is essential to validate the efficacy of dust control techniques [7].

There are several silica monitoring methods currently used to measure the concentration of RCS in different mine environments. Some of these methods involve the collection of samples on filters and posterior laboratory analysis and calculation of the silica concentration. These techniques usually require sample treatment and redeposition on new filters before the material is analyzed [3]. Some standard silica monitoring methods include the NIOSH 7603 and the MSHA P7 [8, 9]. The NIOSH 7603 method uses FTIR spectra in the range of 1000 to 650 cm^{-1}. The MSHA P7 method collects respirable dust on filters and analyzes the redeposited material after ashing, with FTIR measurements in the range of 1000 to 700 cm^{-1}. Both methods calculate the silica concentration based on the characteristic absorbance peak area at 800 cm^{-1} (12.5 µm

We certify that the submission is original work and is not under review at any other publication.

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wavelength) for quartz. The interference of kaolinite in the quartz absorbance peak area is deducted by calculating the presence of kaolinite based on its absorbance peak area at 915 cm^{-1} and using it to predict the kaolinite contribution to the absorbance peak area at 800 cm^{-1}.

One of the major disadvantages of these methods is the lag time for the samples to be analyzed and the concentration calculated. As a result of having to treat and ash the samples, it may take days or even weeks to get analysis results, which prevents timely action [7]. However, a field-based technique has been developed by NIOSH, which allows the measurement of respirable silica concentrations in a more time-effective manner. The method provides the analysis of RCS concentration at the end of the shift by using a portable FTIR on samples collected on filters using a coal mine dust personal sampler unit (CMDPSU). The CMDPSU consists of a respirable cyclone, dust sampling cassettes, and a personal pump, which are used to collect the dust on PVC filters during the worker's shift. This new technique has shortened the time delay for getting the results, since the samples can be analyzed immediately after the shift. Additionally, it is a non-destructive method, allowing filters to be analyzed by other methods, such as the MSHA P7 [7].

An alternative technique to FTIR analysis for determining the RCS concentration of aerosol consists of measuring sample transmission with a tunable quantum cascade laser (QCL). The sampling method is similar to the ones applied in the standard techniques and also involves the treatment and redeposition of the sample on a new filter prior to analysis. The quantification of alpha quartz is obtained through analysis of infrared transmission measurements employing the QCL and a mercury-cadmium-telluride detector [10].

The common problem that these current methods have is that the sampling time usually occurs over one shift, which can only provide the time-weighted average of silica concentration [3]. The results, therefore, cannot directly identify and differentiate the various concentrations of silica dust based on operations and locations in a mine in order to trigger a dust control system. A real-time silica monitoring approach is necessary to determine the efficiency of the engineering controls for RCS dust concentrations in mines. Such a method would also be able to account for different concentrations at specific locations in a mine and prevent exposure to concentrations above permissible limits. As a result, we discuss photoacoustic measurements aimed at monitoring RCS dust concentrations for mine environments in real-time. Comparisons to the NIOSH FTIR technique are shown. We also discuss the use of measurements at different wavelengths to obtain kaolinite and coal dust concentrations and the use of absorption spectra for water vapor that is always present in the environment to calibrate the real-time instrument.

2 Methods

2.1 Aerosol Testing Chamber

In order to carry out the evaluation and calibration of a host of instruments simultaneously, NIOSH has developed an aerosol chamber. This chamber, also known as Marple Chamber, has the capability of evaluating many instruments at the same time. The NIOSH Marple chamber is equipped with a rotating table that allows the samplers to collect similar samples from different regions in the chamber [11].

