Enabling crystalline silica analysis of mine respirable dust sampled by real-time gravimetric personal dust monitor (PDM3700)

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ABSTRACT

Monitoring of personal exposure levels of respirable coal dust (RCD) and respirable crystalline silica (RCS) is an important step in protecting mine workers from dust related occupational respiratory lung diseases. Timely monitoring and reporting of personal exposure levels is crucial for dust control and exposure management. The personal dust monitor (PDM3700) is a real-time mass-based gravimetric respirable dust sampler in mines. PDM3700 has been successfully used as a compliance monitor in the USA to aid the miners in reducing their exposure to coalmine dust, and also deployed in Australian coalmines since 2016.

The PDM utilises a tapered element oscillating microbalance (TEOM) technology that continuously weighs a filter as the respirable dust is deposited on it, thus monitoring in real time personal exposure levels of RCD. However, as in any currently available compliance gravimetric sampler, it does not provide information specific to RCS without further analytical process. As PDM3700 uses a glass fibre filter for dust sampling, Fourier Transform Infrared (FTIR) and X-ray diffraction (XRD) analytic methods cannot be directly applied to silica analysis due to analytical measurement interference. This paper reports a redeposition methodology to determine the silica content of respirable coalmine dust collected over the PDM glass fibre filter using FTIR method. The newly developed quick CSIRO analytical process has been demonstrated to effectively detach dust particles from the PDM filter without causing sample contamination by glass fibres. In addition, the paper provides an update on the development of a new type of PDM filter with a non-silica filter material and a novel filter assembly structure to enable field-based direct-on-filter silica analysis of PDM-collected dust samples. These new developments greatly advance TEOM based PDM technology to achieve both real-time RCD monitoring throughout the shift and RCS measurement at the end of the shift by using a single PDM sampler. It is envisaged that these would significantly improve RCD and RCS personal exposure monitoring capabilities to identify potential high personal exposure areas and tasks for effective engineering controls, implement on-site quick turnaround compliance assessment by empowering the worker and eliminate the delays in the current gravimetric sampling and off-site silica analysis.

INTRODUCTION

The personal dust monitor (PDM3700) represents the latest advanced technology in legislated personal compliance exposure monitoring of respirable coal dust (RCD) by undertaking real-time continuous gravimetric measurement (Volkwein *et al*, 2004; Belle, 2017). It was developed and adapted for coalmine use through a two-decade long research and development program in the USA by the National Institute for Occupational Safety and Health (NIOSH) and Mine Safety and Health Administration (MSHA) and is widely regarded as being significantly superior to the traditional gravimetric sampling method for RCD exposure monitoring. It utilises a tapered element oscillating microbalance (TEOM) technology that continuously weighs a filter as dust is deposited on it, and thus monitoring in real time personal exposure levels of RCD. In TEOM monitors, air is drawn through a filter placed on the top of an oscillating glass rod. The air flow rate through the filter is constant and the mass of the particles that attaches onto the filter will influence the oscillation frequency, which in turn makes it possible to calculate the particle mass and express this per volume of air. The key aspect of PDM3700 is that it directly measures the mass of sample dust on a filter according to the principles of physics regardless of dust composition, size and physical characteristics.

The real-time gravimetric PDM has been successfully used to aid miners in reducing their dust exposure by making changes to their work activities based on the continuous reading of the device. Similar to the current manual gravimetric personal dust samplers, it does not provide information specific to personal exposure of respirable crystalline silica (RCS). Miners are exposed to silica-bearing mine dust which can lead to silicosis, a potentially fatal lung disease. The current practice for measuring the exposure to RCS is to submit the filter dust sample collected by the traditional gravimetric sampler to an external laboratory where the mass content of silica in the collected dust sample is measured by the Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD) analytical methods. The filter media for sampling in the above FTIR and XRD methods are generally PVC membranes which are readily ashed by incineration to redeposit collected dust particles for subsequent silica analysis. Results generated using the above standard laboratory analysis are generally not available for several days to several weeks after sampling, which delays timely and effective intervention for engineering control of dust exposure. For timely RCS measurement, the NOISH has developed a field-based silica analysis method using a compact FTIR spectrometer and the FAST (Field Analysis of Silica Tool) field-based tool to directly measure silica content on-site at the end of the sampling shift. This field-based FTIR method is to directly analyse RCS of loaded dust over a gravimetric PVC filter without ashing and redeposition, which is called the direct-on-filter method (Miller *et al*, 2012; Cauda, Miller and Drake, 2016). It requires a traditional gravimetric sampler so has limited capability of real-time monitoring of RCD. Currently no miniaturised and proven worker-wearable instrument is available to offer real-time silica monitoring. True real-time silica analysis is also hindered by need for sufficient silica mass (ie sample accumulation during sampling). The end-of-shift direct-on-filter silica analysis is considered as the most practical and promising way to provide timely RCS exposure data on-site.

