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Effect of Size Reduction Processes on the Apparent Fiber Content of Rock Samples

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KEYWORDS

Actinolite, asbestos, byssolite, chrysotile, grinding, CARB 435, concentration, fibers, polarized light microscopy (PLM), serpentine aggregate, tremolite

ABSTRACT

Natural occurrences of asbestos and potential exposure to such materials have drawn significant public and regulatory interest in recent years. Revisions to a published analytical method, CARB 435, that incorporate inappropriate sample comminution strategies have been examined. Tests demonstrating the effects of grinding and preparation of asbestos minerals have been conducted. Sample preparation procedures used by some laboratories in apparent compliance with CARB 435 destroy the characteristics of asbestos, thereby potentially biasing the results. The authors are proposing that the grinding be done in stages, removing the fines at each stage, in order to minimize the effects of grinding. These tests also show that when non-asbestos amphibole particles are crushed, elongated particles are created that could be confused with asbestos fibers (false positives in various analyses). The effects of sample comminution must be considered when creating or revising asbestos analytical procedures.

INTRODUCTION

When rocks that contain veins or pockets of natural occurrences of asbestos are crushed, there is a possibility that asbestos fibers will be liberated and become airborne. If the airborne asbestos is of respirable size, these fibers pose a potential hazard to workers and bystanders who may breathe the fibers if the exposure is high enough or occurs frequently.

Fortunately, minerals rarely grow as asbestos fibers, but more generally grow as (comparatively) larger crystals that do not possess the characteristics of asbestos fibers. Unfortunately, when non-asbestos amphibole minerals are fractured, many will fracture as elongated particles that have dimensions that are encompassed by various regulatory analytical procedures. For example, OSHA (29 CFR §1910.1001) and MSHA (30 CFR §56.5001) regulate airborne asbestos fibers if they are at least 5 μm long, have a minimum aspect ratio (length:width) of 3:1 and are visible in an optical microscope. Fractured non-asbestos minerals that frequently occur with asbestos are routinely observed with these dimensions even though they are not asbestos.

Therefore, it is necessary to be able to analyze samples (both bulk and airborne samples) to determine the amount of asbestos (independent of any non-asbestos mineral) in the samples. The California Air

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Resources Board (CARB) 435 method (1), specifically written for mines and quarries where natural occurrences of asbestos may be disturbed, is a widely used method for analyzing bulk samples of rock. Like any analytical method, it could and should be updated as knowledge of its implementation is gained.

CARB 435

CARB 435 has been written for the analysis of rock samples and provides guidance on size reduction (comminution) of the sample (1). The CARB method, written specifically for the analysis of serpentine aggregate, has been used extensively for the analysis of quarry samples and has been extended by commercial laboratories to the analysis of soils. The method does not adequately address issues related to bulk sample collection, especially statistically representative samples, a subject beyond the scope of this paper.

CARB 435 requires the collected sample to pass through a two-stage crushing/grinding procedure such that "the majority shall be less than 200 Tyler mesh" (75 μm). The first stage, if needed, reduces the collected sample to a "nominal size of less than three-eighths inch" (9.5 mm) through the use of a small jaw crusher. If the collected material is smaller than 9.5 mm, this step is not necessary. The method provides no other description of a "majority" finer than 75 μm, nor does it describe how this is to actually be accomplished. It does recommend the use of a Braun mill, a type of attrition mill, for the grinding to 75 μm.

The intent of grinding the sample is to liberate all possible asbestos fibers as well as homogenize the sample so that a representative aliquot can be removed for analyses using polarized light microscopy (PLM) to determine if the asbestos content of the sample (if any) equals or exceeds 0.25%, which is the regulatory control limit in California. Homogenization is achieved by increasing the number of particles and thoroughly mixing them. A rock that is a 9.5 mm cube contains the equivalent of approximately two million 75 μm cubes. Assuming the components of the rock all pulverize in a similar manner, the proportion of asbestos in the rock is equivalent to the proportion or number of asbestos fibers in the ground sample.

In order to be able to view the particles in the PLM, all particles need to be of uniform size and small enough for light to pass through the particles. Uniform size suggests the particles have a relatively limited range of dimensions, primarily centered (for CARB 435) around 75 μm. A moderately uniform particle size is important to minimize bias in the statistical evaluation of the sample's asbestos concentration. To be able to view the particles in the PLM, the particles should be no thicker than 30 μm (the thickness of a thin section of rock), which is necessary to make the mineral transparent (2). These concepts — uniformity and transparency — tend to be mutually exclusive in the CARB 435 procedure as most particles (by mass) in the target size range will be too thick for light to pass through.