The University of Nevada, Reno (UNR) dust chamber can be considered a "Marple" type chamber, for it allows the use of multiple instruments, both inside and outside the chamber, at the same time and also presents a uniform aerosol concentration in the test section. The chamber is $1.7 \times 0.8 \times 2.0$ m, for a volume of 2.7 m^3. The chamber is located at the ventilation lab at UNR and was initially built to control ionizing radiation by using high efficiency particulate air (HEPA) type filters. The chamber was re-designed for aerosol measurements. In order to control the suspended aerosol in the chamber, there is a PVC piping system connected to a centrifugal fan (S/N 91–86,972-1–1 from Twin City Fan & Blower Company) that provides for quick evacuation of the aerosol plume. The aerosol delivery system consists of a metal container (bowl) containing the material to be dispersed and a compressed air unit, which is connected to a tube placed above the bowl inside the dust chamber via a feedthrough connector. Once the system is ready, compressed air is fired, letting it hit the bowl for approximately 5 s. Other feedthrough connectors allow dust to be sampled by the photoacoustic instrument and a CMDPSU reproducing the NIOSH standard technique. Additionally, a low-cost air quality sensor (Sensirion SPS30) is installed inside the chamber, allowing the monitoring of the total concentration and size distribution of the aerosol in real time. The chamber has been used in the generation of different types of aerosols (silica, kaolinite, graphite, coal, and mixtures of these) that stabilize after 6 min and slowly decay over about 2 h, which allows the collection of samples by different sampling methods. Once the sampling is finalized for each experiment, the ventilation system attached to the chamber is activated, clearing the chamber after about 4 min. Figure 1a shows the dust chamber assembled with the external instruments used in this project.

2.2 Filter Sampling, Gravimetric and FTIR Measurements, and FAST Calculations

The filter sampling process aims to reproduce the field-based silica quantification performed by the NIOSH

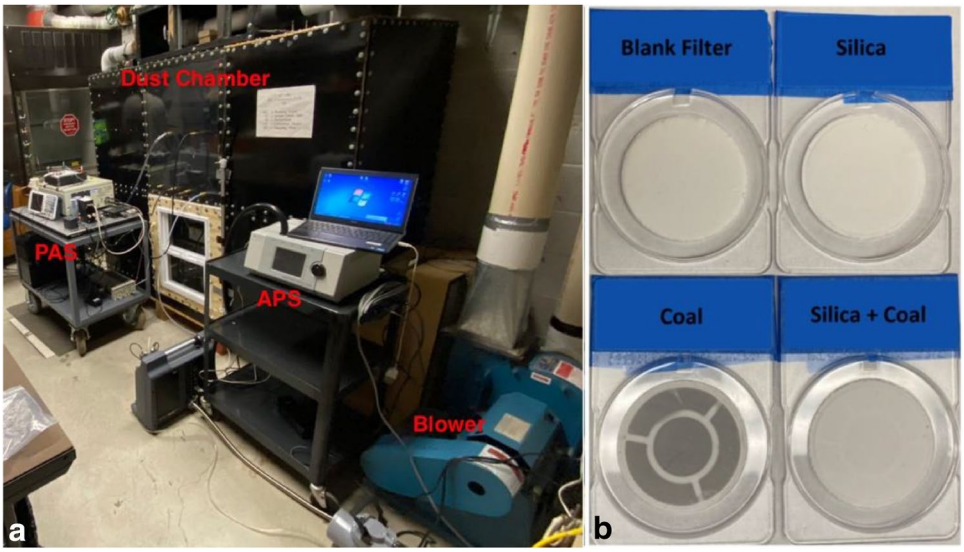

Fig. 1 **a**) Dust chamber and instrument. **b**) Examples of filters

end-of-shift method. Filters are loaded with different mineral dusts (Fig. 1b), and the samples are analyzed in a FTIR instrument in order to generate the spectra used for the silica concentration calculations. The sampler arrangement consists of an "SKC type" aluminum "PM_4" cyclone that passes respirable dust (Zefon International, Catalog NO. ZA0060), cassettes (Zefon International, REF 745PVC-CF-FTIR), 37 mm PVC filters (Zefon International, REF FSP37R), and tubing connected to a pump (Zefon International, N 629,946). The cyclone sampler operates at 2.5 LPM, for a 4-µm cut-point equivalent to passing 50% of aerosol having this aerodynamic diameter [12]. This cyclone is comparable to the one mentioned in the NIOSH 7500 and NIOSH 0600 [13, 14] used for sampling respirable dust. Once the sample is suspended, the pump is turned on after the dust concentration decay curve has stabilized as measured by the SPS30. The cyclone sampler is only removed after the chamber is cleared and is considered safe to be opened. The cassette is carefully disassembled, and the filter is removed and placed into a filter holder for FTIR measurements after gravimetric measurements are obtained. The area of the filter that collects the sample is 6.92 cm^2. Gravimetric measurements of aerosol mass concentration were obtained by subtracting pre-exposure-mass from post-exposure-mass of the filters and dividing by the total sample volume, typically 7.5 L.

