Characterization of Particle Size and Composition of Respirable Coal Mine Dust

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Abstract: Respirable coal mine dust (RCMD) particles, particularly the nano-sized fraction (<1 µm) of the RCMD if present, can cause severe lung diseases in coal miners. Characterization of both the particle size and chemical composition of such RCMD particles remains a work in progress, in particular, with respect to the nano-sized fraction of RCMD. In this work, various methods were surveyed and used to obtain both the size and chemical composition of RCMD particles, including scanning electron microscopy (SEM), scanning transmission electron microscopy (S-TEM), dynamic light scattering (DLS), and asymmetric flow field-flow fractionation (AsFIFFF). It was found that the micron-sized fraction (>1 µm) of RCMD particles collected at the miner location, from an underground coal mine, contained more coal particles, while those collected at the bolter location contained more rock dust particles. Two image processing procedures were developed to determine the size of individual RCMD particles. The particle size distribution (PSD) results showed that a significant amount (~80% by number) of nano-sized particles were present in the RCMD sample collected in an underground coal mine. The presence of nano-sized RCMD particles was confirmed by bulk sample analysis, using both DLS and AsFIFFF. The mode particle size at the peak frequency of the size distribution was found to be 300–400 nm, which was consistent with the result obtained from SEM analysis. The chemical composition data of the nano-sized RCMD showed that not only diesel particles, but also both coal and rock dust particles were present in the nano-sized fraction of the RCMD. The presence of the nano-sized fraction of RCMD particles may be site and location dependent, and a detailed analysis of the entire size range of RCMD particles in different underground coal mines is needed.

Keywords: respirable coal mine dust; particle size; mineral composition

1. Introduction

In the coal mining industry, respirable coal mine dust (RCMD) is generated during mining operations [1]. These airborne dust particles are comprised of a respirable fraction (<10 µm), which can be inhaled by miners and deposited in the distal airways and gas-exchange region of the lung. Overexposure to RCMD particles can cause serious health problems, including coal workers’ pneumoconiosis (CWP) [2]. CWP is a lung disease that can be fatal in its most severe form, and progresses even after exposure is removed. Exposure to an excessive amount of crystalline silica can cause silicosis [1,3].

The Federal Coal Mine Health and Safety Act of 1969 established the coal mine dust standard of 2.0 mg/m³ at the maximum 5% allowable percentage of silica. Recently, the Mine Safety and Health Administration (MSHA) in the United States set a lower respirable dust standard of 1.5 mg/m³ for underground coal mining operations. Compliance with this standard is monitored through periodic collections of occupational dust samples. The
RCMD generated in underground coal mines has many sources, including dust particles generated by coal seams and rocks adjacent to coal seams, rock dust (generally pulverized limestone dust) for the control of explosions in mines [4], and other dust particles associated with various mining activities. Therefore, RCMD particles can be comprised of various components, such as coal, silica, aluminosilicate, calcite, dolomite, diesel particulate matter, and others [5]. The compliance-driven approach has led to a significant reduction in incidence of lung disease associated with occupational exposure to RCMD among U.S. coal mine workers. However, since 2000, the number of cases of miners with CWP, and having 25 years or more of exposure, has continued to increase [6]. Past studies implied that US coal miners are being exposed to excessive amounts of respirable crystalline silica [3].

Accurate and real-time sampling is essential for compliance, daily safety checks, and dust control optimization. The conventional sampling approach uses a gravimetric-based device that collects the respirable fraction of the coal mine dust particles. This sampling device contains a battery-powered pump, a cyclone that separates the respirable (<10 µm) fraction from the total airborne dust, and a filter cassette that collects the respirable dust particles on filters. The collected filter samples are pre-weighed and post-weighed to obtain the RCMD mass collected over the total sampling period, which can be used to determine a miner’s exposure over the sampling period. Multiple gravimetric samplers might be needed to minimize the measurement errors, and the averaged data is used for reporting purposes. Various post-sampling characterizations have been conducted. For instance, quartz content is regulated by MSHA. To determine quartz content, the collected RCMD samples are heat treated to decompose organic matter and coal, and the residue minerals are analyzed using X-ray diffraction (XRD) [7,8]. Scanning electron microscopy (SEM) enables the characterization of individual dust particles on the filters [9,10]. Infrared spectroscopy can also be used for quartz analysis. This method measures the absorbance at 800 cm\(^{-1}\) or 915 cm\(^{-1}\) for a given sample, and obtains its quartz content by comparison to a calibration curve for standard quartz samples [11].

