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Respirable dust constituents and particle size: a case study in a thin-seam coal mine

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Abstract
This paper presents a case study of respirable dust characterization in a thin-seam coal mine in southern WV. Samples were collected in the intake, near the feeder breaker, and downwind of an active roof bolter, as well as in three downwind locations from the continuous miner during four separate cuts. The dust was analyzed using: scanning electron microscopy with energy-dispersive X-ray (SEM–EDX) to estimate particle size and mineralogy distributions; thermogravimetric analysis (TGA) to estimate coal, non-carbonate, and carbonate mass fractions; and Fourier transform-infrared (FT-IR) spectroscopy to estimate quartz and kaolinite mass content. SEM–EDX results were generally consistent with those obtained in previous studies of other central Appalachian mines, including presence of relatively high non-carbonate minerals content (primarily aluminosilicates and silica) associated with the rock strata encountered in the mine. Downwind of the miner, fine aluminosilicate particles were particularly abundant and apparently influenced the SEM–EDX analysis for some samples, resulting in underestimation of coal content relative to TGA. Comparison of the microscopy results to those from the TGA and FT-IR indicates some interference between aluminosilicates and coal dust is simply due to high sample loading—however, presence of coal-mineral micro-agglomerates is also indicated.

Keywords Respirable coal mine dust · Dust characterization · SEM–EDX · FT-IR · TGA · Black Lung

1 Introduction

Respirable dust exposures still represent a serious occupational hazard for many coal miners. In the US, the available dust monitoring data suggest that concentrations of respirable dust and crystalline silica (i.e., quartz) in coal mines have generally been declining across all regions for the last few decades [1]. Nevertheless, health surveillance data show a resurgence of occupational lung disease since the late 1990s, which has been especially dramatic in the central Appalachian region (i.e., including eastern KY, southwestern VA, and much of WV), where the most severe and rapidly progressive forms of disease have been reported [2, 3]. Radiographic evidence indicates that crystalline silica exposures are a likely factor in such cases, and several studies including pathology have also shown significant burden of silicate (in addition to silica) particles in lung tissue [4, 5].

Taken together, the above observations have prompted keen interest in the characteristics of respirable coal mine dust [6]. It has been commonly speculated that the tendency to mine thinner and thinner coal seams in central Appalachia—and thus to mine more and more rock strata along with the coal—has gradually increased the mineral content in the respirable dust [7]. Likewise, a reduction in dust particle sizes [8] could have occurred due to changes in mining technology and practices such as increased use and power of continuous mining equipment [6]. However, since routine dust monitoring in US mines only yields the respirable dust mass concentration (mg/m^3) and silica content (reported as % quartz), data on other metrics are scarce (e.g., abundance of constituents other than silica, particle size).

To begin filling in the knowledge gap surrounding respirable dust characteristics, the authors' research group has been sampling and analyzing dust from US mines (e.g., see [9–12]). The use of scanning electron microscopy with energy-dispersive X-ray (SEM–EDX) has been an important tool for that work. While not feasible for routine monitoring, it allows analysis of

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particle size and mineralogy distributions on filter samples. For practical comparison of dust constituents between different samples, defined mineralogy classes have been established that cover most particles found in coal mine dust (Table 1).

Heretofore, the particle-based SEM–EDX results have not been widely compared to other methods. One logical comparison is between SEM–EDX-derived silica (S) and kaolinite (ASK) and standard measures of quartz (crystalline silica) and kaolinite mass, such as achieved using infrared (IR) spectroscopy. Indeed, the US National Institute for Occupational Safety and Health (NIOSH) has recently developed a direct on-filter Fourier transform IR (FT-IR) method intended for analysis of quartz in respirable coal mine dust [13]; the method concurrently quantifies kaolinite since it must be used to correct the quartz result. Furthermore, SEM–EDX data can be compared with those derived from thermogravimetric analysis (TGA). Per [14], TGA can be used to estimate three primary mass fractions in respirable coal mine dust (coal, non-carbonate minerals, and carbonates), which might serve as good proxies for the primary dust sources in many mines (i.e., coal strata, rock strata, and limestone rock dusting products). To make a comparison between SEM–EDX and TGA data, the SEM–EDX data must be collapsed (i.e., C + MC = coal; ASK + ASO + HM + S + OS = non-carbonate minerals; CB = carbonates) and particle number-based data must be transformed into mass.

To gain further insights on respirable dust characteristics and how the above analytical measures compare, this paper presents a case study from a thin-seam coal mine in southern WV. (Data from this study were previously presented at a conference [15], and the study report is expanded here.)