The exposed filters are analyzed in a FTIR instrument (NICOLET 380 FT-IR, by ThermoFisher), which does a spectroscopic transmittance measurement of aerosol deposited on the filter for a given wavenumber range. The transmission spectra are then converted into absorbance to obtain mass concentration of a mineral based on its characteristic absorption peak area in the spectrum. The transmission measurements were taken over a spectral range of 4000–400 cm^{-1} at a resolution of 4 cm^{-1}, after 3 min purging with nitrogen to remove water vapor and CO_2. The transmission was obtained based on an average of 32 readings.

The procedure for obtaining properly normalized silica transmission spectra consisted of two steps. The first step involves using a blank filter to collect a reference spectrum, which permits the removal of the filter media signal. Secondly, normalization is conducted through calculations using the data generated from the FTIR measurements of the post-exposure filters. This second step is important because there is a difference in transmissions of blank filters from the same production lot. Therefore, the transmission from the blank filter used as background is different from the transmittance of the unexposed filter used for sampling. The difference in transmission through filters from the same lot can be explained due to differences in the filter media, differences in the purging time and its effectiveness, contamination of the filter with undesired particles, damages to the filter structure during handling, and the displacement of the filter in the FTIR. Further studies need to be carried out in order to understand the influence of these aspects on the variability of transmissions of filters from the same batch.

Once the FTIR data is collected, the transmission spectrum is plotted as a function of wavelength. The wavelength range of 12 to 13 µm is focused upon for silica quantification, as this is where the two characteristic peaks for silica are found [7]. The normalization is made based on the linear interpolation of a line generated from the transmission values of the limits applied. After interpolating, the normalized transmission is obtained by dividing the original transmission generated by the FTIR by the transmission calculated from the interpolations. According to Wei [10], applying a normalization procedure guarantees that the FTIR transmission spectrum reflects only the mineral dust sample transmittance.

The determination of RCS dust concentration during the shift is carried out by using the NIOSH Field Analysis of Silica Tool (FAST) [7], which receives data from a FTIR instrument after the requisite area of the spectra is integrated. The software then translates it into a silica mass and silica concentration based on the sampling time and pump flow rate for each sample collected [7]. The resulting data needs to contain the mandatory fields: sample name, Q, K, M, D, and C. These variables refer to a specific mineral signature feature: Q is quartz, K is kaolinite, M is microcline, D is dolomite, and C is calcite [15]. FAST calculates the silica concentration based on the integration areas of a spectrum of a single sample, the flow rate, the sampler type, the filter size, the sampling time, and the commodity that represents the mine production [15]. For example, "Q" is calculated from the area under the curve of the FTIR absorbance spectrum between 770 and 815 cm^{-1} [9]. Once Q is calculated, it is input into the FAST software, and the silica mass concentration and total mass are calculated for experiments involving only silica.

The aerosol-on-filter absorption coefficient (B_{abs} in Mm^{-1}) spectra for silica are obtained by transforming the transmittance into absorptance. The attenuation (*ATN*) is calculated as the negative natural logarithm of the transmitted light intensity normalized by incident light intensity. The calculations are based on the *ATN*; filter area (*A*) exposed to a sample in cm^2; the sample flow rate in liters per minute (*F*) and sample duration (*t*) in minutes. The following equation is used to perform the transformation [16]:

$$B_{abs} = \frac{ATN \times A \times 100000}{F \times t} \tag{1}$$

2.3 Photoacoustic Spectrometer

When light is shined upon an aerosol plume, any or all of three interactions take place. The light can be transmitted through the plume, scattered in a new direction, or absorbed by the aerosol. In the case of absorption, thermal energy is transferred from the heated aerosol to the surrounding air, causing it to expand [17]. Figure 2 is a diagram of the photoacoustic instrument. Laser beam transmission with pure scattering as well as absorbing aerosol have been used in the past for calibration [18].