A continuous personal dust monitor (CPDM) is a real-time sampler, which was originally developed by NIOSH and approved by MSHA [12]. In the United States, a CPDM is used for compliance purposes. The CPDM uses a tapered-element oscillating microbalance (TEOM) to obtain a real-time gravimetric-based measure of respirable dust concentration. The CPDM provides coal workers with real-time information on dust concentration in each shift. This information can be used by coal workers to monitor dust exposure, and aid miners’ decision-making in reducing their exposure to RCMD by making appropriate changes to their work activities. It has been found that the results obtained from the CPDM were consistent with those obtained using the conventional gravimetric sampler [13]. Unfortunately, the dust samples collected using a CPDM cannot be used for quartz and other analyses. Furthermore, the gravimetric data from CPDMs cannot be used to evaluate the source of dust particles in the underground coal mines.

Gathering information on potentially important factors affecting miners’ health will be essential to meet the current regulatory requirements. It was previously shown that the – 200 mesh fraction of the dust particles in coal mines in the United States was about 29–37% [14], and the percentage of nano-sized dust particles in the RCMD collected at the bolter location was over 60% [5]. A recent study published by NIOSH showed that 10–23% of quartz dust particles were concentrated in the nano-sized fraction of RCMD particles [15]. Information on particle size and chemical composition of RCMD particles, particularly the nano-sized fraction of RCMD particles, in underground coal mines is essential for developing dust control and mitigation strategies. It has been documented that the nano-sized particles can be more adverse to lung health than micron-sized dust particles [16]. Herein, we surveyed a number of characterization methods for characterizing both the particle size and chemical composition of RCMD particles. The collected coal dust samples were characterized to determine both the size and composition of individual dust particles using scanning electron microscopy (SEM) and scanning transmission electron microscopy (S-TEM). The particle size distribution (PSD) of the dust particles, particularly the nano-
sized fraction of the RCMD particles, was determined using dynamic light scattering (DLS) and asymmetrical flow field-flow fractionation (AsFIFFF).

2. Materials and Experimental Methods

2.1. Sampling

Coal mine dust samples from an underground coal mine were collected using gravimetric dust samplers. Dust samples were taken from a number of locations, such as at the intake, return, bolter, and miner locations. The dust sampler used a 10 mm Dorr-Oliver cyclone to separate the respirable fraction (<10 µm) from the coarse fraction (>10 µm) of the dust particles. The respirable fraction was deposited on a filter, while the coarse fraction was collected at the bottom of the cyclone. The cyclone pump operated at an air flow rate of 2 L per minute. Various filters were used, including 0.8 µm polycarbonate (PC) filters, 0.8 µm silver filters, and 5 µm PVC filters. To retain the RCMD particles, particularly the nano-sized fraction of the RCMD particles, filters with smaller pore sizes, such as the 0.8 µm PC and 0.8 µm silver filters, were preferred. The use of 0.8 µm silver filters was not ideal for identifying individual RCMD particles due to poor image contrast between the dust particles and the filter substrates. Therefore, 0.8 µm PC filters were used in this study to determine both the particle size and chemical composition of RCMD particles. Filters were pre- and post-weighed to determine mass gain, which was used to determine dust exposure over the sampling period.

2.2. Scanning Electron Microscopy (SEM)

2.2.1. Imaging and Chemical Composition Analysis

Imaging and elemental composition analyses of dust particles on the filters were conducted using scanning electron microscopy (SEM). PC filters were carefully removed from the cassette. Two 6 × 6 mm² pieces were cut from each sample. One piece was cut from the edge, and the other piece was from near the center of the filter. This was to ensure accuracy of the results since the RCMD particles were not evenly distributed across the surface of the filter. The cut filter pieces were attached to an aluminum pin using double-sided tape. The particle size and chemical composition of individual dust particles on the filters were determined using SEM coupled with energy-dispersive X-ray spectroscopy (EDX). Prior to the SEM characterization, the filter specimens were coated with a 5–10 nm thick layer of Pt, and elemental mapping was performed in a Philips XL40 Environmental Scanning Electron Microscope (ESEM) (Amsterdam, The Netherlands) under high-vacuum conditions. Images were obtained under the back scattered electron (BSE) mode at a 15–17 kV accelerating voltage.