2 Materials and Methods

2.1 Site Details

The mine sampled for this study is a room and pillar operation in US MSHA District 12. It is extracting the Upper Alma coal seam, with an average thickness of 0.8 m. The average total mining height is about 2.4 m, including about 1.2 m of shale (roof), 0.2 m of sandy shale (floor), and 0.2 m of mudstone. The mine uses exhausting ventilation with line curtain at the face. The supersection sampled had seven entries, and split ventilation with the intake in the central #4 entry and returns in #1 and #7 entries. On each side of the supersection, the mine was operating one continuous miner (CM), one roof bolter, and two shuttle cars during all of the sampling.

2.2 Dust Sampling

Respirable dust sampling was conducted over two days in March 2020, in six key locations on a single side of the supersection (Fig. 1). Samples were collected in the intake (I), just downwind from an active roof bolter (B) which was operating upwind of the CM, and adjacent to the feeder breaker (F) on Day 1. On Day 2, samples were collected at the CM curtain mouth (CM1), at the next break downwind from the CM1 location (CM2), and further downwind in the return (R). The CM1, CM2, and R samples were collected simultaneously during four separate events corresponding to four consecutive CM cuts. Cuts 1 and 4 were in the #5 entry heading and the cut depth was about 4.6 m with the CM scrubber turned off for the entire cut (in compliance with the mine's approved ventilation plan). For Cut 2 (# 7 entry) and 3 (# 6 entry), the cut depth was about 6.1 m and the scrubber was turned on for the entire cut.

In each location, samples were collected in sets and a total of 15 sets were collected (i.e., I, B, F, and 4 sets of CM1, CM2, R). Each set contained four sampling trains, consisting of an Escort ELF pump operated at 2.0 L/min with a 10-mm Dorr Oliver nylon cyclone (Zefon International, Ocala, FL) to collect only the respirable-sized dust (i.e., top size of about 10 μm and d_{50} of about 3.5 μm). Dust was collected directly onto 37-mm filters housed inside 2-piece styrene cassettes. In each sample set, two of the filters were track-etched polycarbonate (PC; 0.4 μm pore size) appropriate

Table 1 SEM–EDX mineralogy classes defined for respirable coal mine dust, and likely sources of particles [adapted from 12]

Mineralogy class	Abbreviation	Sources
Carbonaceous	C	Coal strata, organic matter in roof/floor rock strata, and diesel particulate matter
Mixed carbonaceous	MC	Coal, roof/floor rock strata
Aluminosilicates—kaolinite	ASK	Roof/floor rock strata
Aluminosilicates—other	ASO	Roof/floor rock strata
Carbonates	CB	Rock dusting products
Heavy minerals	HM	Metal sulfides/oxides in coal or rock strata, etc
Silica	S	Roof/floor rock strata
Other silicates	OS	Roof/floor rock strata
Other	O	Other minerals, biological particles, etc

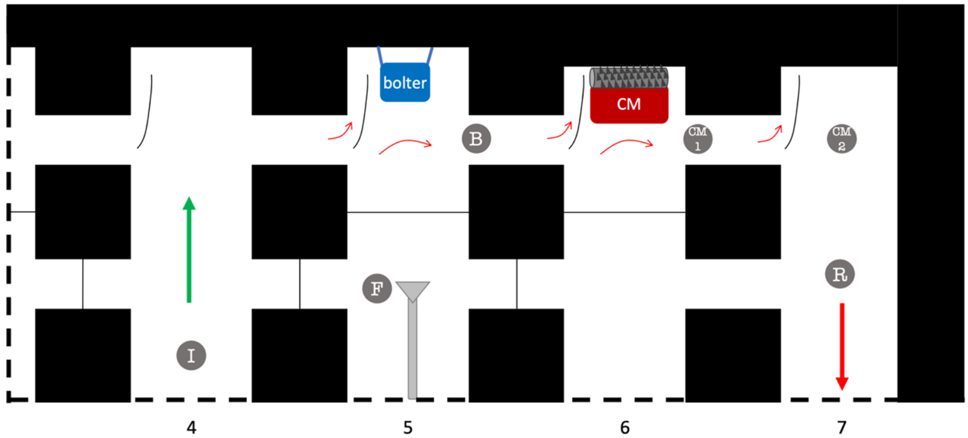

Fig. 1 Schematic of sampling locations in the study mine

for SEM–EDX and TGA, and the other two were polyvinyl chloride (PVC; 5 µm pore size) for gravimetric and FT-IR analysis. For each sample set, a metal frame was used to position the cyclone inlets parallel to one another. Samples were collected over 1–3 hr, depending on the expected dust concentration or the time required for the miner cut.