In Fig. 2, sample air is pulled through the resonator section with a vacuum pump. A wavelength-tunable quantum cascade laser (QCL) is used as the light source. The laser beam enters the window on the left, interacts with the aerosol in the resonator, and then exits on the right. The laser light that is not absorbed or scattered then enters the integrating sphere where a mercury-cadmium-telluride (MCT) detector records the laser power. The modulation frequency of the QCL is set to the resonance frequency of the resonator. Light power is absorbed by aerosol, which is then transferred to the air surrounding the particle, causing thermal expansion. The modulation of the light source and aerosol light absorption thus creates a standing sound wave as the air expands and contracts, which is measured by the microphone using phase sensitive detection [19]. In order to quantify airborne respirable silica, kaolinite, and coal, the QCL is tunable and covers the wavelength range of 11 to 13 µm [20]. By using sound pressure P_m, laser power P_L, resonator cross-sectional area A_{res}, ratio of specific heats γ for air, resonator quality factor Q, and resonance frequency f_0, the aerosol light absorption coefficient is given by [19].

$$B_{abs} = \frac{P_m}{P_L} \frac{A_{res}}{\gamma - 1} \frac{\pi^2 f_0}{Q} \tag{2}$$

When combined with an empirical or theoretical mass absorption efficiency *MAE* (see Fig. 3), respirable silica mass concentration is obtained from $PM_4 = B_{abs}/MAE$.