One issue with the use of PC filters is that the emitted X-ray signal from the substrate (i.e., the PC filter) interferes with that from the deposited coal dust particles, since the chemical compositions of both PC and the dust particles are similar. Therefore, in this study, the EDX elemental mapping was conducted through the secondary electron (SE) mode at a 10–12 kV accelerating voltage. The choice of a 10–12 kV acceleration voltage was to ensure that the EDX spectra collected at the 10–12 kV accelerating voltage reflected the actual composition of the dust particles on the surface. In addition, the EDX analysis enabled a characterization of micron-sized dust particles. For the nano-sized fraction of the dust particles, an accurate determination of their chemical composition can be challenging.

Particles were categorized into seven different types, including coal, diesel, calcite, dolomite, quartz, kaolin, illite, and others. Coal particles primarily consisted of carbon (~80% by weight). Diesel particles had the same composition as coal, but exhibited an amorphous structure. Calcite particles contained at least 3% by weight calcium (Ca), in addition to carbon and oxygen. Dolomite particles contained at least 3% by weight magnesium (Mg) and Ca combined. Quartz particles contained at least 1% by weight of silicon and no Ca and Mg. Both kaolin and illite particles were identified as aluminosilicate particles that contained at least 1% by weight of both Si and Al. The others were particles that contained heavy metals, such as iron.
2.2.2. Image Analysis

The size of individual dust particles deposited on the filter substrate was determined using FIJI software (ImageJ, National Institute of Health). The first image processing procedure was done by manually circling and enumerating particles [17]. The area parameter was determined, and the equivalent disc diameter was calculated from the projected particle area. Particles with different textures and shapes were detected. For aggregates of smaller particles or in cases where smaller particles were deposited on larger particles, clusters of particles with no visible boundary between particles were treated as one particle. A second image processing procedure counted dust particles with diameters of ~100 nm and above. This was achieved by thresholding the image, then selecting and numbering the particles.

2.3. Particle Size Analysis

Particle size distribution of the dust samples was measured using dynamic light scattering (DLS) and asymmetrical flow field-flow fractionation (AsFIFFF). Dust particles on PC filters were first dispersed in an aqueous surfactant solution. A Zetasizer Nano instrument (Malvern, PA, USA) was used for the DLS measurements. The Zetasizer Nano yielded information on the estimated particle size distribution of nano-sized dust particles in the batch mode.

Asymmetrical flow field-flow fractionation (AsFIFFF) is a particle separation and elution technique. In AsFIFFF, the sample fractionation takes place in a thin, flat, open channel (no stationary phase) by applying an external physical force to an incoming flow of sample particles suspended in a solution. Particles are eluted by a laminar flow perpendicular to the direction of the external force, and smaller particles in the sample elute first with the faster streams of the laminar flow; and the larger particles elute later [18]. AsFIFFF analyses were performed using a Postnova AF2000 system (Landsberg, Germany) with UV detection at 254 nm. Filter samples were suspended in 2 mL of a surfactant solution, and 100 µL samples were injected into the channel. The sample contained larger particles that were settled, and excluded from the AsFIFFF analysis during the injection step. The results obtained from the AsFIFFF and DLS procedures were compared with those obtained from the SEM image analysis.