2.3 Dust Analysis

For the SEM–EDX analysis, a. circular 9-mm subsection was cut from the edge of one PC filter in each sample set, and sputter coated with Au/Pd. The samples were analyzed with a FEI Quanta 600 FEG environmental SEM (FEI, Hillsboro, OR) equipped with a Bruker Quantax 400 EDX spectroscope (Bruker, Ewing, NJ). A computer-controlled routine described by Johann-Essex et al. [16] was used to select, size, and classify about 500 particles (1–10 µm) per sample; classification criteria are shown in Table 2. The routine was run using Bruker's Esprit software (Version 1.9), and the following SEM settings: 1,000 × magnification, 12.5 mm working distance, 15 kV accelerating voltage, and 5.5 µm spot size.

To transform SEM–EDX particle data for each sample into relative mass abundance (%) per mineralogy class, the following procedure was used: to estimate the volume of each particle, its projected area (output by the Esprit software) was multiplied with its short dimension (determined using its width from the Esprit software and an assumed short-to-width dimension ratio, SW, as shown in Table 2). Then, the particle mass was estimated from its volume and an assumed value for specific gravity based on its mineralogy (Table 2). Finally, the mass for all particles in each class

Table 2 Classification criteria for each mineralogy class shown in Table 1 (reproduced from [12]). Assumptions for short-to-width dimension ratio (SW) and specific gravity (SG) are also shown

Atomic %									Particle size to mass assumptions	
Class	O	Al	Si	C	Mg	Ca	Ti	Fe	SW	SG
C	< 29	≤ 0.30	≤ 0.30	≥ 75	≤ 0.50	≤ 0.41	≤ 0.06	≤ 0.15	0.6	1.40
MC		< 0.35	< 0.35		≤ 0.50	≤ 0.50	≤ 0.60	≤ 0.60	0.6	1.40
ASK[1]		≥ 0.35 (≥ 39)	≥ 0.35 (≥ 32)		(< 15)	(< 8)	(< 13)	(< 13)	0.4 0.4	2.60
ASO[1]		≥ 0.35 (< 39)	≥ 0.35 (< 32)		(≥ 15)	(≥ 8)	(≥ 13)	(≥ 13)	0.4	2.60
OS[2]			≥ 0.33						0.4	2.60
S[3]			≥ 0.33						0.7	2.65
M		> 1%					> 1%	> 1%	0.7	4.96
CB	> 9				> 0.5%	> 0.5%			0.7	2.70

[1]To differentiate ASK from ASO, additional limits for Al, Si, Mg, Ca, Ti, and Fe are shown in parenthesis (normalized to exclude C and O).

[2]Additional limits for OS: Si/(Al + Si + Mg + Ca + Ti + Fe) < 0.5

[3]Additional limits for S: Al/Si < 1/3 and Si/(Al + Si + Mg + Ca + Ti + Fe) ≥ 0.5

was summed and mass % in the class was estimated based on the total mass for all particles analyzed in the sample.

For TGA analysis, the second PC sample from each set was used. Sample preparation and analysis followed the methods described by [14]. Briefly, dust was recovered from each PC filter by sonication in isopropyl alcohol (IPA); deposited into a clean, tared pan; and the IPA was allowed to evaporate to dryness (i.e., stable sample weight using the microbalance). Then, a thermogravimetric analyzer (TA Instruments, New Castle, DE) was used to track sample weight loss during a predefined thermal routine. Finally, a series of mass balance equations per [14] was applied to the weight loss data to apportion the sample mass into three fractions: coal, carbonates, and non-carbonate minerals.

The PVC filters were pre- and post-weighed using a microbalance (Sartorius MSE6.6S, Gottingen, Germany) to allow gravimetric determination of respirable dust mass concentration in each location (i.e., using recorded pump flow rate and sampling time).

The PVC filters were also used for direct-on-filter FT-IR analysis using an ALPHA II spectrometer (Bruker, Billerica, MA). For this, each filter was carefully removed from its cassette, placed into an FT-IR-compatible 4-piece cassette (Zefon International, Ocala, FL) and a 6-mm spot in the center of the filter was scanned using Bruker's OPUS software to produce the absorbance spectrum (4000 cm^{-1} to 400 cm^{-1} with a resolution of 4 cm^{-1}; 16 scans per sample). The spectrum was corrected for a blank PVC filter, then used to determine the massees (µg) of quartz and kaolinite based on their characteristic peaks at 800 cm^{-1} and 915 cm^{-1}, respectively. To convert measured peak areas to analyte mass, calibration models established by NIOSH (i.e., using pure quartz or kaolinite dust materials) were used. A data correction based on Miller et al. [17] was also applied to adjust for the fact that the study samples were collected in 2-piece cassettes, rather than 3-piece cassettes like NIOSH's calibration samples.