Therefore, the degree to which the sample is ground is critical for an accurate analysis of the sample. Grinding the sample too fine will make the particles smaller than the resolution of the microscope and destroy the fibers that are to be quantified, leading to an underestimation of the fiber concentration. Conversely, if the rock is not ground fine enough, the coarse particles will be too thick to observe their optical properties, and fibers that should be counted will be bound together in the sample leading to an underestimation of the content. The goal of sample preparation is to maximize the visibility of the asbestos while minimizing interferences. Determining whether the concentration equals or exceeds 0.25% may not be achievable due to two competing processes: 1) grinding may not liberate all of the asbestos fibers (however, asbestos is typically present in veins within the host rock and the vein is typically where the rock would break), and 2) grinding may either destroy asbestos characteristics or make the fibers too small to see in the microscope.

CARB has indirectly addressed some of these issues by suggesting that the degree to which a rock is pulverized (or ground) may affect the ability of the laboratory to detect any possible asbestos component of the rock (3). Based on the results of a round-robin evaluation of CARB 435, CARB reported that the finer a rock is ground, the less asbestos that is reported by a laboratory (either because the asbestos is too small to be observed or because the grinding destroys the asbestos characteristics). Unfortunately, CARB's solution to the grinding issue ignores some basic principles of comminution.

Based on their round-robin testing, CARB has recently proposed (4) modifying CARB 435 by requiring all grinding to be conducted in a disc grinder such that the product has a top size of about 250 μm with 40%–60% passing 75 μm. This grinding is accomplished by first calibrating the grinding ability of the machine by using sand (presumably quartz sand), grinding the sand and testing the fineness of the ground material. If not fine enough, the gap between the grinding discs is narrowed and another aliquot of sand is tested. Once it has been demonstrated that the sand can be ground to the desired specification, it is assumed that the test sample can also be ground to the same desired fineness. This modification fails to recognize that different rocks will fracture at different rates and to different degrees of fineness (5).

STAGE GRINDING

One solution to this preparation problem is to grind the sample in stages, with targeted size reduction ratios (feed:product) of no more than 2:1. At the end of each stage, the fines are removed and the oversize re-ground. In this procedure, the possible asbestos fibers in the sample would not be ground to such a degree as to minimize their visibility in the PLM. High particle size reduction ratios (feed size:product size) are known to increase the generation of extremely fine powders (5, 6), a well known concept in the mineral processing field.

A typical stage grinding procedure would start with the rock sample after crushing it in the laboratory jaw crusher. The gap in the disc grinder **(Figure 1)** would be opened to about 5 mm (the distance between the rotating disc and the stationary disc) and the sample ground. After grinding, the sample is then hand sieved using a 150 μm (100 mesh) sieve to remove the fines. The fines are reserved and the oversize reground after closing the gap in the grinder to 2 mm. The ground product is then resieved with the oversize ground again after closing the gap to 1 mm. The process is repeated one more time with a gap between the grinding discs of 0.5 mm (any closer and there is a risk that the discs will jam, stopping all grinding).

The choice of 150 μm (100 mesh) sieve is based on the fracture mechanics. Most ores (on a mass basis) will grind such that the distribution of particle size will follow a logarithmic distribution (a graph of the cumulative percent less than size versus size will plot with a slope of about one on a log-log graph) (5). Using a top size of 150 μm will result in a distribution with 50% finer than 75 μm (200 mesh), the target size of CARB 435. The larger sieve openings (100 mesh) will permit easier size separations than if a 200 mesh sieve is used.

EXPERIMENTAL

A project was designed to demonstrate the effect of comminution on rocks potentially containing asbestos minerals. Four samples, samples shown in Figure 2A–D (page 6), were obtained and used in the testing. Each sample was 50–75 mm, which is a typical size of railroad ballast or rock that would be used as base material for road building. Each sample contained a vein or veins of visibly fibrous minerals that were identified as the amphibole minerals tremolite asbestos, actinolite (byssolite[1]) or the serpentine mineral chrysotile.

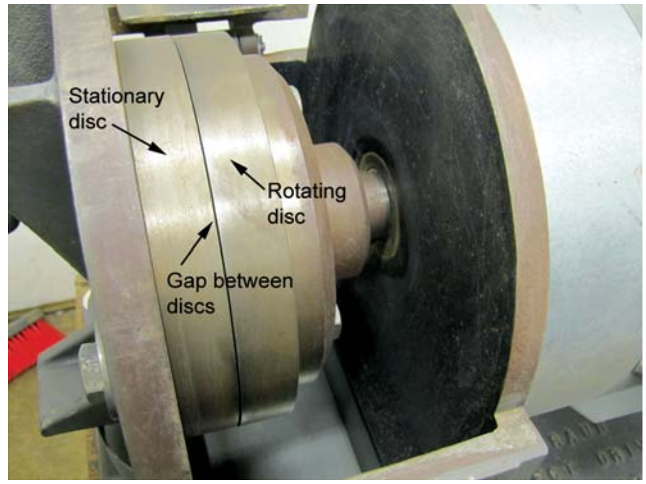

Figure 1. The grinding machine has stationary and rotating discs with a gap in between that can be decreased to grind the specimen more finely.