2.4. Scanning Transmission Electron Microscopy (S-TEM)

The elemental composition of the nano-sized fraction of the dust particles was determined using scanning transmission electron microscopy (Titan Themis 200 S-TEM, FEI). The electron micrographs and energy dispersive X-ray spectroscopy maps were obtained using an aberration corrected scanning transmission electron microscope operated at 200 KeV. The point resolution in the aberration-corrected mode was 0.08 nm. The microscope was fitted with a SuperXTM X-ray detector, which is a combination of four detectors for fast X-ray mapping in the STEM mode. For extremely stable (mechanical and e-beam interaction) samples, atomic resolution maps are possible. For the present experiment, nm resolution EDX maps were taken with an average beam current of 100 pA. The size of each map was 512 × 512 pixels, and a 50 µs/pixel dwell time was used for collecting the signal. All maps were generated by summing over 10 frames. The drift correction during data collection and subsequent analysis was performed using Velox software.

3. Result and Discussion

3.1. Particle-by-Particle Characterization of Micron-Sized RCMD

Figure 1 shows a SEM analysis result of RCMD particles collected at the bolter location in an underground coal mine. The particle size information of individual micron-sized dust particles was obtained using an image processing protocol. Table 1 shows both the equivalent disc diameter and chemical composition of individual dust particles as labelled on the SEM image in Figure 1a.
Figure 1. Particle-by-particle size and composition analyses of respirable coal mine dust particles collected on a filter. (a) An original SEM image; (b) a processed SEM image with micron-sized dust particles circled. The numbers labelled in (a) are individual micron-sized particles with atomic composition shown in Table 1.

Table 1. The size, atomic composition, and mineral type of individual respirable coal mine dust (RCMD) particles as identified in Figure 1a.

| Label | C (%) | O (%) | Mg (%) | Al (%) | Si (%) | S (%) | Ca (%) | Fe (%) | Size (µm) | Type      |
|-------|-------|-------|--------|--------|--------|-------|--------|--------|-----------|-----------|
| 1     | 45.11 | 38.86 | 0.38   | -      | -      | -     | 15.65  | -      | 1.69      | Calcite   |
| 2     | 23.50 | 41.51 | 0.08   | -      | -      | -     | 34.91  | -      | 2.76      | Calcite   |
| 3     | 63.62 | 30.19 | -      | 2.88   | 3.31   | -     | -      | -      | 3.58      | Kaolin    |
| 4     | 80.10 | 19.90 | -      | -      | -      | -     | -      | -      | 1.24      | Coal      |
| 5     | 52.96 | 37.99 | 2.53   | -      | -      | -     | 1.09   | 5.42   | 1.12      | Dolomite  |
| 6     | 44.56 | 41.07 | -      | 2.31   | 12.06  | -     | -      | -      | 1.08      | Kaolin    |
| 7     | 38.53 | 46.53 | 5.82   | -      | -      | -     | 9.13   | -      | 1.86      | Dolomite  |
| 8     | 77.49 | 22.51 | -      | -      | -      | -     | -      | -      | 1.12      | Coal      |
| 9     | -     | 67.20 | 12.90  | -      | -      | -     | 19.91  | -      | 1.40      | Dolomite  |
| 10    | 75.90 | 24.10 | -      | -      | -      | -     | -      | -      | 2.05      | Coal      |
| 11    | 58.02 | 33.92 | 1.37   | -      | -      | -     | 6.68   | -      | 2.50      | Dolomite  |
| 12    | 40.57 | 46.43 | 5.13   | -      | -      | -     | 7.87   | -      | 1.00      | Dolomite  |
| 13    | 80.40 | 19.60 | -      | -      | -      | -     | -      | -      | 1.30      | Coal      |

As shown, there were 13 micron-sized dust particles with equivalent disc diameters of above 1.0 µm over an area of 45 × 60 µm. Each individual coal dust particle was classified as (1) coal, (2) diesel, (3) calcite, (4) dolomite, (5) illite, (6) kaolin, (7) quartz, or (8) others. Since both the coal and diesel particles have a similar chemical composition, the classification between the coal and diesel particles was based on the difference in morphology, since the diesel particles exhibited amorphous structure with tiny nano-size particles agglomerated together [19]. As shown, 4 out of 13 particles were coal particles, and 7 out of 13 particles were either calcite or dolomite particles. The equivalent disc diameters of these 13 dust particles were in the range of 1.00 µm to 5.00 µm. Note that from the SEM image, there was a significant amount of nano-sized dust particles on the filter. The particle size information of these nano-sized particles was determined using a second image processing procedure, as described in the experimental section.

