Morphological characteristics of asbestos in ground bulk mineral powders

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Introduction: Differences exist in the morphological features of asbestos and the abilities of measuring tools over the spectrum of the particle size distribution in ground bulk mineral powders. Amphiboles and serpentine most often occur naturally without an asbestos component, although amphiboles typically produce elongate mineral particles when ground. For reasons of defining health risks, attempts to conclusively distinguish between asbestos and non-asbestos occurrences are warranted. Asbestiform morphological characteristics are more readily visible in the coarser size fraction of a ground mineral powder. Therefore, a procedure was developed to isolate and analyze the coarse size fraction of mineral mixtures for asbestos.

Methods: The morphological characteristics of asbestos spiked into ground mineral powders at 100 ppm and 500 ppm concentrations were evaluated. Three types of asbestos, Lone Pine tremolite, Health and Safety Laboratory (HSL) chrysotile, and short-fiber Calidria chrysotile, were spiked into a coarsely crushed talc matrix. Spiked samples were aggressively co-ground to simulate a milled mineral powder final product. The coarsest particles in the mixture were isolated using a wet-sieving technique and evaluated by scanning electron microscopy (SEM) and polarizing light microscopy (PLM). A production-scale ultrafine milled talc sample containing trace amounts of amphibole was also analyzed using the wet-sieve approach.

Results: Asbestos was readily detected by both SEM and PLM in the coarsest size fraction at these low concentrations, despite aggressive grinding. Classic morphological features of asbestos, such as occurrence in bundles and presence of long, thin fibers showing curvature, were observed for all types of asbestos by both SEM and PLM at both concentrations. There were no morphology differences between the 500 ppm and 100 ppm concentrations, although fewer particles were observed in the latter, as expected. Trace amounts of amphibole asbestos were detected in the production-scale ultrafine milled talc sample which showed classic asbestiform morphology even though the sample had been ground to a 1.5 µm median particle size.

Discussion: Results show that the presence of asbestos can be confirmed in ground bulk mineral powders using this analysis approach and detection by SEM and/or PLM is limited only by the amount of material analyzed. This approach is opposite to types of asbestos analyses that concentrate on the finest particles (such as those performed by transmission electron microscopy (TEM)), which can sometimes be inconclusive. The production-scale ultrafine milled talc sample

provided proof of concept for this approach. The techniques described can be used to accurately determine if a mineral product contains asbestos or an asbestos component.

1 Introduction

Characteristics of asbestos, if present in bulk mineral powders, show differences in morphological features over the spectrum of the particle size distribution (PSD). Differences also exist in the abilities of measuring tools over the full range of the PSD, and if asbestos is present in trace amounts, the overall morphology may be difficult to assess for the few particles present unless concentration methods can be employed. For example, the finest particles of asbestos in a ground mineral powder, as measured by transmission electron microscopy (TEM), may not display typical asbestiform characteristics (such as occurrence in bundles and presence of curvature). Furthermore, because all amphiboles produce elongate particles when ground (Wylie, 1990), amphibole asbestos particles in the finer size range may look similar to non-asbestos particles, as referenced in the Scope section of ISO TEM method 10312, which states "The method cannot discriminate between individual fibres of asbestos and elongate fragments (cleavage fragments and acicular particles) from non-asbestos analogues of the same amphibole mineral" (International Organization for Standardization ISO, 2019a).

Although SEM has the best ability to show surface morphological features of particles and can span the entire PSD of a mineral powder (coarse to fine), conventional SEM may have limitations in the ability to conclusively distinguish between minerals with similar elemental compositions, such as low iron anthophyllite vs. talc (International Organization for Standardization ISO, 2019b). In contrast to SEM, mineral identification can be more conclusive by refractive index assessment in PLM or by crystallography assessment in TEM.

Characterization of asbestos in natural bulk mineral samples is further complicated by the fact that natural occurrences of asbestos typically include a component of the non-asbestos variety along with the asbestos component. It is noted that "the asbestiform varieties occur in veins or small veinlets within rock containing or composed of the common (non-asbestiform) variety of the same mineral" (Campbell et al., 1977). In fact, commercially viable asbestos deposits typically contain only 5%–6% asbestos, with a notable exception of the Coalinga chrysotile deposits in California, United States, which are reported to contain 50% or more (International Agency for Research on Cancer IARC, 2012; U.S. Geological Survey USGS, 2001; Virta, 2006). This means that even in asbestos mines with the highest asbestos content, historic mining processes produced products and other powder samples that are likely to contain a component that is not asbestiform even after beneficiation. Therefore, it is difficult to obtain samples of asbestos that do not contain some amount of non-asbestos material.

However, the opposite situation is not necessarily true, and non-asbestos amphibole and serpentine can, and most often do, occur without an asbestos component. Although amphiboles and serpentines are common rock-forming minerals (Deer et al., 2013), asbestos occurrences are relatively rare (Ross et al., 2008). In fact, amphiboles in general are estimated to make up at least 5%–10% of the Earth's crust (Klein et al., 1993; Wylie and Candela, 2015) and are found to be ubiquitous in U. S. soils (U.S. Department of Agriculture USDA, 2010; Thompson et al., 2011). However, "only a very small quantity of the amphibole and serpentine minerals under particular geologic circumstances occur as the asbestiform variety of the mineral" (Campbell et al., 1977). It follows that in the absence of the specific geologic circumstances needed to form asbestos, natural occurrences of amphibole and/or serpentine can be completely devoid of the asbestiform variety of these minerals.

For reasons of defining health risks, attempts to conclusively describe the asbestiform habit are warranted. While the adverse health effects of asbestos are known, non-asbestiform amphibole and serpentine minerals have not been shown to have the same potential for causing asbestos-related disease (Ilgren, 2004; Addison and McConnell, 2008; Mossman, 2008; Guthrie et al., 1993). In fact, the OSHA ruling of 1992 officially recognized that non-asbestiform amphiboles should not be regulated as asbestos (Occupational Safety and Health Administration OSHA, 1992), and methods allow for distinction of morphology between the two (Occupational Safety and Health Administration OSHA, 1995; Occupational Safety and Health Administration OSHA, 1997).

Earlier methods did not make this distinction. Prior to the 1992 OSHA ruling, earlier 1987 "interim" methods were adopted by the EPA in response to the 1986 Asbestos Hazard Emergency Response Act (AHERA). These methods were for the specific application of removal of asbestos-containing materials (ACM) in schools, with ACM defined as containing >1% asbestos (U.S. Environmental Protection Agency USEPA, 1987). Although the original AHERA PLM bulk method documents asbestos particles with a ≥3:1 aspect ratio for quantifying ACM, and the AHERA TEM air method counts airborne asbestos particles with a ≥5:1 aspect

ratio for clearance purposes, neither of these aspect ratios are diagnostic for asbestos, and most certainly include non-asbestiform particles unless used in the intended environment (National Institute for Occupational Safety and Health NIOSH, 2011).

Following the 1992 ruling by the OSHA, the EPA developed an updated method with a full description of the asbestiform habit (U.S. Environmental Protection Agency USEPA, 1993). The 1993 EPA method was proposed to replace the earlier 1987 interim method, as the prior method does not have a descriptive definition of asbestiform and aspect ratios are contradictory, with mentions of 10:1, 5:1, and 3:1 for different counting purposes. This could lead analysts to erroneously conclude that asbestos is present when it is not [see the discussion by Beard (1992); also reproduced in the Supplementary Material)]. Although never officially promulgated with respect to the earlier AHERA rule, the updated 1993 method is widely used by laboratories and is recognized by the National Voluntary Laboratory Accreditation Program (NVLAP).

Recognizing discrepancies in prior terms, the NIOSH in 2011 defined the term "elongate mineral particle" (EMP) along with a roadmap proposing studies to further distinguish terms (National Institute for Occupational Safety and Health NIOSH, 2011). The term EMP includes both asbestos and elongate non-asbestos particles known as "cleavage fragments" in mixed environments, as well as particles counted by earlier methods. The term EMP is not specifically related to disease.

Attempts to correlate descriptions of the asbestiform habit with asbestos-related disease usually involve measurements of individual particle dimensions. It has been recognized that the characteristically small widths of asbestos particles are a key parameter in the mechanism for disease (Wylie et al., 1993; Wylie and Korchevskiy, 2023), and a maximum width of 1 µm was proposed for counting particles in mining environments (Harper et al., 2008). However, asbestos fiber widths related to translation of fibers from lungs to pleura may be much smaller, as summarized by Wylie et al. (2020). In this article, widths of <0.4 µm (Lentz et al., 2003), <0.3 µm (Carbone et al., 2012), <0.2 µm (Pooley, 2018), and <0.15 µm (Lippmann, 2014) are referenced.

A notable study by Chatfield directly relates animal dosing studies and mesothelioma occurrence (Chatfield, 2018). In this study, a positive correlation was found between the number of high-aspect-ratio particles in amphiboles with mixed morphologies and mesothelioma occurrence. Chatfield noted that particles associated with mesothelioma, and therefore consistent with asbestiform classification, can be categorized into three size classes based on aspect ratio (AR), length, and <1.5 µm width, as measured by TEM: 1) ≥35:1 AR for lengths between 5 µm and 10 µm, 2) ≥30:1 AR for length between 10 µm and 20 µm, and 3) ≥20:1 AR for lengths ≥20 µm. In comparison to these particle size classes, called "extra-criteria EMPs," "sub-criteria EMPs" do not correlate with disease.

Further attempts to develop discriminant mathematical functions have been successful in differentiating between asbestos and non-asbestos amphiboles, also based on measurements of individual particles (Wylie et al., 2022; Wylie and Korchevskiy, 2023; Van Orden, 2023; Korchevskiy and Wylie, 2025), with the latest 2025 proposed model effective with a high degree of accuracy (R^2 = 0.96) (Korchevskiy and Wylie, 2025). While the dimensions of individual particles or fibrils of asbestos appear to be predictors of disease, confirmation of the asbestiform habit is often done based on

macro features displayed in samples before they are ground. However, features specific to the asbestiform habit, such as occurrence in bundles and presence of long, thin fibers showing curvature, are definitive for the presence of asbestos, even in ground mineral powders. Larger composite particles (i.e., bundles) are often excluded from particle counts based on dimensions alone, or if particle counts are done by TEM, larger composite particles and extremely long, thin particles may not be included in preparations.