The current commercial PDM filter consists of borosilicate glass fibres with polytetrafluoroethylene (PTFE) polymer binder (Tuchman, Volkwein and Vinson, 2008). The collected dust particles are deposited onto the glass fibre filter media. The FTIR and XRD analytic methods cannot be directly applied to the silica analysis for PDM-collected dust samples due to interference by the glass fibre filter. The major issues with the current PDM filter are the difficulty in removing the collected dust sample from the glass fibre filter by incineration for redeposition and silica analysis, and its inability to accommodate the direct-on-filter analysis due to the blockage of IR beam by the bulky polypropylene filter holder.

Recently we have developed a methodology to enable the silica analysis of coalmine dust sample collected over the PDM glass fibre filter, which involves an invented detachment method to effectively detach the collected dust from the PDM filter into a solvent, followed by a filtration process to redeposit the dust particles onto a PVC membrane filter (Jin *et al*, 2024). The redeposited dust sample onto the PVC membrane will be measured for silica content with FTIR by using an on-filter method without ashing the filter sample. Such an on-filter FTIR analysis method is preferred for field applications at the mine site, due to its simple process and no requirement for complex laboratory sample preparation for polymer filter destruction. Further to methodology development, we have been developing the alternative PDM filter with a non-silica organic membrane material and a novel filter assembly structure to enable the direct-on-filter silica analysis of PDM dust sample with a field-based FTIR method.

The paper provides an update on the progress of both parts of the research with promising results and developments in expanding the capability of PDM to achieve both real-time respirable dust monitoring throughout the shift and silica content measurements at the end of the shift. The overall goal for the research is to develop a rapid reliable field-based technology for optimal monitoring of both RCD and RCS exposure levels by wearing a single dust sampler of PDM, which will significantly enhance the capability of the coal mining industry in monitoring and preventing the health hazards associated with personal exposure to respirable coal and silica dust. The technology has potential to be applied widely in mining and manufacture sectors.

METHODOLOGY FOR SILICA ANALYSIS OF REAL-TIME GRAVIMETRIC PDM DUST SAMPLES

Dust sampling was carried out with a laboratory dust test chamber to obtain a variety of dust samples for methodology development (Figure 1a). In order to validate the measured silica contents of PDM samples, parallel sampling was conducted by using PDM3700 with the commercial glass fibre filter and a traditional gravimetric personal dust sampler (Casella Apex2 Pro) with a PVC filter of 25 mm in diameter. The obtained parallel samples deposited onto the PDM filter and PVC filter will have a same content of respirable silica. The coal contains kaolinite that interferes with the FTIR analysis of quartz (silica dust). As the infrared spectrometry is sensitive to particle size, it is intuitively better to use the same size fractions for quantification of silica and kaolinite (Lee *et al*, 2013). Respirable kaolinite samples (Sigma Aldrich) collected with a personal dust sampler were used for FTIR analysis to establish kaolinite correction following the NIOSH Method 7603 (Schlecht and Key-Schwartz, 2003). The mixture of standard respirable quartz (NIST 1878b) with the coal sample (<125 μm) was fed into the dust chamber for sampling. The mixture ratios and dust feeding parameters were adjusted to vary the dust-loading amount and silica content of collected dust samples.