Table 2 compares dust compositions of RCMD particles collected at four different locations within an underground coal mine, including the intake, bolter, miner, and return locations.
locations. These data summarize the results from 30 SEM images at each sampling location. As shown, dust particles collected at the bolter location consisted of ~45% coal particles, ~30% calcite and dolomite particles, and ~16% aluminosilicate particles. On the contrary, dust particles collected at the miner location contained a higher percentage (72%) of coal particles, with only ~11% of calcite and dolomite combined. The quartz content of the samples collected at the miner location was higher than the quartz content of those samples collected at the bolter location. The composition of the RCMD particles may vary with mines in different locations.

Table 2. Comparison of dust composition of RCMD particles collected at different locations within the mine.

| Location | Coal (%) | Diesel (%) | Calcite (%) | Dolomite (%) | Illite (%) | Kaolin (%) | Quartz (%) | Others (%) |
|----------|----------|------------|-------------|--------------|------------|------------|------------|------------|
| Intake   | 66.67    | 0.00       | 7.94        | 14.29        | 3.17       | 6.35       | 1.59       | 0.00       |
| Bolter   | 45.50    | 3.15       | 3.60        | 26.58        | 5.86       | 10.36      | 2.25       | 2.70       |
| Miner    | 71.78    | 0.00       | 3.47        | 8.42         | 5.94       | 3.47       | 5.45       | 1.49       |
| Return   | 53.43    | 0.00       | 11.34       | 21.49        | 6.57       | 5.37       | 3.88       | 2.39       |

The RCMD generated in the underground coal mine had many sources, including dust particles generated by the coal seam and rock layers adjacent to the coal seam, rock dust (i.e., pulverized limestone dust) for explosion control in the mine, and any other particles associated with the mining activities. Diesel particulate matter (DPM) particles were mainly associated with the use of diesel-powered equipment. Both the calcite and dolomite were probably from rock dust products. Rocks adjacent to the coal seam had elevated concentrations of aluminosilicate. The quartz particles were present in the coal seam.

To better understand the correlation between particle mineralogy and particle size, the correlation between the chemical composition and particle size was investigated. Figure 2 shows particle size distribution for individual types of dust particles including coal, diesel, calcite and dolomite, aluminosilicate, and quartz for micron-sized particles. As shown, there was no clear correlation between particle size and chemical composition, at least for the micron-sized fraction of the RCMD particles, except for rock dust particles. As shown, both coal and aluminosilicate particles were evenly distributed in the size range between 1 µm to 5 µm. However, the rock dusts, i.e., dolomite and calcite, were concentrated in the finer fraction. There were limited amounts of quartz particles found at the bolter location, and therefore no conclusion can be reached for the quartz particles.

3.2. Particle Size Distribution

Particle size distribution (PSD) was determined from the SEM images using the second image processing procedure, as described in the experimental section. Figure 3a shows a SEM image at 1000× resolution, and Figure 3b shows the processed SEM image with dust particles circled. All dust particles on the filter were counted and summarized. Figure 3c shows the particle size distribution of the dust particles. The size histogram data shows that the mode particle size was found to be 200–300 nm. By number, 85% of the RCMD particles were nano-sized particles with equivalent disc diameters of below 1 µm.
3.2. Particle Size Distribution

Particle size distribution (PSD) was determined from the SEM images using the second moment method. As shown, there was no clear correlation between particle size and chemical composition, at least at the level of mineralogical phases. Figure 2 shows particle size distribution for individual types of dust particles including coal, aluminosilicate, calcite and dolomite, and quartz. Over 2000 particles were counted. All particles were detected and summarized. Figure 4 shows the particle size histogram data with dust particles circled. All dust particles on the filter were counted and summarized.

Figure 2. Relationship between mineral composition and particle size of a micron-sized fraction of RCMD particles collected at the bolter location.

3.3. Particle Size Analysis of Nano-Sized Fraction of RCMD

Figure 3c shows the particle size distribution of the dust particles. The size histogram data with dust particles circled. All dust particles on the filter were counted and summarized. Figure 4 shows the particle size histogram data with dust particles circled. All dust particles on the filter were counted and summarized.