While relatively pure asbestos standard materials have been characterized (i.e., Lone Pine tremolite asbestos research material, National Institute of Standards and Technology (NIST) and Health and Safety Laboratory (HSL) standard reference materials, etc.), natural bulk mineral powders that contain adequately characterized trace amounts of asbestos (without mixed or non-asbestos morphology) are difficult to obtain, and the morphology of asbestos particles in ground mixtures has not been fully assessed. Mischaracterization of mineral powders containing amphibole or serpentine with non-asbestos morphology may result in misleading conclusions about the presence of asbestos in ground mineral powders and with respect to hazard identification for use in studying adverse health effects.

It is proposed that the key to determining if a ground mineral powder is asbestiform, or contains an asbestiform component in mixed samples, is the observance of the morphology of the largest particles in the ground mixture. If asbestos is present in natural ground mineral powders, composite particles that occur in bundles and/or particles displaying curvature should be present. These features are more likely to be associated with the largest particles in the ground mixture than the smallest ones.

For milled bulk mineral powders, it is expected that the bulk of asbestos will preferentially occur in the upper portion of the particle size distribution due to its resistance to grinding. It is a well-known principal of comminution (reduction of solid material, or ore, from one average particle size to a smaller average particle size) that in a mixture of minerals with different grinding profiles, "the relatively tough mineral particles remain coarse while the more friable ones are finely ground" (Pryer, 1958). Therefore, harder minerals and/or minerals that have properties that make them more resistant to grinding, such as increased tensile strength, become enhanced in the coarser size fraction of the mixture. The enhanced tensile strength of asbestos is a well-known property that is included in most definitions of asbestos (Lowers and Meeker, 2002).

The particle size of the largest particles in a milled mixture, or "top-size" if the ground mixture is a mineral product, is a variable parameter that depends on the degree of milling. In this study, a procedure was developed to isolate and analyze the coarser particles in milled mineral powders for the purpose of asbestos analysis, and this was shown to be effective, even for ultra-finely milled talc products.

2 Materials and methods

2.1 Overview

In this study, a spiking procedure was developed that simulates the milled production of a mineral powder product. This procedure, presented at the 2014 and 2017 ASTM Johnson Conferences on Asbestos (Pier, 2014; Pier, 2017a; Pier, 2017b), involves spiking a coarsely crushed talc with known trace concentrations of asbestos

FIGURE 1
Starting materials of ~0.01% (100 ppm) asbestos (right) in coarsely ground talc chips (left). Further co-milling these materials together provides a thoroughly dispersed mixture with maximum homogeneity, simulating a production-scale ground mineral powder.

and subjecting the mixture to aggressive grinding to simulate a typical production-scale ground mineral product. In this study, 0.05% (500 ppm) and 0.01% (100 ppm) levels were prepared.

A simple wet-sieving technique, first presented at the 2011 ASTM Johnson Conference on Asbestos (Pier and McCarthy, 2011) and at subsequent ASTM Johnson Conferences (Pier, 2014; Pier, 2017a; Pier, 2017b), was used to concentrate coarser particles for SEM and PLM analysis. The morphological characteristics of asbestos particles in the mixtures were evaluated by both techniques. Spiked samples are compared to an actual production-scale ultrafine milled talc sample containing both asbestos and non-asbestos amphibole trace components.

Spiking materials used in this study were a Lone Pine tremolite asbestos research material obtained from the Research Triangle Institute (Harper et al., 2015), a chrysotile standard reference material obtained from the United Kingdom. Health and Safety Laboratory (HSL), and a Calidria (short-fiber) chrysotile sample, also obtained from the Research Triangle Institute. The base material was a hydrothermal talc from the Guangxi region of China and was confirmed not to contain asbestos using TEM by the Research Triangle Institute (verbal communication).

Interference from non-asbestiform components is minimized with hydrothermal talc deposits as, the geologic process of formation is known not to be associated with serpentine, and hydrothermal talc deposits are typically formed at lower temperatures than those that produce amphibole (Van Gosen et al., 2004). Hydrothermal talc deposits are formed from carbonate-based host rocks and represent more than 70% of the world production of talc (McCarthy et al., 2009). For comparison to spiked materials with known concentrations, a production-scale ultrafine milled talc sample, containing trace amounts of tremolite, was obtained for evaluation from an uncertain external (non-USA) mining source.

2.2 Spiking procedure with grinding to produce standards with known concentrations

Due to the discrete nature of asbestos occurrence and its resistance to grinding, asbestos is difficult to disperse into a matrix material in a way that is both homogenized and accurately represents a natural occurrence. Because of its unique properties, asbestos cannot simply be added to matrix materials without some level of pre-grinding. However, the best simulation of an occurrence of asbestos in a ground mineral product is to mix the appropriate weights of coarse components in the desired concentration and aggressively co-grind the two materials together to produce the median particle size (MPS) of a typical ground mineral product. This not only ensures that the resulting mixture has the correct concentration, but also that the grinding action serves to thoroughly mix and disperse the components.

In this study, samples of asbestos were tweezed from reference asbestos materials that had been pre-ground using a mortar and pestle and weighed along with coarse talc chips to produce approximate starting target concentrations between 0.01% and 0.1% asbestos in talc (Figure 1). The starting amount of the talc base material was 2–3 g. The entire quantities of the weighed materials were washed into a benchtop Retsch® micronizing mill, marketed by the McCrone Group, using isopropyl alcohol (IPA). This mill uses an aggressive vibratory wet milling action with 48 cylindrically shaped agate milling media. Technical notes for this mill indicate that an optical glass silica sand standard was reduced in size from 285 μm to 5 μm median diameter after only 10 min of grinding. In a study involving different grinding times of talc, it was determined that the typical median particle size of a cosmetic grade talc product (~12 μm) was achieved in approximately 5 min as determined using a Malvern® laser particle size analyzer. In the present study, the milling time used was 30 min, which is more than sufficient to produce a resulting sample in the appropriate size range of a thoroughly ground mineral product.

After milling, the ground asbestos-in-talc mixtures were washed from the McCrone grinding canister, using a specialized cap with two diametrically opposed holes for rinsing, directly into a disposable mixing canister from a FlackTek Speedmixer®. The rinsing procedure from the McCrone mill allows for virtually 100% sample recovery. If needed, adjustments to the final target concentrations of 0.05% (500 ppm) and 0.01% (100 ppm) were achieved by the addition of appropriate measured weights of talc, also ground for 30 min, directly into the disposable canister for mixing.

The final concentrations were thoroughly mixed in the FlackTek apparatus, which uses a bladeless, planetary dual asymmetric centrifugal action for mixing. Potential sample loss is minimized because there are no internal mixing blades in the apparatus. Potential sample loss from transfers is further minimized as the mixed slurry is washed directly onto a sieve used for the following wet sieve procedure. Note that the starting mixtures and any additions to achieve 0.05% and 0.01% final concentrations were added together in their entirety before mixing. Therefore, no subsampling was necessary, and there is high confidence that the finished amount prepared represents actual known weight concentrations.

The above-described procedure was used for all mixtures using Lone Pine amphibole asbestos as well as the HSL and Calidria chrysotile mixtures. For the Lone Pine amphibole spikes, additional preparations were made from a bulk 0.1% starting mixture of pre-ground Lone Pine asbestos spiked into ground talc. For these preparations, the material was subsampled and ground for 30 min in the McCrone micronizing mill, as above. The milled material was diluted to concentrations of 0.05% (500 ppm) and 0.01% (100 ppm) with McCrone-milled talc chips, as described above, and mixed using the FlackTek Speedmixer™. Both Lone Pine preparations produced similar results.

2.3 Wet sieve procedure to isolate larger particles in ground mineral powders

The intent of the wet sieve procedure is to concentrate and isolate larger particles in the ground mineral mixture for analysis by PLM and SEM, as these are the ones that have a better chance of displaying macro-scale morphological features. In comparison to dry sieving, which produces particles that are covered with finer material, wet sieving effectively washes the finer material from the surface of the coarse particles through the sieve using IPA so that finer particles do not interfere with refractive index determination by PLM or energy-dispersive X-ray spectroscopy (EDS) elemental analysis and visualization of the morphology of the larger particles by SEM. Note that this is the opposite approach to that which is normally done for asbestos analysis, as it has been thought that methods that analyze only the smallest particles are appropriate due to the small size of individual fibrils. In fact, this has been a long-standing criticism of optical microscopy methods, which do not have sufficient resolution to observe individual fibrils of chrysotile and may be at the limit of resolution for individual fibrils of amphibole asbestos.

However, it had been shown that even for the most finely ground talc products, some portion of particles larger than a 400-mesh sieve are present. This was confirmed with the production-scale ultrafine milled talc sample, which had a measurable retention when passed through a 400-mesh sieve, even though its median particle size (1.5 μm) was much finer than the 37 μm size of the mesh opening (Pier and McCarthy, 2011). In this case, 100 g of the starting material was necessary to obtain a measurable retention, which is more than that needed for other, less finely ground samples. For coarser ground materials, a starting weight of 2 g is typically sufficient. Note that for preparations of the production-scale milled sample, the grinding portion of the preparation procedure is not necessary, as samples had already been subjected to aggressive grinding during production-scale milling operations.

For preparations of standards with known concentrations, material that was prepared using the spiking and grinding procedure described above was transferred directly to a 400-mesh sieve and washed through the sieve using mixtures of IPA and deionized water to isolate the larger particles. For all talc powders, IPA is necessary due to the hydrophobic nature of talc and for the reduction of foaming due to air entrainment of the fine particle slurry. Retained residues were washed from the screen and dried before preparation for PLM and SEM.

2.4 Preparation and investigation of wet-sieved samples by SEM

For SEM analysis, the residue retained on the 400-mesh sieve was filtered through a 5-μm pore size polycarbonate membrane filter in its entirety. This pore size is significantly larger than the 0.2-μm pore size filters typically used for TEM analysis to facilitate the filtration process and because particles of interest for this analysis have already been pre-screened using the 400-mesh sieve which is coarser than the 5 μm pore size of the filter. Sections of the dried filters were excised and placed onto an adhesive tab on an SEM sample stub and coated with a conductive coating (carbon or gold/palladium) prior to analysis. SEM analyses were completed on a Hitachi 4300 SE-N thermal field emission SEM equipped with an EDAX Genesis XM4 SDD EDS detector.