3 Results and Discussion

Table 3 summarizes the SEM–EDX, TGA, FT-IR, and gravimetric results from all 15 sets of respirable mine dust samples. Relative dust concentrations (determined gravimetrically) are generally consistent with expectations based on the conditions in specific sampling locations. The dust concentration was very low in the intake, higher near the roof bolter and feeder breaker, and was highest at the miner ventilation curtain mouth (CM1)—although there was wide variability in that location and no consistent trend with the application of the miner scrubber. Moving downwind from CM1, the dust concentration was reduced at CM2 and again at the R location. (It should be noted that the reported dust concentrations cannot be used to infer personal exposures; the study samples were collected over relatively short time periods in particular locations, not on individuals, and the results have not been corrected using the MRE-conversion factor used for compliance monitoring).

Mineralogy distributions determined by SEM–EDX are graphically displayed in Fig. 2. Results for the I, B, and F locations are generally consistent with results obtained in other central Appalachian mines (e.g., see [11, 12]). Respirable dust in the intake, while low in concentration, contains a variety of constituents, including those associated with the coal (C and MC particles) and rock strata (ASK, ASO, and S) and application of limestone rock dust (CB), which is required in US mines to mitigate explosibility hazards. (It is noted that C particles can be associated with diesel particulates in some mines, but the study mine does not operate any diesel equipment and so C particles in this mine are most likely coal.) Near the roof bolter, nearly all the dust appears to be sourced from the roof rock, whereas near the feeder breaker there is more coal content as expected.

Fig. 2 also indicates that just downwind from the continuous miner the influence of rock-strata sourced dust is very strong; and the relative height of rock strata being mined (about 1.6 m) was very high compared to the total mining height (about 2.4 m). Based on the SEM–EDX analysis, AS particles (ASK + ASO) account for 62–97%, and AS + S accounts for 66–100%, of all classified dust particles in the CM1 location across the four different miner cuts. (Again, no trend could be discerned with the operation of the miner scrubber.) Coal-strata sourced dust (C + MC) accounts for most of the other dust in the CM1 location, up to 11%. Moving downwind into the return airway, a consistent trend is observed wherein the proportion of AS + S is decreased and that of C + MC is increased. SEM–EDX analysis of respirable dust in other mines, particularly thin-seam mines, has also yielded inordinately high percentages of AS (or AS + S) dust near the production face with somewhat more C + MC in the return [10–12], however, the reason(s) for this trend have not been elucidated heretofore. One possibility is that very fine silicate dust being generated at the face interferes with the SEM–EDX analysis of larger dust particles such as coal. In samples with particularly high loading (e.g., CM1 samples from miner cuts 2 and 4), this could happen simply due to fine AS particles depositing on the sample filter in close proximity to other particles [12]. Interference could also occur if AS particles attach to the surface of other particles to form agglomerates.

Figure 3 shows the overall particle size distribution by mineralogy class (top), sampling location (middle), and for the predominant classes (C, MC, total AS, and S) in the CM1 and R locations downwind of the continuous miner (bottom). Across all six sampling locations, the variability in particle size can generally be summarized as

Table 3 Summary of SEM–EDX results for all 15 sets of respirable mine dust samples. Gravimetric results are presented as an average of the two PVC samples analyzed in a given set. (For the FT-IR quartz analysis, the I sample was < LOD.)