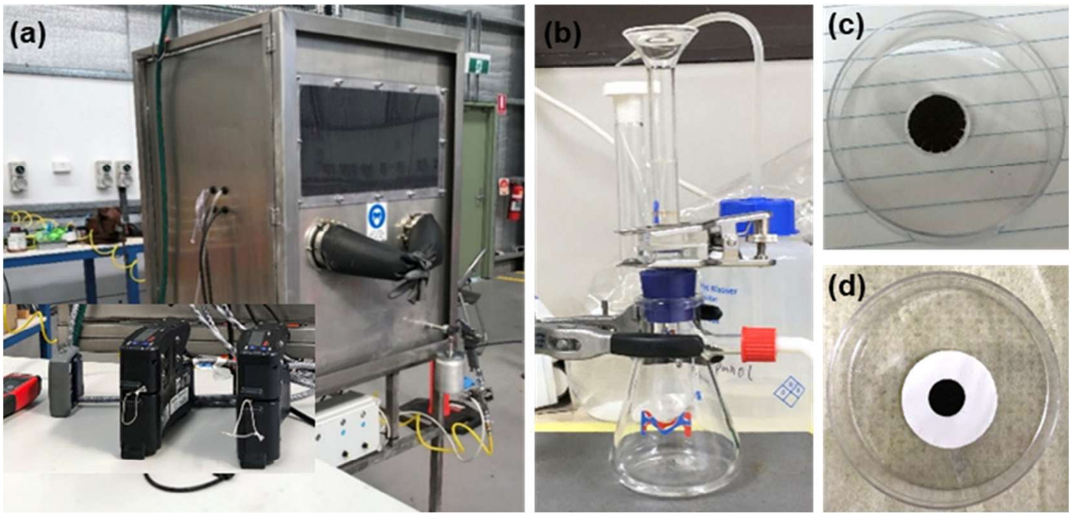

FIG 1 – (a) Laboratory dust test chamber for parallel sampling with PDM3700 and gravimetric samplers, (b) the filtration system for dust redeposition, (c) PDM filter sample and (d) redeposited PDM dust sample onto a PVC membrane.

The key step of the methodology is dust detachment to remove the collected dust from the PDM filter without introducing the contamination of glass fibres from the PDM filter into the detached dust sample. We have devised a novel solvent contact process, in which dust particles are selectively removed from the PDM filter and dispersed in an isopropanol solvent. The redeposition of detached dust in the solvent was carried out by a filtration method similar to the NIOSH 7603 with the filtration apparatus as shown in Figure 1b. Figure 1c and 1d show the PDM sample filter and redeposited dust filter, respectively. FTIR analysis of silica content of redeposited dust filter was conducted with a spectrometer (Thermo Scientific Nicolet iS50) and followed the method as described in the NIOSH 7603 using the IR absorbance peak of 800 cm^{-1} with the baseline from ca 820 to 670 cm^{-1}. The standard respirable quartz (NIST 1878b) was used to establish the calibration curve of silica quantification (silica mass content versus peak height at 800 cm^{-1}). The kaolinite correction was applied in the calculation of the peak height at 800 cm^{-1} for coal dust samples.

For silica analysis of dust samples collected by a gravimetric personal dust sampler, the PVC filter sample was immersed in isopropanol in a beaker. The dust was detached from the PVC filter with an ultrasonic bath and then redeposited onto a PVC filter using the same redeposition process as described above. The redeposited gravimetric sample was analysed for silica content by the FTIR method for comparison with the measured content of the PDM sample collected in the same parallel sampling run. Parallel PDM and gravimetric samples should have a similar silica content.

It has been demonstrated that the developed solvent contact process is efficient to detach the collected dust from the PDM filter with minimum contamination of filter glass fibres. The detailed optical microscope and scanning electronic microscopy (SEM) studies revealed that nearly no glass fibre contamination was observed in the redeposited PDM dust sample. In comparison, significant contamination of glass fibres was observed in the redeposited DPM sample that was detached by the ultrasonic process. The SEM-energy dispersive X-ray (EDX) analysis of the redeposited dust samples was used to identify silica dust particles, glass fibres and kaolinites, and the presence of various silicon-containing components was evident.