It is a common SEM practice to screen particles that have aspect ratios of 3:1 or greater and greater than 5 μm in length, often with parallel sides as a further screening parameter. However, further characterization of these coarser particles is required to evaluate whether asbestiform morphology is present, as dimensions with a 3:1 aspect ratio are not specific for asbestos or asbestiform particles and may not be diagnostic, as described in the NIOSH Roadmap (National Institute for Occupational Safety and Health NIOSH, 2011). Energy-dispersive X-ray spectroscopy (EDS) is used with SEM to confirm that particles have chemistry consistent with the phase of interest. Although the resolution of SEM can span the entire range of particle sizes, for practicality with respect to analysis time, a decision must be made for the magnifications chosen to document particles either in the finer range (as in TEM) or in the coarser range (as in PLM). In this analysis, it is the coarser particles that are of interest; therefore, magnifications lower than those used for TEM were used for SEM.

2.5 Preparation and investigation of wet-sieved samples by PLM

For PLM analysis, wet-sieved samples were prepared on at least 3 separate microscope slides using a total of approximately 5 mg of the powder retained on the 400-mesh sieve into a few drops of
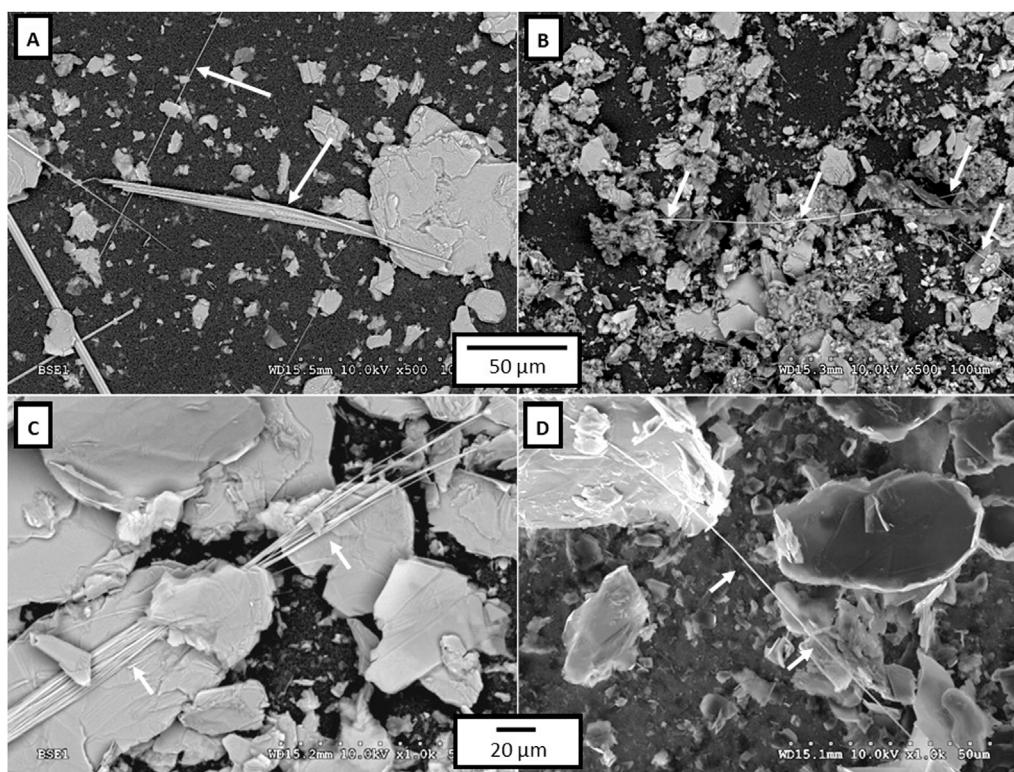
FIGURE 2
SEM images of a 0.05% (500 ppm) concentration spike **(A,B)** and a 0.01% (100 ppm) concentration spike **(C,D)** of Lone Pine tremolite asbestos standard research material in talc. The mixture was co-milled 30 min prior to analysis, and coarser particles were concentrated using a 400-mesh sieve on the milled material. Original magnifications were 500× and 1,000×, respectively. Classic asbestiform features, such as bundles and long, thin fibers showing curvature, are clearly visible despite aggressive grinding.

refractive index liquid after rinsing the residue from the sieve and drying off the liquid. A coverslip is applied, and the material is scanned in its entirety instead of traditional methods of point counting. This increases the total amount of the material analyzed and improves the resulting detectability. Multiple slide preparations in multiple refractive index liquids can be prepared in this manner. PLM analyses were performed on a Nikon Optiphot-POL polarizing light microscope.

Particles are screened for further characterization using the PLM dispersion staining technique at 100×, achieved using a standard 10× dispersion staining objective, further magnified by 10× oculars. The effectiveness of this protocol was unexpected, especially for chrysotile. The full suite of optical properties is confirmed at higher magnification, typically 400×, for particles screened by the dispersion staining method. This higher magnification is achieved using a standard 40× objective, further magnified by 10× oculars.

Refractive index liquids for screening by the dispersion staining method are the same as those typically used in bulk building materials, 1.605 for non-commercial amphibole asbestos and 1.55 for chrysotile. In 1.605 refractive index liquid, a characteristic yellow dispersion staining color is produced for amphibole particles with elongation oriented parallel to the lower E-W polarizer. In this orientation, the γ index of refraction is measured, and the yellow dispersion staining color indicates γ is higher than 1.605, confirmed with Becke line analysis. The more variable α' index of refraction for amphiboles is measured when particle elongation is oriented perpendicular to the

lower E-W polarizer. The α' index of refraction is more variable depending on the crystallographic orientation of the particle. In the perpendicular orientation, the α' refractive index is closer to that of the 1.605 liquid as determined by a closer-to-matching dispersion staining color and lower relief. In contrast, talc particles have refractive indices that are lower in all orientations than the 1.605 refractive index liquid and produce a pale blue dispersion staining color.

Chrysotile particles are a near match in the 1.55 refractive index liquid, displaying colors that are typically magenta when particle elongation (and γ) is oriented parallel to the lower E-W polarizer but could range from magenta/orange to blue. The dispersion staining colors of chrysotile are typically blue or pale blue when elongation is oriented perpendicular to the lower E-W polarizer and the α index of refraction is measured. Alternatively, talc platelets have refractive indices (β and γ) that are very near to each other and both higher than 1.55, producing a characteristic yellow dispersion staining color for the full rotation of the stage in this liquid. In this orientation, talc platelets have low apparent birefringence.

However, in orientations where talc is on edge, a possibility for elongate talc particles, talc shows the characteristic yellow dispersion staining color when particle elongation is oriented parallel to the lower E-W polarizer, indicating the measured refractive index (γ) is higher than the liquid, and a pale blue color with particle elongation oriented perpendicular to the lower E-W polarizer, indicating the measured refractive index (α) is lower than the liquid. In this orientation, apparent birefringence is much higher. Note that for
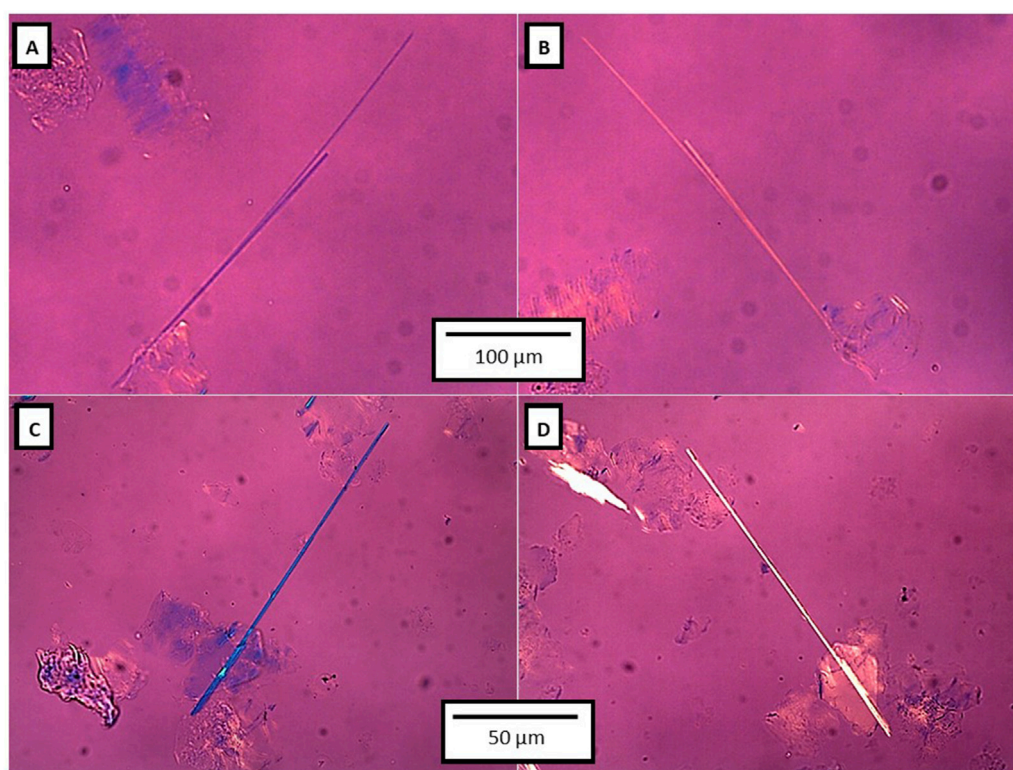
FIGURE 3
PLM images of a 0.05% (500 ppm) concentration spike **(A,B)** and a 0.01% (100 ppm) concentration spike **(C,D)** of Lone Pine tremolite asbestos standard research material in talc, mounted in 1.605 refractive index liquid. The mixture was co-milled 30 min prior to analysis, and coarser particles were isolated using a 400-mesh sieve on the milled material. Images were taken with crossed polars and the first-order red accessory plate inserted (original magnifications were 200× and 400×, respectively). Colors indicate a positive sign of elongation consistent with tremolite. The bundle structure of the tremolite asbestos is apparent, and many fibrils showed curvature. Some "straight" fibers were actually composed of twisted fibrils, visible at higher magnifications.

yellow and pale blue dispersion staining colors, which indicate refractive indices are significantly different than the refractive index liquid, precise quantitative refractive index determination is not possible, and additional liquids would be needed if relative determinations are not sufficient to distinguish particles.