As shown, the mode particle size was approximately 230 nm. Approximately 75% of the RCMD particles collected at the bolter location. Over 2000 particles were counted. All particles were detected and summarized. Figure 4 shows the particle size histogram data with dust particles circled. All dust particles on the filter were counted and summarized.

To obtain statistically significant size data for the dust particles collected on the filter sample, over 30 SEM images were taken on both the center and edge pieces of the filter. All particles were detected and summarized. Figure 4 shows the particle size histogram of the RCMD particles collected at the bolter location. Over 2000 particles were counted. As shown, the mode particle size was approximately 230 nm. Approximately 75% of the RCMD particles were at an equivalent disc diameter of below 1 µm.
3.2. Particle Size Distribution

Particle size distribution (PSD) was determined from the SEM images using the second image processing procedure, as described in the experimental section. Figure 3a shows a SEM image at 1000× resolution, and Figure 3b shows the processed SEM image with dust particles circled. All dust particles on the filter were counted and summarized. Figure 3c shows the particle size distribution of the dust particles. The size histogram data shows that the mode particle size was found to be 200–300 nm. By number, 85% of the RCMD particles were nano-sized particles with equivalent disc diameters of below 1 μm.

Figure 3. Particle size analysis of individual dust particles collected on the filter. (a) An original SEM image, (b) a processed SEM image with individual dust particles labelled, and (c) particle size distribution of the dust particles.

To obtain statistically significant size data for the dust particles collected on the filter sample, over 30 SEM images were taken on both the center and edge pieces of the filter. All particles were detected and summarized. Figure 4 shows the particle size histogram of the RCMD particles collected at the bolter location. Over 2000 particles were counted. As shown, the mode particle size was approximately 230 nm. Approximately 75% of the RCMD particles were at an equivalent disc diameter of below 1 μm.

Figure 4. Particle size distribution of RCMD particles collected at the bolter location.

3.3. Particle Size Analysis of Nano-Sized Fraction of RCMD

To confirm the presence of nano-sized dust particles in the sample, the dust samples were analyzed for particle size using both the DLS and AsFIFFF methods. Figure 5 shows the DLS results with the peak particle size data shown in Table 3. The DLS data suggests the presence of the nano-sized coal dust particles in all three samples. Small peaks indicating the presence of larger particles were observed in samples from both the miner and bolter locations. Particles from the miner location had hydrodynamic diameters ($d_h$) in the 105–1000 nm range, with a peak at 405 nm, and a few particles in the range of 4000–6500 nm (peak at 5560 nm). Dust particles from the bolter location showed a relatively wider distribution at 60–1100 nm and 1200–6500 nm, with peaks at 200 nm and 1200 nm, respectively. A bimodal distribution was observed for the samples obtained from the feeder location, with $d_h$ in the ranges of 40–100 nm and 110–600 nm, and peaks at 62 and 327 nm, respectively.

It must be noted that batch mode DLS data need to be interpreted with caution as they are most accurate for dilute, mono-dispersed spherical particles, and the intensity-based DLS data are biased towards the larger sized particles. Thus, a small population of larger sized particles would result in larger peaks at the micron-sized range. The low Z-average hydrodynamic diameters obtained for all three samples suggested the presence of a high population of nanoparticles in all three samples.

Figure 6 shows the size distribution, as determined by AsFIFFF, of the RCMD particles obtained from three different coal dust samples collected at the feeder, miner, and bolter locations. All three samples showed broad size distributions. The results suggest the presence of slightly coarser RMCD particles at the miner location compared to those collected from the bolter. The results also suggest the presence of the finest RCMD particles at the feeder location, with a peak particle size of 180 nm. Unlike DLS, which is conducted in batch mode, AsFIFFF is a separation and elution technique, where each sample population is detected separately, minimizing the detection interference between sample segments. The results from both the DLS and AsFIFFF experiments were consistent with those obtained with the SEM/EDX analysis, confirming the presence of nano-sized particles in the coal dust samples.
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Figure 5. Dynamic light scattering (DLS) size distribution of coal mine dust samples collected at the miner, bolter, and feeder locations. The results from three replicates are colored differently.