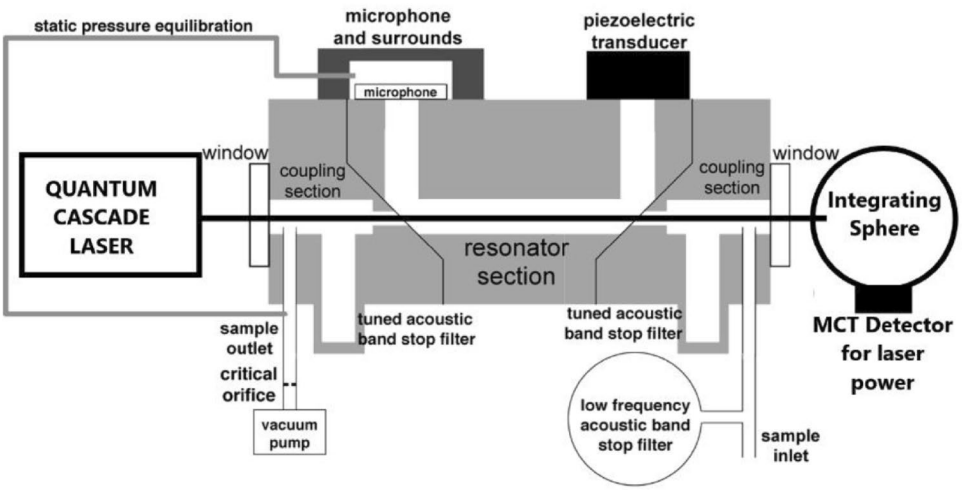

Fig. 2 Diagram of the photoacoustic instrument

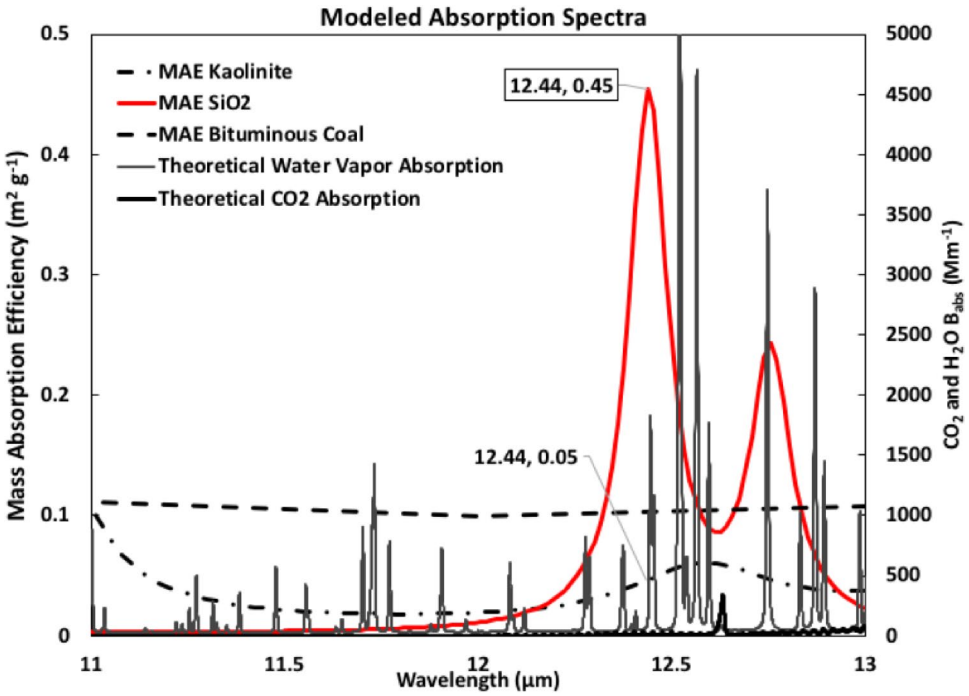

Fig. 3 Modeled absorption *spectra*

2.4 SPS30 and APS Measurements

During the measuring process, it was important to be able to monitor the particulate matter [21] concentration within the aerosol chamber, both for safety and for confirmation regarding the results of silica concentration obtained with FTIR and photoacoustic measurements. Two instruments were employed for this. The first is the Sensirion SPS30, a low-cost, compact air quality sensor. The instrument uses a laser and a photodetector to count and size particles via light scattering [22]. The SPS30 was the primary PM measurement device in most cases. In some cases, an Aerodynamic Particle Sizer (APS; Model 3321, TSI Inc.) was also used for size distribution measurements that were integrated for comparison to the SPS30 in order to verify the low-cost instrument's measurements.

3 Results

The following figures present the results obtained from experiments carried out in UNR's ventilation lab, as well as theoretical spectral analysis. The experiments were performed on RCS, kaolinite, and coal dust.

3.1 FTIR and Photoacoustic Spectra for Dust and Water Vapor

Figure 3 shows the theoretical absorption spectra for kaolinite, silica, coal, water vapor, and CO_2 [23]. As seen in the figure, water vapor and kaolinite have some interference on the silica absorption peak area of 12.44 µm. The theoretical analysis of the spectra in Fig. 3 shows the need for removing the interference of kaolinite and other minerals on the silica concentration calculations. Figure 4 presents the silica absorption coefficient spectra from FTIR analysis of an RCS laden filter and photoacoustic measurements. Each photoacoustic measurement had a duration of 4 s. As shown, there is a remarkable agreement between the spectra and the theoretical spectrum for silica, showing the two characteristic peaks centered near 12.44 and 12.8 µm along with water vapor absorption peaks. The filter absorption spectrum is slightly higher than the photoacoustic spectrum, likely due to multiple scattering enhancement of aerosol light absorption by the filter substrate [16].

Figure 5a presents the kaolinite absorption coefficient spectra from FTIR and photoacoustic analysis. Both spectra show the increase of absorption at 11 µm, which is present in the theoretical curve. At the 12.44 µm region, the photoacoustic measurement does not indicate much absorption by kaolinite. Figure 5b shows measured photoacoustic and FTIR absorption spectra for coal. Photoacoustic measurements show a gradual decrease of absorption with water vapor absorption superimposed. It was not possible to properly normalize the FTIR coal spectrum because it is relatively featureless with no regions where absorption approaches zero. However, the general shape of photoacoustic and FTIR spectra is in agreement.