Sign of elongation is determined in crossed polars with the first-order red accessory plate inserted. A blue color with an NE–SW orientation parallel to the "N" refractive index of the accessory plate and a yellow color in the opposite NW–SE orientation indicate a positive sign of elongation. Opposite colors would indicate a negative sign of elongation. The extinction angle is determined with crossed polars but is anomalously near parallel or ambiguous for asbestos, in contrast to non-asbestos particles, which may have a measurable extinction angle (up to 20°). Apparent birefringence is qualitatively noted for the two refractive indices in the plane of the microscope slide. Optical property analysis by PLM is fully described in ISO 22262-1 (International Organization for Standardization ISO, 2012).

2.6 Preparation and investigation of non-sieved samples by TEM

Samples for TEM analysis were not sieved prior to analysis because the standard TEM preparation method naturally selects for finer particles. The procedure involves suspending the particles in particle-free water and withdrawing an aliquot from the liquid with the suspended particles, typically done with one or more dilution steps. Aliquots sampled from the suspension are filtered through a 0.2-pore-size polycarbonate membrane filter and coated with a carbon film. Portions of the filter are placed on a 3-mm-diameter TEM grid, and the filter material is dissolved away using a Jaffe wick procedure, leaving an electron-transparent film encapsulating the particles. This procedure is described in ISO 13794 (International Organization for Standardization ISO, 2019) as well as in other TEM methods. TEM analyses were performed on a JEOL 1200EX transmission electron microscope equipped with an EDAX Genesis XM2 SDD EDS detector.

With the suspended slurry preparation, larger composite particles and extremely long, thin particles may not be included in preparations due to physical constraints of the 3-mm-diameter TEM grids and possible settling of particles during TEM preparation. Particles that settle out of suspension may include larger ground individual particles as well as larger composite particles. In addition, large particles in the suspension may interfere with the integrity of the applied carbon film intended to encapsulate the particles; therefore, large particles may be lost. Even if the larger particles are included in TEM preparations, they may be missed due to the time spent counting the more prevalent particles
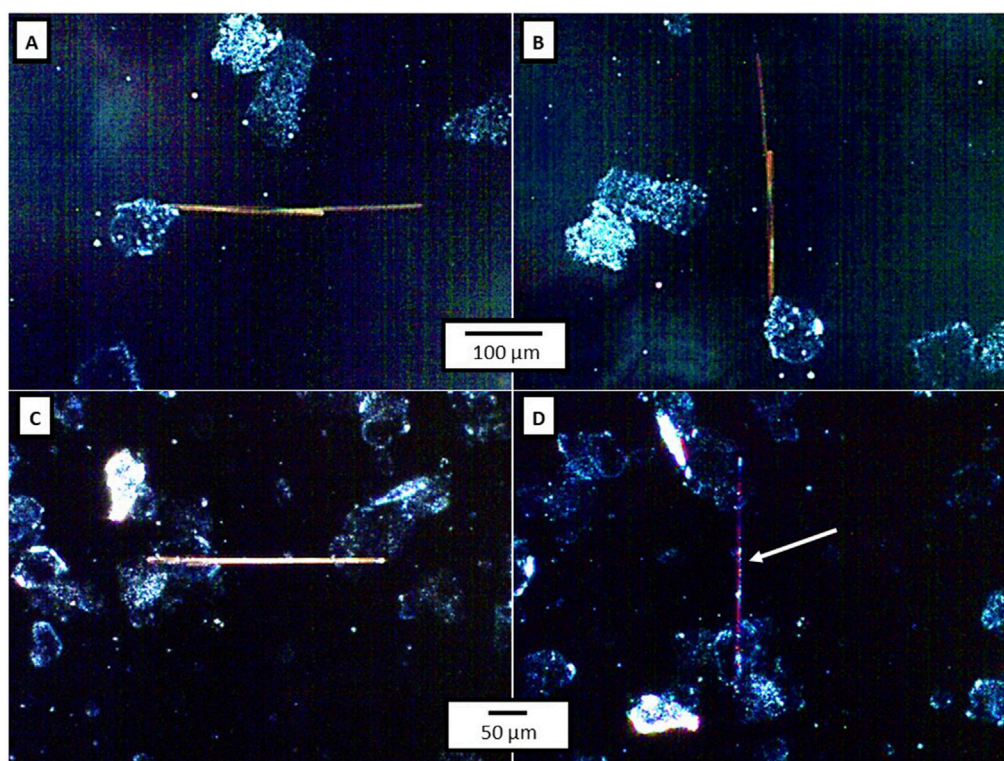
FIGURE 4
PLM images of a 0.05% (500 ppm) concentration spike **(A,B)** and a 0.01% (100 ppm) concentration spike **(C,D)** of Lone Pine tremolite asbestos standard research material in talc, mounted in 1.605 refractive index liquid. The mixture was co-milled 30 min prior to analysis, and coarser particles were isolated using a 400-mesh sieve on the milled material. Images were taken in dispersion staining mode (original magnification 100×). Images A and C show the characteristic yellow color for amphibole particles in 1.605 liquid with particle elongation (and γ) oriented parallel to the lower E-W polarizer, indicating refractive indices are greater than 1.605. Images B and D show lower relief and closer-to-matching dispersion staining colors, characteristic of tremolite with particle elongation oriented in the perpendicular direction (measuring α'). In contrast, the dispersion staining colors of talc platelets (light blue) confirm that the refractive indices of talc are lower than 1.605. Note that yellow and light blue dispersion staining colors are not sufficiently close to the 1.605 liquid for quantitative refractive index determination.

with much smaller dimensions at higher TEM magnifications. Bundles may also be disaggregated due to sonication during the TEM preparation process.

3 Results

3.1 Lone Pine tremolite standard research material spiked into talc (500 ppm and 100 ppm concentrations)

SEM images of 500 ppm and 100 ppm preparations of Lone Pine tremolite asbestos spiked into talc (Figure 2) show the classic morphological features of asbestos for the coarser particles of the amphibole component isolated using wet-sieving techniques. Multiple fiber bundles were observed, as well as very high aspect ratio, long, thin fibers, and fibers showing curvature, despite aggressive grinding for 30 min prior to analysis.

Magnifications used for SEM analyses of preparations with Lone Pine tremolite asbestos (500–1,000×) are at least an order of magnitude lower than those used for TEM analysis (typically 20,000×). Magnifications are qualitatively chosen after a quick scan of the material for the largest particles of interest. Particles

often have lengths that exceed the field of view at these low magnifications. However, using even lower SEM magnifications, where lengths of these particles are completely within the field of view, made small-diameter fibers much less visible. Energy-dispersive X-ray spectroscopy (EDS) was used with SEM to confirm particles had chemistry consistent with tremolite (refer to Supplementary Material for representative SEM-EDS data for the spiking materials).

For PLM analysis, morphological features were best seen at 400×, the highest magnification for conventional PLM (Figure 3), after screening for particles using the dispersion staining technique at 100× magnification (Figure 4). At 400× magnification, bundles with a fibrillar internal structure were visible, and curvature was often seen in long, thin fibers and bundles, composed of smaller individual fibrils. Although some fibers appeared to lack curvature, some "straight" fibers were actually composed of twisted fibrils, visible at higher magnifications.

The characteristic yellow dispersion staining color for amphibole in 1.605 refractive index liquid was displayed when particle orientation (and the γ index of refraction) was oriented parallel to the E-W polarizer, indicating the refractive index is greater than 1.605. In the perpendicular orientation, where α' is measured, a variable dispersion staining color was displayed
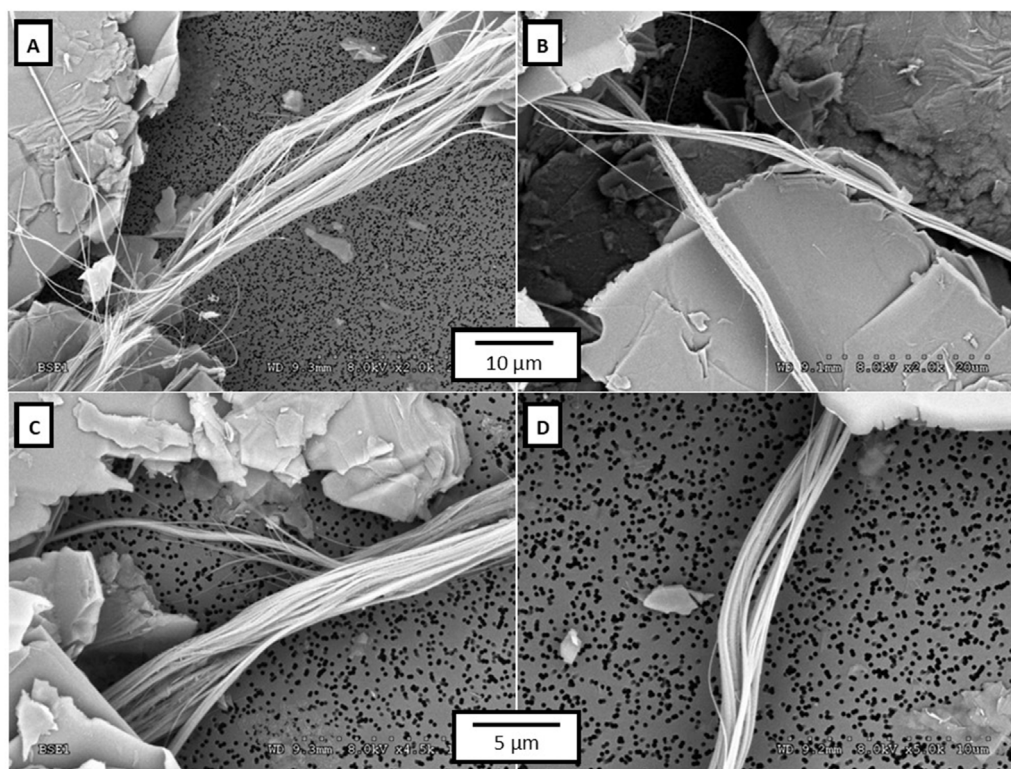
FIGURE 5
SEM images of a 0.05% (500 ppm) concentration spike **(A,B)** and a 0.01% (100 ppm) concentration spike **(C,D)** of HSL chrysotile standard reference material in talc. The mixture was co-milled 30 min prior to analysis, and coarser particles were concentrated using a 400-mesh sieve on the milled material. Original magnifications were 2000× and 5,000×, respectively. Classic asbestiform features, such as bundles and long, thin fibers showing curvature, were clearly visible despite aggressive grinding.