Location (miner cut, scrubber status)		Grav	SEM–EDX										TGA		FT-IR	
		Conc	number % (mass%)										mass %		mass %	
		(mg/m3)	C	MC	ASK	ASO	OS	S	HM	CB	O	Coal	CB	NCB	Q	K
I		< 0.1	35 (7)	18 (9)	6 (17)	15 (43)	2 (1)	10 (9)	1 (1)	12 (12)	2 (0)	77	1	22	-	71%
F		1.5	8 (3)	15 (8)	9 (10)	54 (64)	0 (0)	4 (7)	2 (2)	4 (7)	3 (0)	40	8	51	6	13
B		0.5	0 (0)	1 (0)	27 (18)	68 (77)	0 (0)	3 (5)	0 (0)	0 (0)	1 (0)	39	6	55	9	22
(Cut 1, OFF)	CM1	4.5	2 (0)	9 (4)	19 (16)	64 (71)	0 (0)	3 (5)	0 (1)	1 (3)	1 (0)	57	6	37	4	19
	CM2	2.4	25 (7)	24 (11)	17 (33)	25 (32)	0 (0)	8 (12)	1 (6)	0 (0)	1 (0)	18	7	74	5	21
	R	1.6	33 (12)	27 (13)	12 (24)	16 (25)	0 (0)	6 (14)	1 (1)	3 (12)	2 (0)	22	6	72	3	15
(Cut 2, ON)	CM1	9.6	0 (0)	3 (1)	35 (37)	59 (60)	0 (0)	3 (2)	0 (0)	0 (0)	0 (0)	42	1	57	6	20
	CM2	4.4	8 (2)	22 (7)	26 (23)	37 (50)	0 (0)	4 (5)	1 (1)	1 (12)	2 (0)	27	10	63	5	22
	R	1.2	20 (7)	26 (9)	19 (28)	25 (42)	0 (0)	5 (7)	1 (4)	2 (3)	1 (0)	23	6	71	4	24
(Cut 3, ON)	CM1	2.8	12 (3)	19 (6)	25 (36)	37 (42)	0 (0)	4 (8)	1 (2)	1 (2)	2 (0)	29	1	70	3	20
	CM2	1.1	25 (6)	28 (9)	13 (34)	21 (28)	0 (0)	6 (9)	0 (2)	4 (12)	3 (0)	43	8	49	2	22
	R	0.9	21 (8)	28 (10)	16 (29)	24 (35)	0 (0)	5 (7)	0 (3)	3 (8)	2 (0)	53	20	27	8	25
(Cut 4, OFF)	CM1	11.8	0 (0)	0 (0)	37 (29)	60 (70)	0 (0)	3 (2)	0 (0)	0 (0)	0 (0)	14	8	78	7	19
	CM2	7.5	1 (0)	1 (0)	30 (26)	67 (71)	0 (0)	1 (1)	0 (0)	0 (1)	0 (0)	14	5	81	7	20
	R	3	30 (11)	24 (9)	17 (27)	21 (32)	0 (0)	5 (12)	1 (5)	1 (3)	1 (0)	18	6	75	3	20

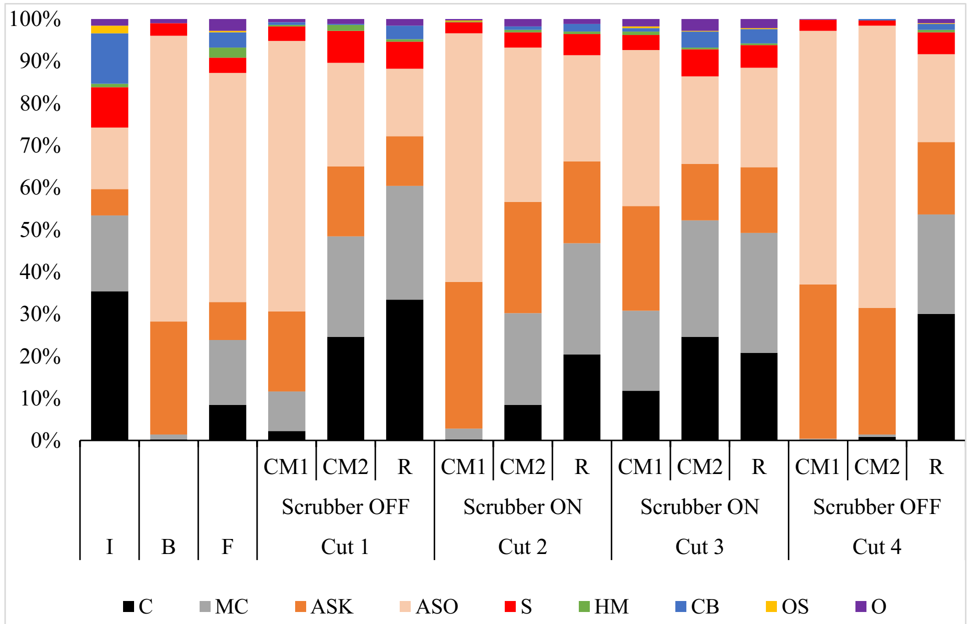

Fig. 2 Mineralogy distribution (number %) of respirable dust particles in each sample set

C, MC < < S < < CB, AS. This agrees with observations in other mines where the same SEM–EDX classification criteria were used: S particles are typically finer than other major minerals (AS and CB), and particles classified as C or MC are typically finer than particles classified as minerals [12]. With respect to sampling location, variability in size can be summarized as CM1, CM2, R, B < F < I. Again, this is consistent with expectations; the finest particles occur nearest to active cutting and drilling.