Table 3. Summary of the intensity-based size analysis data obtained by dynamic light scattering for the samples taken at three locations in the underground coal mine (\( d_h \): hydrodynamic diameter).

| Samples | Peak #1 | Peak #2 | Peak #3 |
|---------|---------|---------|---------|
|         | \( d_h \) (nm) | % Intensity | \( d_h \) (nm) | % Intensity | \( d_h \) (nm) | % Intensity |
| Miner   | 405     | 98%     | 5560     | 2%      | -            | -          |
| Bolter  | 200     | 42%     | 858      | 52%     | 4816         | 6%         |
| Feeder  | 62      | 10%     | 327      | 90.4%   | -            | -          |
Figure 6. Particle size distribution, as determined using asymmetric flow field-flow fractionation (AsFIFF), of coal mine dust particles collected at different locations within a mine.

3.4. Mineralogy of Nano-Sized RCMD

Characterization of the nano-sized fraction of the RCMD particles was attempted using high-angle annular dark-field imaging and energy-dispersive X-ray spectroscopy in the S-TEM. The collected dust particles were prepared by transferring the sample directly to a copper grid for TEM characterization. Figure 7 shows a TEM image of diesel particles. Figure 8 shows TEM and EDX images of three nano-sized RCMD particles excluding diesel particles. The sizes of these three nano-sized particles were in the range of 100–200 nm. Table 4 shows the elemental composition of the individual RCMD particles. Based on the atomic fraction data, particle #1 was a coal particle. Particle #2 was dolomite, and particle #3 was calcite. Both particles #2 and #3 were probably from rock dust. Evidently, there were non-diesel dust particles present in the nano-size fraction of the RCMD.

Table 4. Atomic fraction of individual elements for the three nano-sized particles shown in Figure 8.

| Particle | C (%) | O (%) | Mg (%) | Ca (%) | Type   |
|----------|-------|-------|--------|--------|--------|
| Particle 1 | 93.14 | 6.23  | 0.32   | 0.21   | Coal   |
| Particle 2 | 26.73 | 53.72 | 11.63  | 7.86   | Dolomite |
| Particle 3 | 43.21 | 43.47 | 1.48   | 11.83  | Calcite |

Figure 7. Transmission electron microscopy (TEM) images of diesel particles in the sample collected at the bolter location.
Figure 7. Transmission electron microscopy (TEM) images of diesel particles in the sample collected at the bolter location.

Figure 8. TEM images of nanosized particles collected at the bolter location. The atomic composition of individually labelled RCMD particles can be found in Table 4.

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| Particle 3 | 43.21 | 43.47 | 1.48   | 11.83  | Calcite    |

It should be noted that the characterization of a few of the nano-sized dust particles was achieved using S-TEM in this study, but the characterization of hundreds of individual RCMD particles from a sample can be challenging. In the future, separation of RCMD dust samples into different size ranges, followed by chemical analysis and material composition analysis may be desirable.

4. Summary and Conclusions

Characterization of respirable coal mine dust (RCMD) particles from an underground coal mine was conducted, including examination of both the micron-sized and nano-sized fractions of the respirable coal mine dust (RCMD). The dust samples were collected using the gravimetric sampling method. Individual dust particles deposited on the filter were characterized for chemical composition using scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDX). The SEM images were analyzed using ImageJ image processing software to obtain the particle size distribution.

The results indicate that the RCMD samples consisted of, not only micron-sized dust particles, but also nano-sized dust particles. The composition of the micro-sized RCMD particles was closely correlated with the mining activity. The nano-sized dust particles had a mode particle size of 300–400 nm. The presence of the nano-sized dust particles was confirmed using particle size analyses, including dynamic light scattering (DLS) and
asymmetrical flow field-flow fractionation (AsFIFFF). A small number of nano-sized RCMD samples were analyzed for both size and mineral composition using scanning transmission electron microscopy (S-TEM). The results show that not only diesel particles, but also coal and rock dust particles, were found in the nano-sized fraction of the RCMD. The percentage of the nano-sized fraction of RCMD particles may be site and location dependent, and a survey and detailed analyses should be conducted in the future.

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