depending on the crystallographic orientation of the particle. However, in all cases, the perpendicular particle orientation shows lower relief and closer-to-matching dispersion staining color for amphibole in 1.605 refractive index liquid. Amphibole particles were easily distinguished from talc, which displays light blue dispersion staining colors in 1.605 liquid, indicating that the refractive indices are lower than 1.605. Scanning by dispersion staining at 100× was shown to be effective for this sample.
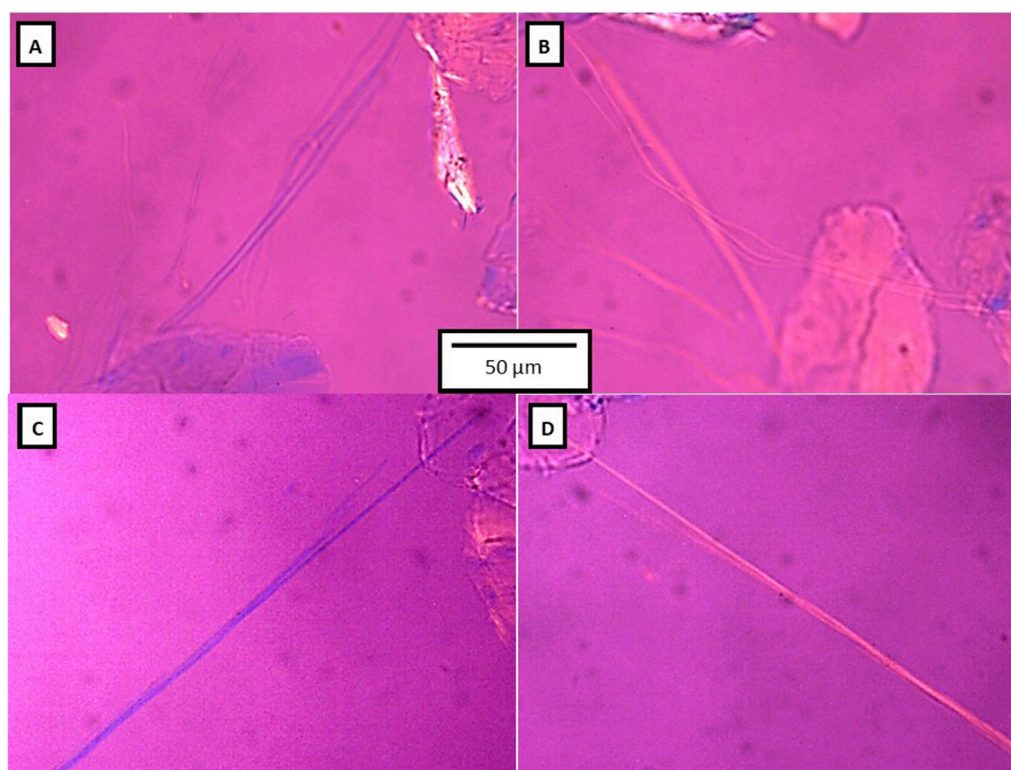
3.2 HSL chrysotile standard reference material spiked into talc (500 ppm and 100 ppm concentrations)

SEM images of 500 ppm and 100 ppm preparations of HSL chrysotile spiked into talc show classic morphological features of asbestos for the coarser particles, isolated using the wet sieve technique (Figure 5). Fiber bundles composed of very high aspect ratio, long, thin fibrils were observed, despite aggressive grinding for 30 min prior to analysis. Fiber bundles show the typical wavy curvature features of chrysotile bundles. Energy-dispersive X-ray spectroscopy (EDS) was used with SEM to confirm particles had chemistry consistent with chrysotile (refer to Supplementary Material for representative SEM-EDS data for the spiking materials).

Magnifications used for SEM analyses (2000–5,000×) are an order of magnitude lower than those used for TEM (typically 20,000×) but higher than those needed for the Lone Pine tremolite sample. Magnifications are different for chrysotile vs. amphibole asbestos spikes due to the different responses of these materials to the grinding procedure. It was unexpected that chrysotile bundles would be so prevalent and easily detected by SEM at these magnifications. Individual fibrils not occurring in bundles were not detected as expected, but the presence of large bundles of chrysotile confirms its resistance to grinding, maybe even more so than for amphibole asbestos. Particles of this size would likely not be included in typical TEM preparations, as discussed in the Materials and Methods Section.

For PLM analysis, morphological features were best seen at 400×, the highest magnification for conventional PLM (Figure 6), after screening for particles using the dispersion staining technique at 100× magnification (Figure 7). At 400× magnification, bundles with a fibrillar internal structure were visible, displaying the classic wavy curvature of chrysotile. Chrysotile fibers within the bundles showed much more curvature than in the Lone Pine tremolite bundles.

Classic dispersion staining colors were displayed (magenta parallel to the E-W polarizer and deep blue perpendicular to the E-W polarizer). These were easily distinguished from the characteristic yellow dispersion staining color displayed by the talc particles. It had previously been speculated that chrysotile would be difficult to detect by PLM due to the

FIGURE 6
PLM images of a 0.05% (500 ppm) concentration spike **(A,B)** and a 0.01% (100 ppm) concentration spike **(C,D)** of HSL chrysotile standard reference material in talc, mounted in 1.55 refractive index liquid. The mixture was co-milled 30 min prior to analysis, and coarser particles were isolated using a 400-mesh sieve on the milled material. Images were taken with crossed polars and the first-order red accessory plate inserted (original magnification was 400×). Colors indicate a positive sign of elongation consistent with chrysotile. Bundles show the wavy extinction characteristic of chrysotile, and the asbestiform morphology is easily distinguishable.

thin dimensions of individual fibrils. However, bundles in the coarse size fraction of talc spiked with trace amounts of chrysotile appear to be prevalent and were easily detected by PLM when the coarser particles are isolated for analysis by the wet sieve technique.

3.3 Calidria chrysotile sample spiked into talc (500 ppm and 100 ppm concentrations)

SEM and PLM images of 500 ppm and 100 ppm preparations of Calidria ("short-fiber") chrysotile spiked into talc show classic morphological features of asbestos for the coarser particles isolated using the wet sieve techniques (Figure 8). Fiber bundles are composed of thin fibrils with a high aspect ratio despite aggressive grinding for 30 min prior to analysis. Fiber bundles are much smaller than the HSL chrysotile but still show the typical curvature features of chrysotile. Energy-dispersive X-ray spectroscopy (EDS) was used with SEM to confirm particles had chemistry consistent with chrysotile (refer to Supplementary Material for representative SEM-EDS data for the spiking materials).

The magnifications needed for morphology analysis by SEM of Calidria chrysotile (20,000×) are similar to those used for TEM and are higher than the magnifications needed for both Lone Pine tremolite asbestos and HSL chrysotile. This "short-fiber" chrysotile deposit is known to have much smaller chrysotile particles than other longer-fiber deposits of chrysotile, so this is not an unexpected result. The difference is that this analysis concentrated on the coarsest particles, isolated by wet sieving, rather than the finer-sized particles typical in TEM preparations. It was unexpected that chrysotile bundles would be so prevalent and easily detected by SEM in the coarse size fraction of Calidria chrysotile. Individual fibrils not occurring in bundles were not detected as expected, but the intact bundles of chrysotile present confirm its resistance to grinding, even for this fine, short-fiber chrysotile. Although particles of this size would likely be included in typical TEM preparations, they may be missed due to the more prevalent presence of particles with smaller dimensions by TEM analysis.

For PLM analysis, morphological features were best seen at 400×, the highest magnification for conventional PLM (Figure 9), after screening for particles using the dispersion staining technique at 100× magnification (Figure 10). At 400× magnification, bundles and fibers show curvature, but particle sizes were much smaller than HSL chrysotile and more difficult to detect. Particles appear to be close to the resolution of the optical microscope but were still detectable.

Dispersion staining colors were blue to blue/magenta parallel to the E-W polarizer and blue perpendicular to the E-W polarizer. Relief is low in both orientations, although slightly lower in the parallel orientation, indicating ɣ is closer to 1.55. In contrast, talc shows the characteristic yellow dispersion staining colors in the
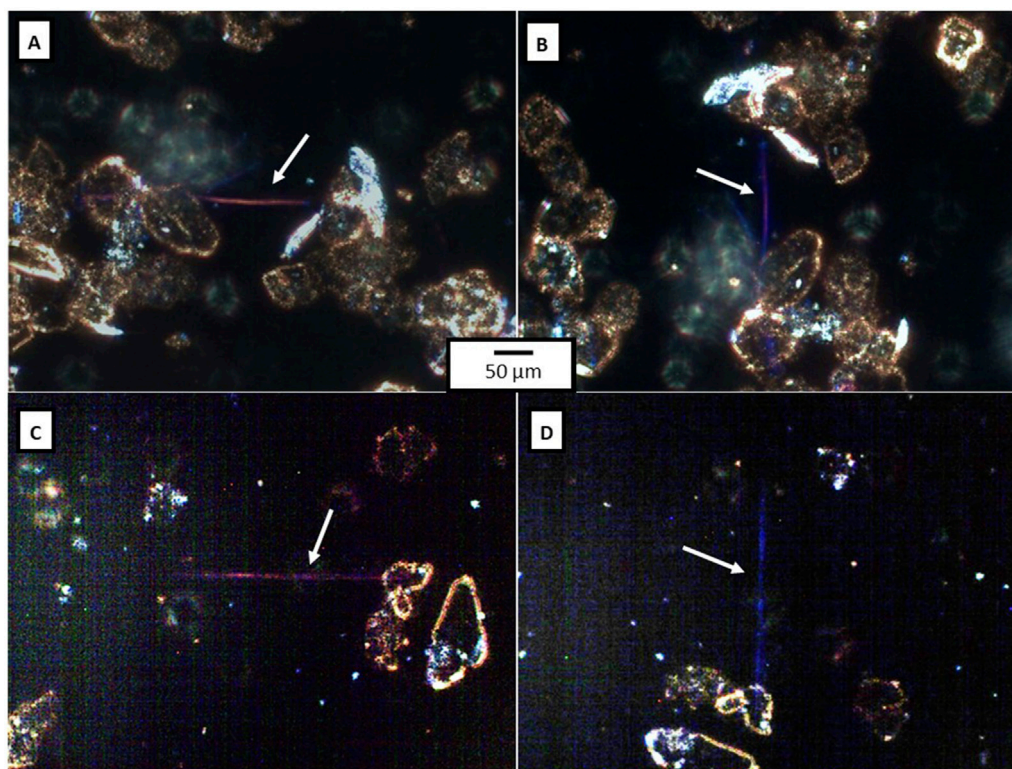
FIGURE 7
PLM images of a 0.05% (500 ppm) concentration spike **(A,B)** and a 0.01% (100 ppm) concentration spike **(C,D)** of HSL chrysotile standard reference material in talc, mounted in 1.55 refractive index liquid. The mixture was co-milled 30 min prior to analysis, and coarser particles were isolated using a 400-mesh sieve on the milled material. Images were taken in dispersion staining mode (original magnification 100×). Images A and C show the characteristic magenta color for chrysotile particles in 1.55 liquid with particle elongation (and γ) oriented parallel to the lower E-W polarizer. These colors, along with lower relief in this orientation, indicate that refractive indices are a near match to 1.55. Images B and D show slightly higher relief, and a blue dispersion staining color indicates that the α refractive index is lower than 1.55. In contrast, dispersion staining colors of talc platelets (yellow) confirm that the refractive indices of talc are higher than 1.55. Note that yellow and light blue dispersion staining colors are not sufficiently close to the 1.55 liquid for quantitative refractive index determination.