To gain further insight on the dust profile moving downwind from the continuous miner, the bottom plot of Fig. 3 shows particle size distributions for the CM1 and R locations during Cut 1 (results were similar for Cuts 2 and 4). Dust in each class appears relatively finer at CM1 than at R. The

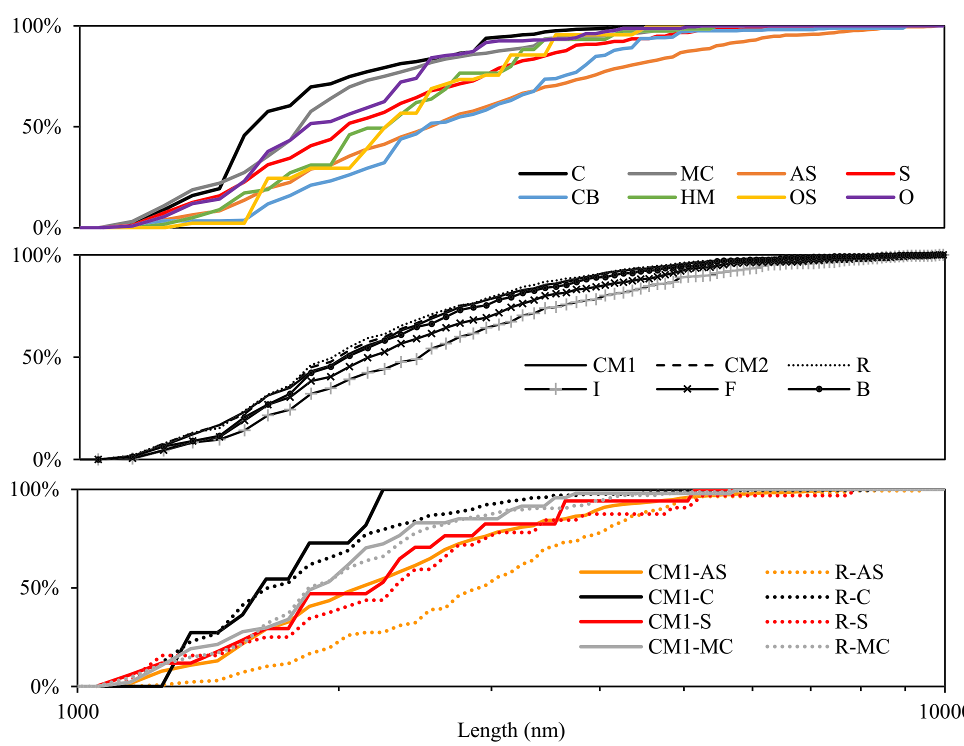

Fig. 3 Cumulative size distributions of respirable dust particles by mineralogy class (top), sampling location (middle), and for the C, MC, AS (total), and S classes in the CM1 and R locations during miner Cut 1 (bottom). To construct the top plot, results were averaged across all six sampling locations; since four samples (one per cut) were analyzed in CM1 and R, results from each cut were first averaged to get a single dataset for each of these locations. For the middle plot, all particles in a given location were averaged

change in AS particle size distribution is most evident and is of particular interest since the finest AS particles near the mine face could be influencing the SEM–EDX classification of other particles. Notably, while the particle size distribution in each class appears to become coarser moving away from the dust source, the shift in apparent mineralogy distribution illustrated in Fig. 2 (from mostly AS to more C + MC) causes the overall size distribution of the dust in the CM1, CM2, and R locations to be quite similar. This is consistent with expectations for respirable sized particles under high velocity conditions.

Fig. 4 offers additional insight on the trends observed from the SEM–EDX results moving from the CM1 to CM2 to R locations. The plot shows the average mass distribution of dust constituents (i.e., across all four miner cuts) based on each analytical method used in this study. Whereas the SEM–EDX results indicate a gradual shift in the coal to mineral ratio (i.e., driven mostly by reduction in ASO and increase in C + MC), the TGA results suggests the ratio is fairly stable. The FT-IR results indicate that mass percentages of quartz (i.e., silica) and kaolinite are stable too, and generally in good agreement with the SEM–EDX-derived values—consistent with findings in another recent study that looked at respirable dust samples from 15 additional mines [18]. These observations support the hypothesis that fine aluminosilicate particles (primarily ASO) might be interfering with SEM–EDX classification of some coal particles. In essence, the mass-based method (TGA) is seeing such coal particles as coal, but the particle-based method sees them as mineral due to sufficient Al and Si elemental content per the classification criteria shown in Table 2.

Given the trends in dust concentration in the CM1, CM2, and R locations (Table 3), high sample loading is one obvious factor that could lead to interference of ASO particles with coal dust classification by SEM–EDX. This factor should be controlled in future studies where microscopy analysis is planned (e.g., by reducing total sampling time in areas with high dust concentrations.) However, presence of coal-mineral micro-agglomerates (e.g., as shown in Fig. 5) might also be a factor. Indeed, even for the samples in the R location (which should be less influenced by loading effects), the SEM–EDX data predicts less coal and more (rock-strata sourced) mineral content than the TGA. Notably, a separate study by the authors recently explored the possibility of micro-agglomerates in respirable coal mine dust [19]. It found that such particles do commonly occur in samples collected using the same methods used here (i.e., standard respirable dust sampling with an air pump and cyclone), and that their formation is probably not only due to the sampling process itself (i.e., micro-agglomerates likely exist in the mine atmosphere). The formation mechanisms and persistence of micro-agglomerates might be important to both exposure assessment and development of improved dust controls.