In the first test, serpentine rock was initially crushed through the laboratory jaw crusher, blended and split into four aliquots. Two aliquots were ground using a single pass through the disc grinder as recommended by CARB 435; two aliquots were stage ground to 150 μm as described above. The ground samples were analyzed using PLM in accordance with CARB 435.

In three other samples (tremolite, actinolite and chrysotile), each sample was stage ground as described above, then split into equal parts. The second part of each sample was further ground to 75 μm (200 mesh) to demonstrate the effect of degree of grinding. All ground samples were analyzed in accordance with CARB 435 using PLM.

Particles of tremolite, actinolite and chrysotile 5 μm and larger were determined by scanning electron microscopy.

ANALYTICAL RESULTS

The asbestos content of the serpentine sample that was ground using either a single-pass or a stage-grinding procedure are compared in Table 1 (page 7). As

[1]Byssolite is a fibrous form of amphibole minerals that has low tensile strength and moderate fiber widths. A portion of the fibers will show crystal faces. Byssolite fibers are not asbestiform fibers and they are not asbestos.

2A

2B

2C

2D

Figure 2. Rock samples used in the test program to demonstrate the effect of comminution on rocks potentially containing asbestos minerals: 2A, serpentine rock collected at the mine in Lowell, Vermont; 2B, pieces of rock containing tremolite asbestos that was obtained from a drill core; 2C, a sample that is also from a drill core but contains actinolite (byssolite); and 2D, two pieces of chrysotile ore from the Lowell mine.

shown, the single-pass samples average 2.5% while the stage-ground samples average 4.25%. These samples contain the same amount of asbestos fiber, but because of the grinding procedure, the stage-ground samples appear to contain 70% more fiber than the single-pass samples.

When the samples are stage ground, but to differing degrees of fineness, the reported fiber contents in Table 2 (page 7) reflect the results of the degree of grinding. Each sample, when ground to 75 μm had lower reported fiber contents than with the coarser grind. Care was taken when splitting the samples so they would be well mixed (homogenized), which ensured that the divided samples did not introduce bias into the analyses. The samples were split using a riffle splitter, which divides the sample into two equal portions. It is assumed for these analyses that there was no preferential grinding; the fiber content of each size fraction is the same as any other size fraction. The observable fiber concentration in the actinolite (byssolite) sample was reduced from 17.25% to 0.75%, a 96% reduction in fiber content. The tremolite asbestos sample was reduced from 18.5% to 1.25%, a reduction of 93%.

The reduction in fiber or asbestos content due to the degree of pulverization indicates CARB's requirement to grind to a nominal 75 μm, which may cause a sample to be considered as not containing asbestos. As a hypothetical example, a rock sample containing 4% asbestos is ground to a nominal 75 μm size, with a concurrent reduction of reportable asbestos to 0.2%. This level is below the regulatory control limit of 0.25% (7).

The size distribution of the samples for particles 5 μm and longer are shown in Tables 3–5 (page 8). As

Table 1. Observed Fiber Content of Samples Processed with Single-Pass or Staged Grinding

Sample	Process	Fiber Content %*
1	Single Pass	2.25
2	Single Pass	2.75
3	Stage Grinding	3.5
4	Stage Grinding	5

*The fiber content was determined by polarized light microscopy.
Point counting with a cross hair ocular was used for quantification.

Table 2. Observed Fiber Content of Samples as Determined Using Polarized Light Microscopy

Sample	Grind	Fiber Content %*
Actinolite	< 150 μm	17.25
	< 75 μm	7.5
Tremolite	< 150 μm	18.5
	< 75 μm	9.0
Chrysotile	< 150 μm	43.25
	< 75 μm	6.5

*The fiber content was determined by polarized light microscopy.
Point counting with a cross hair ocular was used for quantification.

expected, the length of the fibers are shorter in the samples that were more finely ground. However, there was no significant difference in the apparent width of the fibers when comparing the two degrees of grinding.

DISCUSSION

The data from these experiments demonstrate that the issues related to the pulverization of asbestos minerals are complex. Liberation of particles is ongoing throughout all comminution processes. The energy input into a system affects each particle differently, i.e. fibers and/or bundles can be liberated from complex (locked) particles; bundles can be split partially or completely into the individual fiber components; and fibers can be fractured into shorter, thinner, more numerous particles.