Multiple sets of parallel PDM and gravimetric samples with a variety of dust-loading amounts (0.3–2.2 mg) and silica concentrations of (1.0–4.5 wt per cent) were processed using the developed methodology and analysed by FTIR. As shown in Figure 2, the measurement content of silica in the PDM samples are very close to those of gravimetric samples collected in a same run of parallel sampling. The differences of the measured silica contents between PDM and gravimetric samples are less than 10 per cent with the majority of variations below 5 per cent. The FTIR technique is a highly sensitive means for silica quantification, and some of measured samples have a silica content as low as 5–10 microgram.

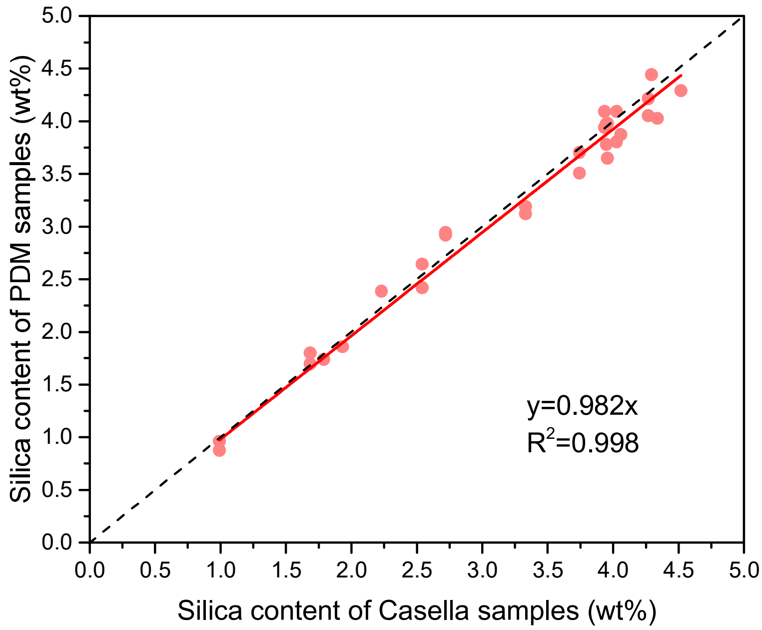

FIG 2 – Comparisons of measured silica contents of parallel PDM and gravimetric samples processed by the developed methodology.

The results have clearly demonstrated that the developed methodology is feasible and practical for enabling silica analysis of PDM-collected dust samples. Some of redeposited dust samples were also analysed with a compact FTIR spectrometer (Nicolet Summit), which gives very similar analysis results to those by the Nicolet iS50 FTIR spectrometer. The developed methodology is a simple and rapid process with very simple set-up and ease of operation for end user or the analytical service provider. The set-up is readily installed at mine site, and the entire process for dust detachment, redeposition and FTIR analysis takes about 15 mins. It has significant potential for use in the end-of-shift silica analysis of PDM dust samples at remote mine sites.

NEW PDM FILTERS FOR DIRECT-ON-FILTER SILICA ANALYSIS

The other part of our research is focused to seek the alternative PDM filter with non-silica organic materials and a novel filter assembly structure to enable the direct-on-filter silica analysis of PDM dust sample with a field-based FTIR method. PDM sampling with the developed new PDM filter will completely avoid the issues with the current glass fibre filter. The objective of this work is to enable FTIR silica analysis directly on the dust sample deposited on the new PDM filter at the end of the shift in the field.

To date, the research team has developed a new structured non-silica polymer filter membrane as an alternative to the current glass fibre filter membrane. The new polymer membrane was installed onto the current PDM filter holder for evaluation of sampling performance. The resultant polymer membrane filter was fitted into a PDM3700 for parallel sampling with another PDM3700 that used the current commercial glass fibre membrane filter. Figure 3 shows the comparison of the parallel sampling results of these two PDM3700 samplers. As shown in Figure 3a, the PDM with the new polymer membrane filter exhibits a slightly higher amount of dust collection than that with the commercial filter during a 4 hour dust sampling, with an overall 6.8 wt per cent more at the end of sampling. The measured dust concentrations for the two PDM units are almost same within most of sampling period except those values of at peak dust concentrations inside the chamber (Figure 3b). The dust concentration surge inside the chamber was resulted from the intermittent dust feeding by compressed air. The variation of measured peak dust concentrations between two PDM units is likely due to the inherent errors of the two PDM units used. The detailed sampling tests and evaluation are underway. The new filter holders with a novel design capable of easy removal of filter membrane from the holder for direct-on-filter silica analysis are fabricated and will be evaluated in parallel sampling tests. The key challenge for developing new sample holder is the mass limit of the holder generally requiring below 0.1 mg to meet the PDM3700 operation requirement.