3.2 Silica Mass Concentration

Figures 6a and b present RCS dust concentrations. Figure 6a shows a comparison between the SPS30 and the APS for RCS dust concentration measured in the UNR chamber. Figure 6b shows the silica mass concentration comparison between the SPS30 and the mass concentration obtained through calculations using the NIOSH FAST software based on the FTIR spectrum.

Figure 7 shows a time series of silica dust concentration and aerosol light absorption measurements. Gravimetric and FAST-FTIR spectra measurements are in good agreement

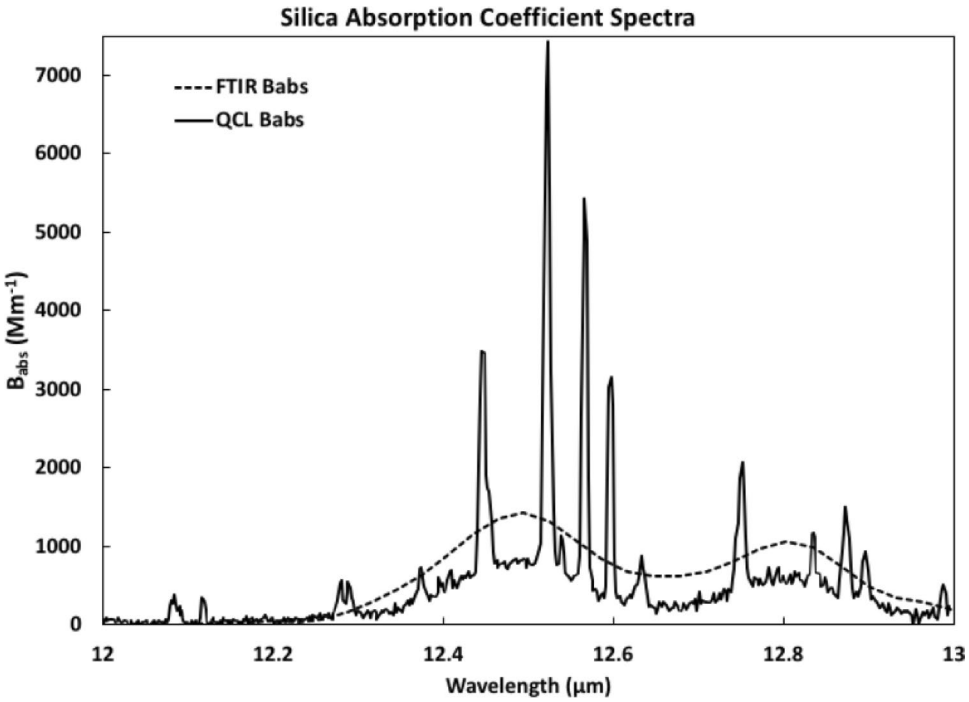

Fig. 4 FTIR and photoacoustic silica spectra

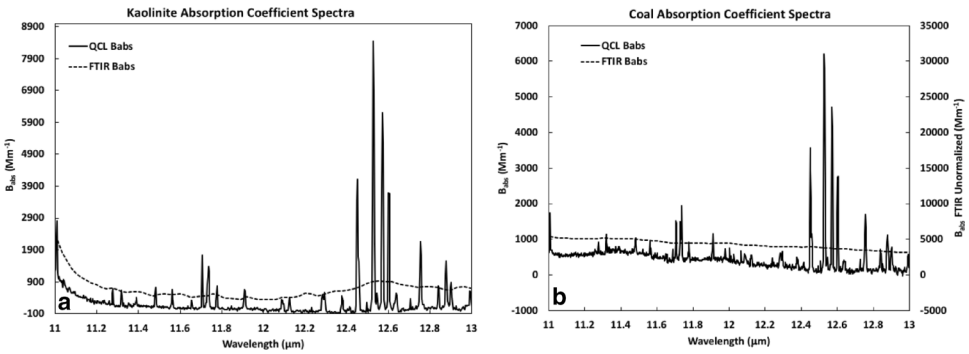

Fig. 5 **a**) FTIR and photoacoustic kaolinite spectra. **b**) FTIR and photoacoustic coal spectra