Incorrect sample preparation will result in biased analytical results. These studies demonstrate:

Stage grinding results in larger reported asbestos concentrations than does single-pass grinding (Table 1). The reduction in top size should be performed in a manner to minimize the production of fines, with the reduction ratio not exceeding 3:1 or 4:1 (feed:product). For example, a one-inch rock should be crushed no finer than 6.4 mm prior to sieving (possibly at 150 μm), followed by grinding the coarse sieve fraction. The grinding could be done in stages, to a top size of about 12 mesh (1.68 mm), followed by another reduction to 40–60 mesh (420–250 μm), and then a final reduction to 100 mesh (150 μm). At every step, the fines (< 150 μm) are removed by sieving. In such a procedure, possible asbestos fibers would be liberated while minimizing size reduction that would prevent observing them in a light microscope.

Over grinding reduces the reported asbestos concentration of samples. As is shown by this study, over-pulverization of samples may result in reduced reported asbestos concentrations by up to 85% (Table 2). This finding is consistent with that reported by CARB in their round-robin evaluation of the CARB 435 method. CARB's study appears to show a relationship between the fineness of a sample after grinding and the amount of reported asbestos (the finer a sample was ground, the less asbestos reported). Therefore, CARB also showed that the properties of asbestos can be destroyed by over grinding.

Table 3. Summary Statistics for the Length (μm) of Fibers in the Pulverized Samples

Sample	Grind	Distribution, Percentile					Mean	Standard Deviation	Geometric Mean
		10	25	50	75	90			
Actinolite	< 150 μm	8.6	14.3	25.6	44.8	70.8	35.8	34.5	25.2
	< 75 μm	5.7	6.4	8.4	10.4	14.4	9.2	3.9	8.6
Tremolite	< 150 μm	6.2	7.5	11.4	18.7	39.8	20.0	38.9	13.3
	< 75 μm	5.6	6.3	8.3	13.5	19.8	11.6	9.3	9.6
Chrysotile	< 150 μm	8.8	13.6	23.2	39.0	70.8	34.3	36.7	24.2
	< 75 μm	5.3	6.4	7.7	11.5	17.7	10.5	9.4	8.9

Table 4. Summary Statistics for the Width (μm) of Fibers in the Pulverized Samples

Sample	Grind	Distribution, Percentile					Mean	Standard Deviation	Geometric Mean
		10	25	50	75	90			
Actinolite	< 150 μm	0.6	1.0	1.8	3.2	5.6	2.9	3.7	1.8
	< 75 μm	0.5	0.8	1.2	1.5	2.3	1.3	0.8	1.1
Tremolite	< 150 μm	0.2	0.3	0.5	1.1	2.2	1.0	1.4	0.6
	< 75 μm	0.2	0.2	0.3	0.5	1.1	0.8	2.3	0.4
Chrysotile	< 150 μm	0.2	0.3	0.5	1.1	1.8	0.8	0.9	0.6
	< 75 μm	0.3	0.4	0.6	1.6	2.5	1.0	0.9	0.7

Table 5. Summary Statistics for the Aspect Ratio of Fibers in the Pulverized Samples

Sample	Grind	Distribution, Percentile					Mean	Standard Deviation	Geometric Mean
		10	25	50	75	90			
Actinolite	< 150 μm	5.7	7.4	13.3	23.8	37.0	19.1	19.0	13.9
	< 75 μm	4.2	5.5	7.4	11.2	16.0	9.4	7.7	7.9
Tremolite	< 150 μm	8.4	13.5	21.6	33.7	55.8	29.6	28.4	22.2
	< 75 μm	7.7	14.5	25.8	39.0	66.0	32.3	28.5	23.3
Chrysotile	< 150 μm	14.5	25.2	44.2	65.7	116.1	57.5	60.7	40.8
	< 75 μm	3.5	5.4	14.4	23.2	39.6	17.6	14.6	12.4

The CARB 435 grinding specification may lead to false negative results. For samples containing low levels of asbestos, this grinding requirement could reduce the observable asbestos content to below the action level of 0.25%, thus creating a false negative finding. Though CARB has proposed redefining “majority” to 40–60%, the one-step grinding procedure will result in an excess generation of fines. As shown by this study, CARB should modify the method to require grinding to a nominal 80% passing 150 μm. CARB should also require staged grinding with sieving at each stage to remove material finer than 150 μm.

This study demonstrates that sample preparation procedures do affect the asbestos fibers. It also suggests that these procedures can be used inadvertently to skew results, reinforcing the concept that standard, but appropriate methods be used for asbestos analyses. These data show that the choice of material to test for evaluation of analytical methods will affect the usefulness of the method evaluation. Any material chosen for method evaluation must be fully and carefully characterized prior to use.

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