1.55 refractive index liquid. Although more difficult to detect by PLM than the HSL chrysotile, the fact that Calidra chrysotile could be found by scanning at 100× magnification was a completely unexpected result, as it had previously been speculated that standard types of chrysotile would be difficult to detect by PLM, due to the thin dimensions of individual fibrils, and it would be even more difficult to detect fine, short-fiber chrysotile, such as Calidria.

3.4 Ultrafine production-scale milled talc sample containing a tremolite asbestos component

In this ultrafine sample, milled at production-scale operations to a median particle size of 1.5 μm, tremolite asbestos particles were present in the coarsest fraction isolated by passing 100 g of the starting material through a 400-mesh sieve. An unexpected result was the low magnifications needed to observe the resulting asbestos fibers, as it was suspected that TEM would be the only technique to adequately characterize the ultrafine particles. However, the wet sieve preparation technique was shown to effectively concentrate larger particles, including asbestos, facilitating SEM and PLM analysis. In fact, fibers concentrated by the wet sieve technique were observed by low magnification stereomicroscopy (~60×) after washing from the sieve and prior to preparation for SEM and PLM.

In this sample, bundles of tremolite asbestos were present along with those that retained their extremely high aspect ratios and showed curvature (Figure 11). These were detected at surprisingly low magnifications by SEM and PLM, both at 400× in this analysis. Further, the wet-sieving technique successfully isolated coarse particles in the range that could be analyzed by PLM, even though the sample had been milled to an ultrafine specification. Conventional PLM preparations, where coarser particles were not concentrated, were unsuccessful at finding these particles due to the overwhelming prevalence of the finer particles. Note that finer particles are difficult to characterize by PLM, and eliminating them by wet sieving greatly enhances the likelihood of encountering particles with information about the habit of the phase that can be more easily characterized.

TEM analysis of the finer samples by typical TEM preparation methods showed shorter particles with parallel sides (Figure 12), but particles detected by TEM were biased toward smaller particles that did not display the typical asbestiform characteristics observed by SEM and PLM. Many of these appeared to be fragments and are most likely the particles that represent the non-asbestiform component in this natural-mined sample (Figure 13). None of
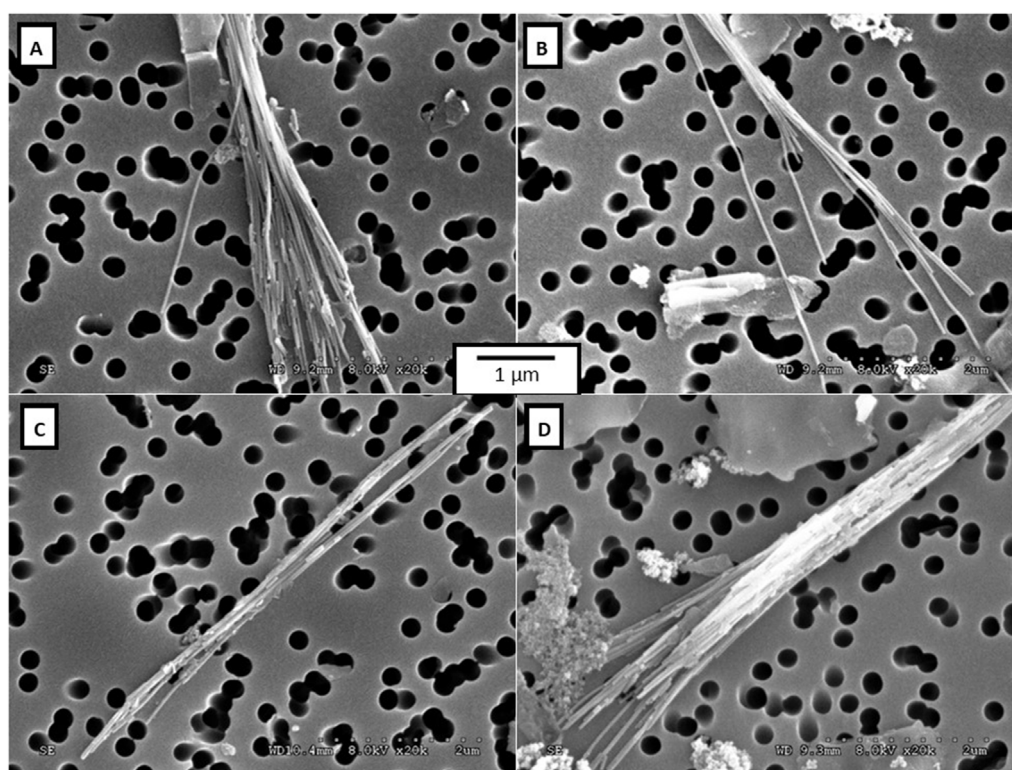
FIGURE 8
SEM images of a 0.05% (500 ppm) concentration spike **(A,B)** and a 0.01% (100 ppm) concentration spike **(C,D)** of a short-fiber Calidria chrysotile sample in talc. The mixture was co-milled 30 min prior to analysis, and coarser particles were concentrated using a 400-mesh sieve on the milled material. Original magnification was 20,000×. Classic asbestiform features, such as bundles and long, thin fibers showing curvature, were clearly visible despite aggressive grinding, although bundles are much smaller in size than spikes using HSL chrysotile.

the extremely long fibers concentrated and detected by SEM were detected by TEM. The total amphibole content of the sample was calculated to be 160 ppm by TEM quantification procedures (American Society for Testing and Materials, Method ASTM, 2008). In this procedure, a known amount of material is suspended in water, and an aliquot is taken from a serial dilution. The fraction of the amount analyzed in a specific number of grid openings of a specific size is compared to the original starting weight to provide a quantitative result. However, this included tremolite particles that were obviously not asbestos; therefore, the content of the actual asbestiform component, while unknown, is estimated to be less than 160 ppm.

4 Discussion

4.1 Overview

The most significant finding of this research is that despite aggressive grinding of the mineral powder, coarse asbestos particles were detectable and retained classic asbestiform morphological characteristics, such as occurrence in bundles and presence of long, thin fibers showing curvature, as measured by SEM and PLM. This was true for samples prepared with known concentrations and for the ultrafine natural talc sample with

unknown concentration that was aggressively ground at production-scale milling operations.

Also significant is that there was no difference in morphology between 500 ppm and 100 ppm asbestos particles in terms of the size and shape of fibers and bundles, although fewer numbers of asbestos particles are present in 100 ppm than in 500 ppm samples, as would be expected. This shows that if particles are properly identified, the approach of isolating and analyzing the coarse part of the particle size distribution is an effective way to detect asbestos, and methods using this approach are limited only by the amount of material analyzed.

The production-scale talc sample provides proof of concept for two major conclusions: a) wet sieving to isolate the coarsest particles in any milled mineral mixture is effective for determining asbestiform vs. non-asbestiform morphology, and b) a preparation that involves grinding, along with wet sieve concentration, is a valid approach for preparing and analyzing standards with known concentrations to evaluate detection limits. Analysis of this sample supports the conclusion that characterization of a mineral's habit is best achieved by evaluating particles found in the coarsest size fraction of the mixture. Further, if asbestos is present in a mineral powder mixture, analysis by SEM and PLM of particles in the coarsest size range show features that are consistent with conventional descriptions of asbestiform (U.S. Environmental Protection
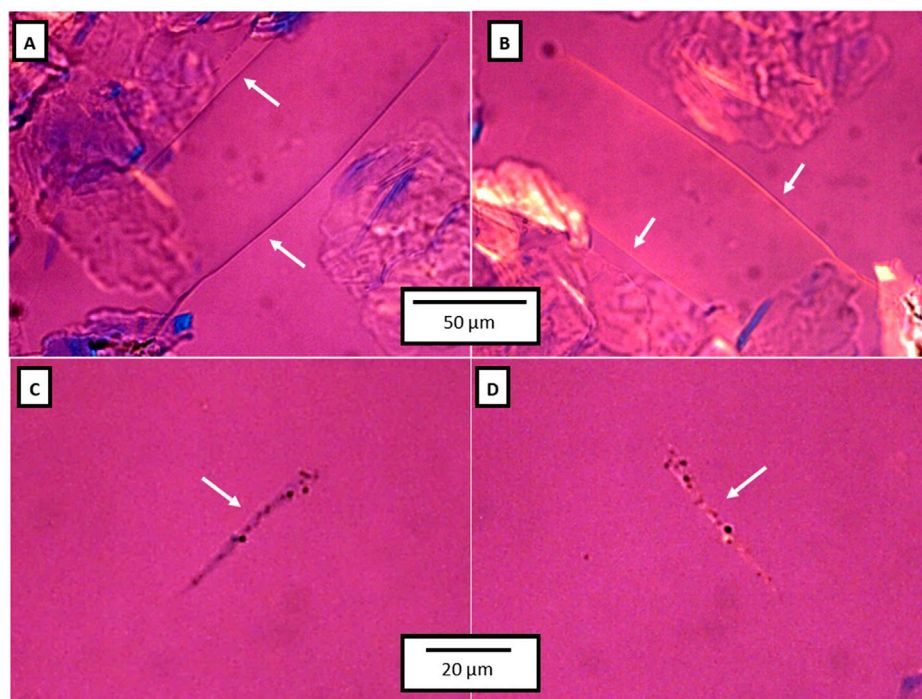
FIGURE 9
PLM images of a 0.05% (500 ppm) concentration spike **(A,B)** and a 0.01% (100 ppm) concentration spike **(C,D)** of a short-fiber Calidria chrysotile sample in talc, mounted in 1.55 refractive index liquid. The mixture was co-milled 30 min prior to analysis, and coarser particles were concentrated using a 400-mesh sieve on the milled material. Images were taken with crossed polars and the first-order red accessory plate inserted (original magnification was 400×). Colors indicate a positive sign of elongation consistent with chrysotile. Bundles and fibers show curvature, but particle sizes were much smaller and difficult to detect.