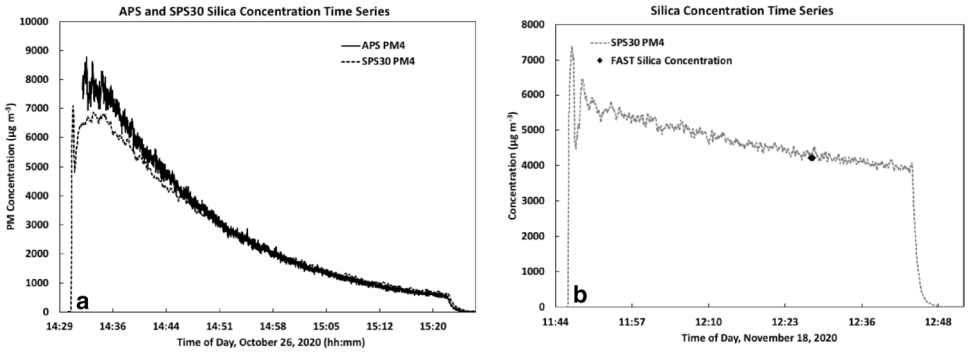

Fig. 6 **a**) Time series of PM concentration comparing APS and SPS30 instruments. **b**) SPS30 PM concentration compared to FAST calculated silica concentration obtained through FTIR filter measurements

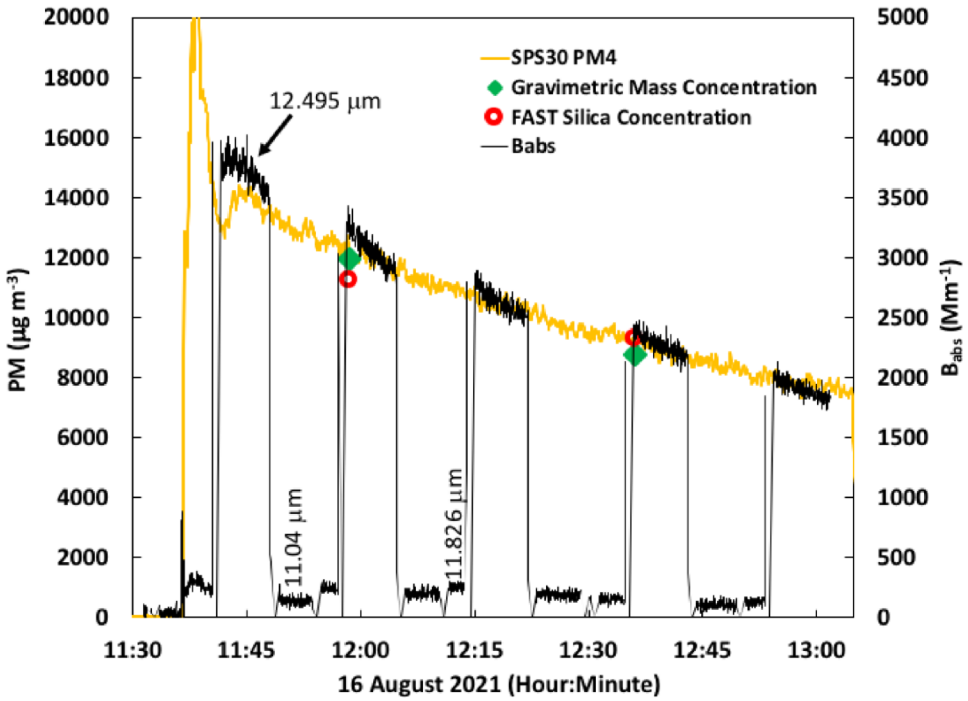

Fig. 7 Time series of PM concentration (left axis) and photoacoustic light absorption measurements(right axis) for silica dust. Gravimetric and FAST FTIR mass concentration measurements are shown by green diamond and red circle symbols, respectively. The filters sampled dust for 3 min. Light absorption measurements with the photoacoustic spectrometer are in black. For long, middle, and short time segments, the QCL was set to wavelengths 12.495 mm, 11.04 mm, and 11.826 mm, respectively. Photoacoustic measurements were obtained every second, while SPS30 values were every 4 s