4 Conclusions

In this case study, respirable coal mine dust constituents were generally consistent with expectations based on similar analysis of samples collected in other central Appalachian

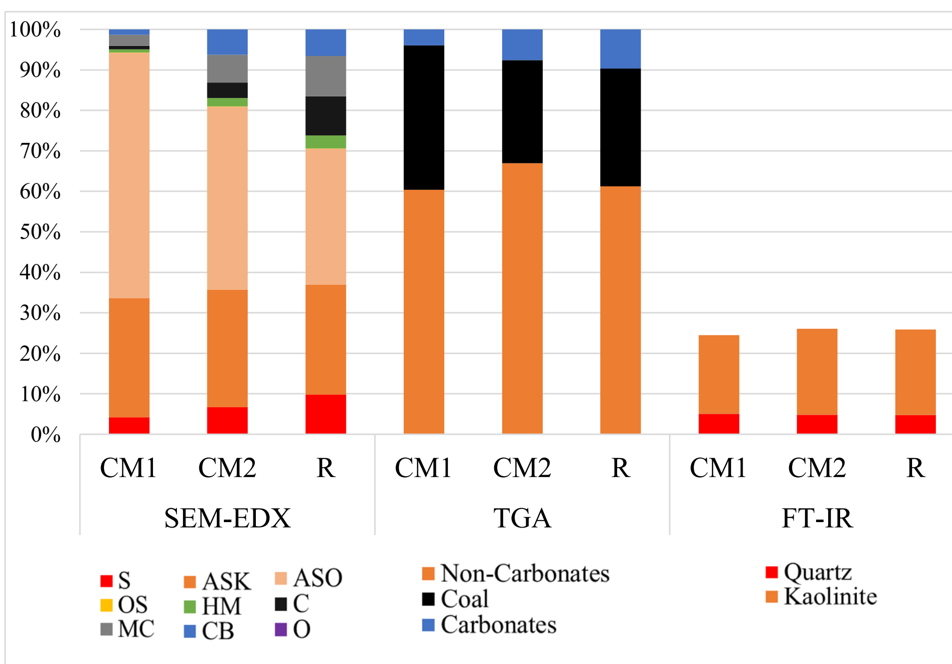

Fig. 4 Average distribution of respirable dust mass (%) in the CM1, CM2, and R locations derived from each analytical technique. To construct this plot, results were averaged across the four continuous miner cuts. SEM–EDX, TGA, and FT-IR data were used directly from Table 3; the SEM–EDX data shown in Table 3 display the mass % estimated values in each mineralogy class (i.e., S = silica, ASK = kaolinite, ASO = other aluminosilicates, OS = other silicates, HM = heavy minerals, C = carbonaceous, MC = mixed carbonaceous, CB = carbonates, and O = other)