Agency USEPA, 1993; International Organization for Standardization ISO, 2012; Health and Safety Executive of the U. K. HSE, 2021), regardless of the degree of milling.

If an asbestos component is present in a ground mineral powder that contains amphibole or serpentine, the techniques described in this study can be used to conclude whether an asbestos component is present, even if the mixture is aggressively milled. Conversely, the absence of the features associated with asbestiform morphology (i.e., occurrence in bundles and long, thin particles that show curvature) would indicate that the sample is non-asbestiform or does not contain an asbestos component within the confines of established method detection limits.

Because multiple particles were observed and characterized in preparations of known concentrations of 500 ppm and 100 ppm by both SEM and PLM for all asbestos types, ultimate detection limits using these tools are expected to be significantly lower than 100 ppm. In samples that contain both an asbestiform and non-asbestiform component, the likely situation if a natural milled mineral powder contains asbestos, the asbestiform component would also be confirmed by results of analysis of the coarser size range of particles, as shown in the production-scale ultrafine milled talc sample.

It is apparent that SEM provides the highest resolution for evaluation of surface morphology features. This is due to the focus conditions, with SEM having a greater depth of field and greater surface sensitivity. In addition, SEM has the ability to resolve individual fibrils with minimal widths and should be able to distinguish the "ribbon" morphology of elongate talc particles at higher magnifications and by EDS elemental analysis of the particles. However, EDS may not provide definitive identification of particles for all interferences, for example, the anthophyllite/talc interference, for which particles could have similar chemistries.

Asbestiform morphology can also be observed by PLM in sieve-isolated particles, especially at magnifications higher than those used for dispersion staining screening, although fine focus is more difficult to document for conventional optical microscopy techniques due to the lower depth of field. Although optical techniques have a resolution limit greater than the widths of single fibrils of chrysotile and near the resolution limit of the widths of single fibrils of amphibole asbestos, results show that composite particles in the size range easily characterizable by optical methods are present in ground mineral mixtures containing asbestos, even at trace levels. This confirms that PLM can be appropriate for asbestos analysis in mineral powders, even for those that were aggressively ground.

Although TEM is known to have the highest point-to-point resolution of the microscopy methods, asbestiform characteristics were not as readily apparent in the finer particles analyzed by TEM of the production-scale ultrafine talc sample. In this sample, asbestiform characteristics were much more readily detected when the coarser particles were analyzed by SEM and PLM, indicating that TEM of the finest particles may not be necessary to achieve detection in milled bulk mineral powders.
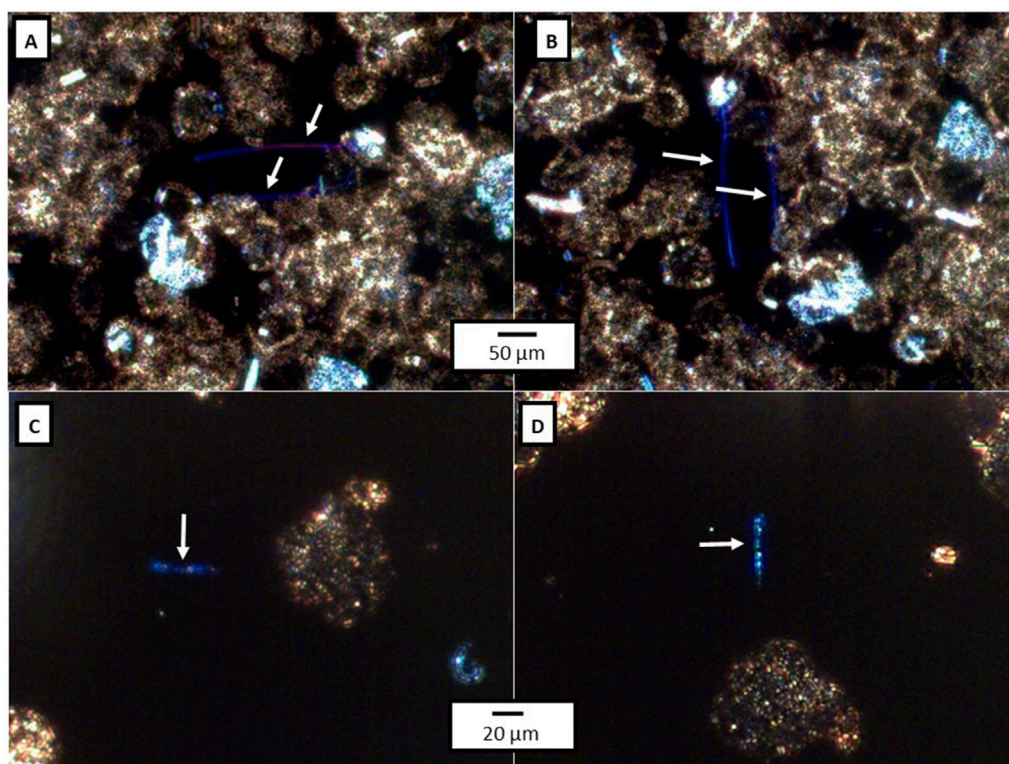
FIGURE 10
PLM images of a 0.05% (500 ppm) concentration spike **(A,B)** and a 0.01% (100 ppm) concentration spike **(C,D)** of a short-fiber Calidria chrysotile sample in talc, mounted in 1.55 refractive index liquid. The mixture was co-milled 30 min prior to analysis, and coarser particles were isolated using a 400-mesh sieve on the milled material. Images were taken in dispersion staining mode (original magnification 100×). Images A and C show a blue dispersion staining color with particle elongation (and ɣ) oriented parallel to the lower E-W polarizer. The blue color in the parallel orientation differs from the HSL sample but can also be consistent with chrysotile. Images B and D show slightly higher relief and a blue dispersion staining color. Dispersion staining colors and relief indicate that refractive indices are lower than 1.55, with ɣ slightly closer to the liquid. In contrast, the dispersion staining colors of talc platelets (yellow) confirm that the refractive indices of talc are higher than 1.55. Note that the yellow and light blue dispersion staining colors are not sufficiently close to the 1.55 liquid for quantitative refractive index determination.

4.2 Particle size distribution of asbestos in mixed ground mineral mixtures

Ground natural mineral powders provide several advantages for asbestos analysis over bulk building materials. In bulk mineral powders, the entire particle size distribution is present for all phases, including coarse and fine particles that are appropriate for all microscopy techniques, including optical microscopy, scanning electron microscopy, and transmission electron microscopy. This contrasts with other types of samples that may only contain specific particle sizes, such as naturally size-selected air samples, which contain only finer airborne-sized particles, or bulk building materials into which specific size ranges of asbestos as a processed raw material may have been intentionally added. Therefore, bulk mineral powders provide a unique opportunity to study the full particle size distribution.

Particle size distribution curves of typical milled mineral products, in this case, coarse and fine talc products, are shown in Figure 14. Similar curves can be produced for all ground mineral types. These are typically not measured by microscopy techniques but by bulk particle size analysis techniques, such as laser diffraction or techniques based on sedimentation rates. In this case, the particle distribution curves were measured using a Malvern® laser particle analyzer. Particles show a range from coarse to fine for both ground mixtures, with a shift toward a finer median for the sample ground to a finer particle size specification. The differential grinding effect between talc and asbestos makes concentration by the sieve technique even more effective for asbestos than other matrix minerals. The differential grinding effect was also effective between asbestos and non-asbestos varieties of the same mineral (Pier, 2014; Pier and McCarthy, 2011).