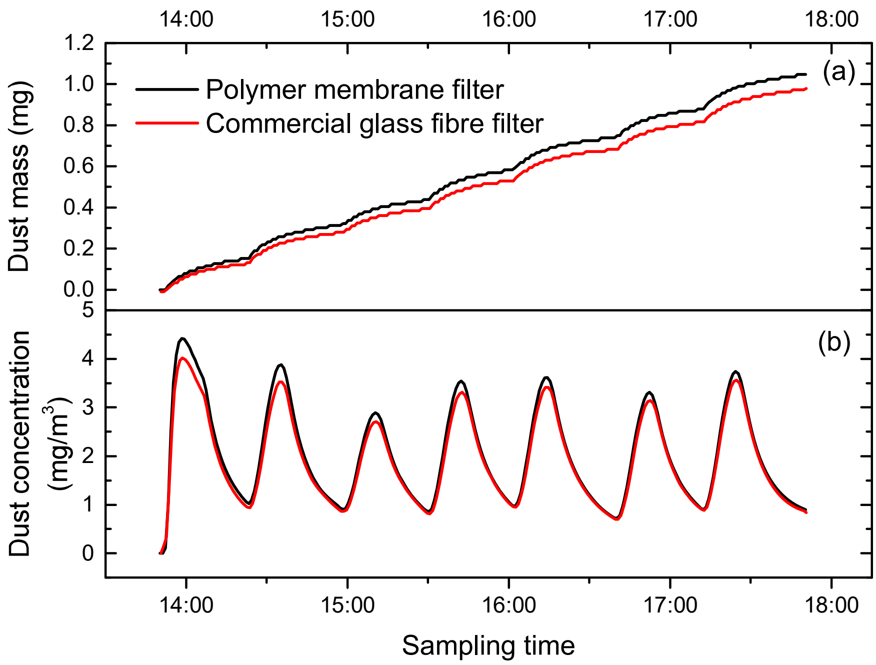

FIG 3 – Parallel sampling results of two PDM3700 units with a commercial glass fibre membrane filter and a newly developed non-silica polymer membrane filter respectively: (a) total mass of collected dust, and (b) dust concentrations throughout the sampling period.

CONCLUSIONS

At the time introduction of real-time gravimetric PDM3700 sampler, there was an incorrect perception created and communicated by various interested parties that the NIOSH (USA) approved and MSHA (USA) legislated PDM3700 sampler is not mass-based gravimetric sampler and do not collect coalmine dust over the filter for silica analyses. These specific misgivings and a result of shortcomings of non-acceptance of US Intrinsic Safety (IS) approval in Australia have resulted in them not being used as an effective compliance tool to empower the workers for personal dust exposure management. This paper updates our research progress on enabling the silica analysis of PDM-collected respirable dust for the deployment of advanced real-time gravimetric TEOM mass-based monitor to achieve both real-time RCD monitoring throughout the shift and RCS measurement at the end of the shift. The methodology developed by the CSIRO, which involves an innovative sample dust detachment process followed by filtration redeposition, has been demonstrated as an efficient technique to measure the silica content of mine dust samples collected by PDM3700 with the current commercial glass fibre filter. The developed methodology combined with a compact field-based FTIR spectrometer has a significant potential to be implemented for end-of-shift silica analysis at the mine site. Moreover, the research team is currently working on the development of alternative

PDM filters with a non-silica organic polymer membrane and a novel filter assembly structure to enable the direct-on-filter silica analysis of PDM dust sample with a field-based FTIR method. The parallel sampling tests with a new polymer membrane filter have showed promising performance with its RCD monitoring result very close to that of the currently used glass fibre dust filter. The success of the research will directly enhance the RCD and RCS monitoring capabilities for the mining industry.

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