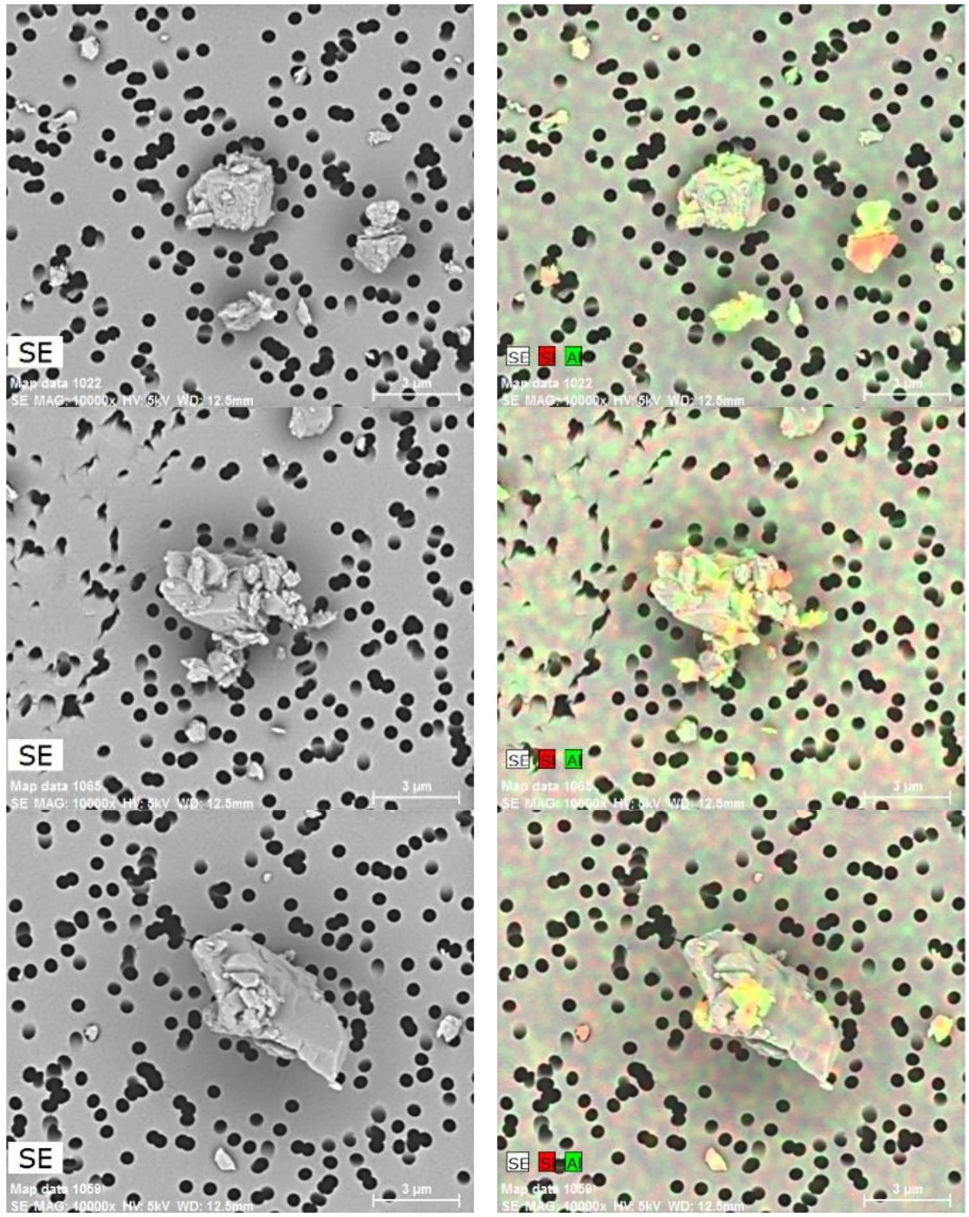

Fig. 5 Examples of micro-agglomerates imaged using SEM–EDX in the CM2 sample collected during Cut 3. For each image (captured at 5 kV and 10,000 × magnification), an elemental map is also shown to highlight concentrations of silicon (red) and aluminum (green); maps clearly indicate that the agglomerate in center of each image includes a relatively large coal particle (i.e., free from Si and Al above noise background) with finer mineral particles on its surface

mines [12, 18]. The relatively high abundance of minerals in the dust (mostly aluminosilicates, some silica) is attributed to the high proportion of rock strata within the total mining height; for the current study, the coal seam thickness accounted for just 33%, on average, of the total mining height during sampling.

An effort was made investigate the effect of the continuous miner scrubber on respirable dust characteristics by sampling during periods when the scrubber was operating versus not operating. However, no discernable trends in particle size or specific constituents could be observed in relation to the scrubber status—probably due to variability in geologic strata across the four miner cuts sampled. Further investigation is needed to understand the effect of scrubbers (and other dust controls) on dust characteristics.

Simultaneous sampling at three locations downwind of the miner enabled investigation of the respirable dust profile as it is transported toward the return. The SEM–EDX data indicated the relative abundance of fine particles which is somewhat higher nearest to the mine face (i.e., where the primary dust generation activity is occurring). This data also suggested a non-intuitive shift in dust constituents (i.e., very high mineral content near the face and increasing coal content moving further downwind). However, while additional analysis of the dust samples using TGA confirmed high mineral content overall downwind the miner, it did not indicate a substantial shift in dust constituents—nor did additional analysis by FT-IR. Rather, for samples nearer to the mine face, it appears very fine aluminosilicate particles caused some mis-classification of coal dust by the SEM–EDX method. High sample loading can contribute to EDX interference between particles and should be avoided when collecting samples for microscopy. Results presented herein suggest that other factors, such as agglomeration between coal and fine aluminosilicates, might also be important. Future studies that seek to characterize respirable dust should consider utilizing multiple analytical methods to allow interrogation of such influential factors. Moreover, sampling locations should be carefully selected to suit specific research objectives (e.g., understanding dust sources versus relating results to regulatory standards.)

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Declarations

Conflicts of Interest The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, or in the decision to publish the results.

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