with SPS30 measurements. Photoacoustic light absorption measurements are shown for three wavelengths to illustrate tunability. An aerosol filter was used to zero the photoacoustic instrument before each QCL wavelength change. The wavelength 12.495 µm was chosen because it is near the peak of the silica absorption spectrum shown in Fig. 4 and is away from strong water vapor absorption. Wavelengths 11.04 µm and 11.826 µm were chosen because they may be useful in quantifying kaolinite and coal dust, given that silica does not absorb strongly at these wavelengths. Figure 7 suggests that silica dust mass concentration can be obtained from photoacoustic light absorption measurements at 12.495 µm by multiplying them by a factor of 4 as a calibration factor. Research is currently underway to show that silica, kaolinite, and coal dust may be obtained from spectral measurements with the photoacoustic instrument when all three components are present.

The detection of silica by photoacoustic measurements becomes more sensitive with increasing integration time and laser power. Currently, the laser power is 6.3 mW, and for an integration time of 1 s, a silica mass concentration 120 µg m^{-3} can just be distinguished from measurement noise. We estimate that to detect 0.5 µg m^{-3} would require a 10-min time average and 63 mW of laser power.

4 Discussion and Conclusions

4.1 QCL and FTIR Comparison Conclusions

In Figs. 3 and 4, one of the immediately obvious features of the graphs are the large, localized spikes created by water vapor in the absorption spectra. By using a relative humidity sensor within the flow of the instrument, the water vapor concentration can be calculated and used to obtain theoretical water vapor spectra. These spectra can then be used to check the calibration of the silica absorption spectra in a manner similar to the use of NO_2 gas to calibrate 0.532 µm photoacoustic instruments [24]. The pair of peaks between 12.4 and 12.7 µm are characteristics of silica dust. So long as a measurement wavelength is chosen that lies between water vapor spikes and is located near to the top of one these two peaks, RCS dust concentration can be measured with minimal interference by water vapor.

Figures 4 and 7 are the crux of this research. The solid line in Fig. 4 shows the silica absorption spectra as measured by the photoacoustic instrument in situ. As noted

previously, the large spikes in the absorption spectra are a result of water vapor in the air. The underlying wider pair of peaks are indicative of silica at this wavelength range. When compared to the absorption spectra of silica obtained via the FTIR filter method (Fig. 4), and the silica mass concentration measurements (Fig. 7), it becomes clear that the photoacoustic instrument is highly capable of quantifying airborne silica in real-time.

One of the problems that may arise is the interference of other airborne pollutants like kaolinite and coal when measurements are taken in the field. However, because the instrument utilizes a tunable QCL, the instrument is also able to quantify these interferents for removal from the signal. Figures 5a and b show measured kaolinite and coal absorption spectra. By using absorption values collected at 11.04 µm and 11.826 µm, values for kaolinite and coal respectively can be obtained for removal from the silica signal. Ongoing research continues this analysis.

4.2 SPS30, FAST, and APS Comparison Conclusions

The mass concentration measurements were carried out using the SPS30 and the APS instruments. As shown in Fig. 6a, the results obtained by applying both instruments present a remarkable agreement between them. Additionally, the results from the SPS30 were compared to the calculations performed on the FTIR filter measurements and NIOSH FAST software. Figures 6b and 7 show that the SPS30 agrees strongly with the results obtained from FAST-FTIR spectra and gravimetric mass for silica concentration. This suggests that a low-cost air quality sensor such as the SPS30 can reliably provide total PM_4 measurements for aerosol experiments involving silica dust.

4.3 Future Work

We are working further in prototyping the photoacoustic spectrometer toward the development of a more compact, lower frequency instrument. A more compact size will allow for portability, while a lower frequency will help minimize thermal heat transfer phase shifts in the signal. Experiments to obtain silica dust concentration in the presence kaolinite and coal dust are underway.

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Declarations

Conflict of Interest The authors declare no competing interests.

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