While analysis for asbestos has been thought to have the best sensitivity when analyzing the smallest particles, this study shows that asbestos not only exists as individual fibrils, but also as composite bundles when the entire particle size distribution is present. In fact, theoretical calculations of analytical sensitivity for the smallest particles detectable by optical microscopy vs. the smallest particles detectable by TEM methods, 5 by 0.25 μm and 0.5 by 0.025 μm, respectively, indicate that the PLM may be more sensitive in certain cases on a fibers-per-gram basis, considering the total amount of material analyzed. It should be noted that typical amounts analyzed by PLM (approximately 5 mg) are 50,000 times larger than amounts typically analyzed by
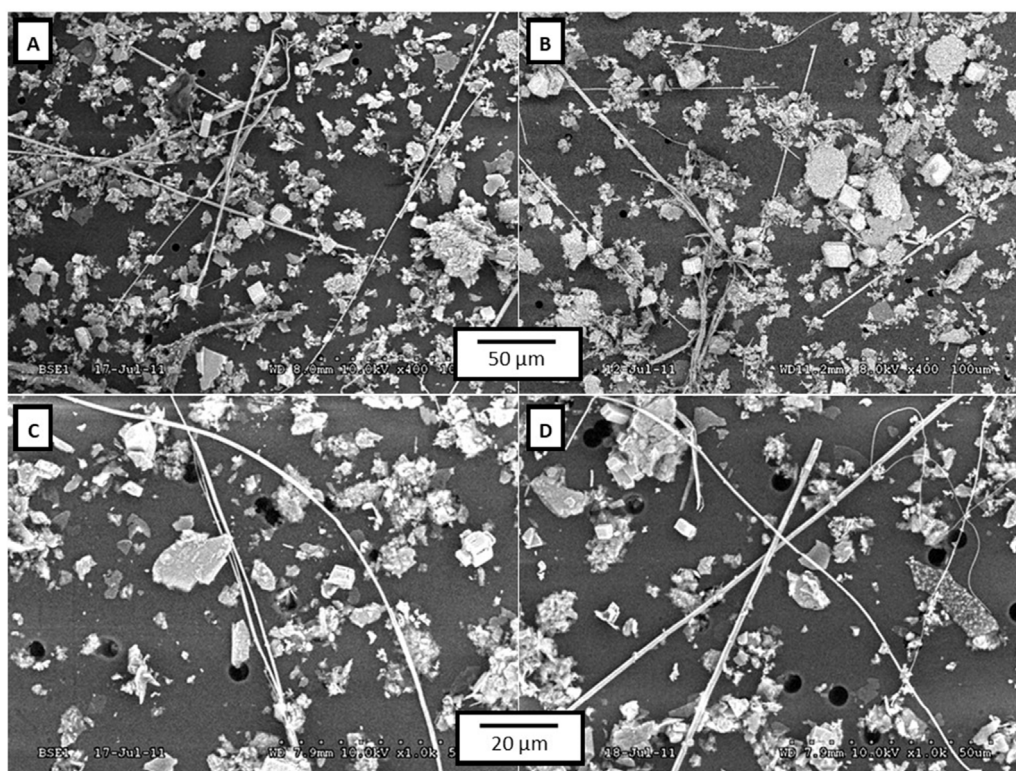
FIGURE 11
SEM images of an ultrafine milled natural mineral sample (talc) that contained a trace amount of tremolite with a component that was asbestiform. SEM original magnifications were 400× **(A,B)** and 1,000× **(C,D)**. Additional milling was not necessary, as the sample had been ground on production-scale equipment to a median particle size of 1.5 μm (measured by laser particle size analysis). Chemical analysis, performed by EDS analysis with SEM, was consistent with tremolite.
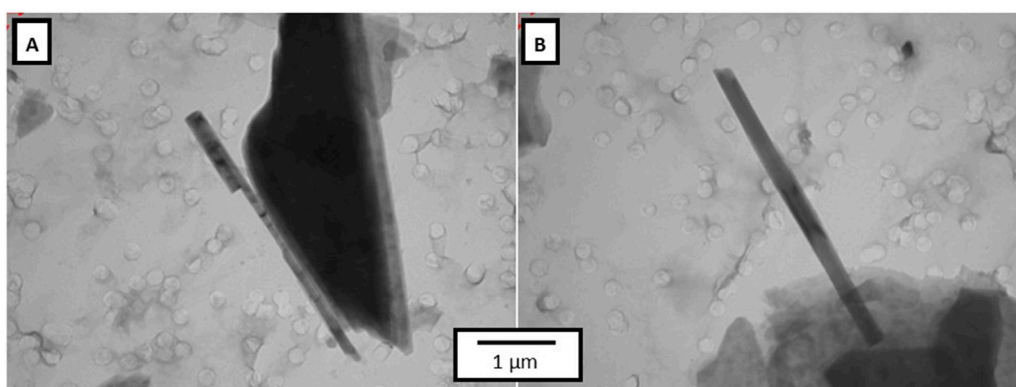

FIGURE 12
TEM images of the unconcentrated ultrafine milled natural mineral sample (talc) that contained a trace amount of tremolite with a component that was asbestiform. The original magnification was 20,000×. TEM analysis showed the presence of higher aspect ratio particles, some with parallel sides (shown in images **A** and **B**), but none similar to the particles as detected by SEM (Figure 11).

conventional TEM (approximately 0.1 μg). With the concentration effects of wet sieving, the likelihood of encountering larger particles by PLM is further enhanced. The fact that PLM analysis could be more sensitive than conventional TEM analysis for bulk materials was shown empirically by Sanchez et al. (2023).

4.3 Use of talc as a matrix material in mixed ground mineral mixtures

The differential grinding phenomenon (Pryer, 1958) is particularly evident when considering mixtures of talc that might contain asbestos. Serpentine and amphibole in all forms have higher
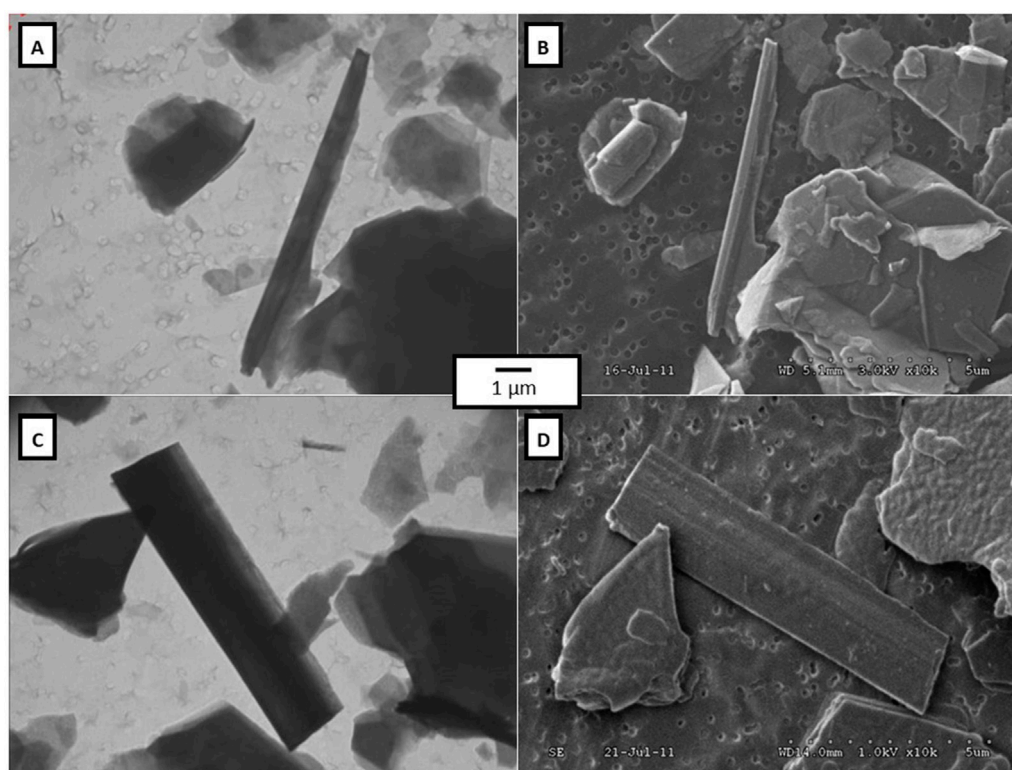
FIGURE 13
TEM images of an ultrafine milled natural mineral sample (talc) that contained trace amounts of tremolite with a component that was asbestiform. Numerous particles were present that did not show obvious asbestiform characteristics by TEM (images **A** and **C**). These same particles were relocated for SEM analysis for morphology analysis (images **B** and **D**). Compare morphology of these particles with that of asbestos fibers in Figure 11. TEM original magnification was 20,000×; corresponding SEM images were taken at 10,000× and enlarged.
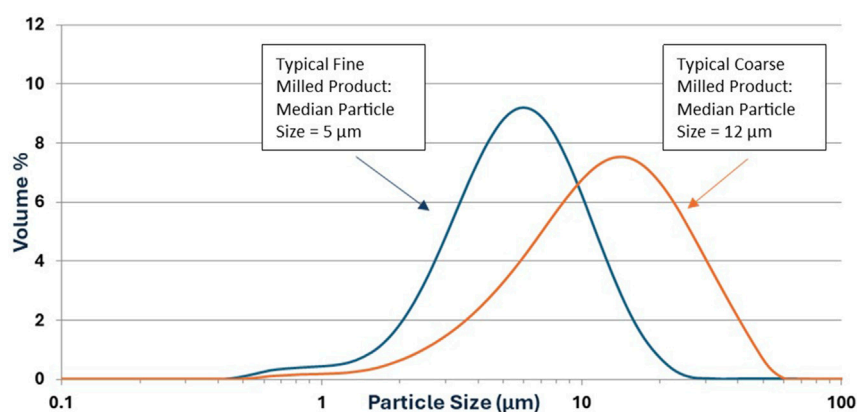

FIGURE 14
Particle size distribution curves of a typical coarse milled talc product (orange) vs. a typical fine milled talc product (blue) measured using a Malvern® laser particle size analyzer. The median particle size of the coarse ground product was 12 μm, and that of the finely ground product was 5 μm. Particles show a range from coarse to fine for both distributions, with a shift toward a finer median for the more finely ground product.

Mohs hardness values than talc, so they are naturally more difficult to grind than talc, the softest mineral known, with a Mohs hardness value of 1. The asbestiform variety of serpentine and amphibole, with its elevated tensile strength, has an even greater resistance to grinding. It follows that the differential grinding profile of asbestos makes it not only preferentially occur in the larger particle size range of a talc–asbestos mixture, but the difference increases as grinding increases, thus justifying analysis of the larger particles to detect asbestos. In addition, the fact that this property is preserved after grinding may be an indirect measurement of tensile strength.

Grinding concerns with asbestos in talc were noted in a previous study by Pang et al. (1987). In this study, it is indicated that asbestos fibers in talc matrices (1% and 0.1%) were reduced in size by grinding, and that the number of elongate particles detected was reduced for longer periods of grinding as analyzed by TEM methods. However, because TEM was used to count and document particles and the preparation procedure for TEM (aliquot withdrawal from a suspension of particles) naturally selects for the finer size fraction particles, larger particles may not have been evaluated. It is possible that more information may have been obtained by the observation of the coarser particles in the ground mixtures by SEM and/or PLM, especially after these are concentrated using the wet sieve technique. Although it is acknowledged that grinding does generally reduce particle size, as noted by the shifting of the entire particle size distribution curves toward the finer range (refer to Figure 14), the current study shows that coarse asbestos particles should still be present, even in the finest grinds of talc/asbestos mixtures. Therefore, concern for false negatives may be offset by analyses of coarse particles, as confirmed by the ultrafine talc sample containing an asbestos component that was an order of magnitude lower than the concentrations used in the Pang study.

4.4 Air vs. bulk methodology used for measuring asbestos particles in ground mineral powders

Traditional air methods utilizing TEM directly measure particles per volume of air (i.e., particles are not further manipulated during preparation, so fiber counts are applicable). While TEM methods can positively distinguish between chrysotile and non-asbestiform serpentines, which have unique selected area electron diffraction (SAED) patterns, TEM is not necessarily definitive between amphibole asbestos and non-asbestos amphibole of the same mineral for individual particles, which may not have unique SAED patterns (International Organization for Standardization ISO, 2019a). Further, a natural bias exists toward measuring the finer particles, which may not be definitive for asbestos nor proven to be associated with disease, by TEM.

Air methods by TEM, which typically call for documenting particles that have ≥3:1 AR for lengths ≥5 μm (WHO) or ≥5:1 AR for lengths ≥0.5 μm (AHERA), do not mandate further distinction between asbestos and non-asbestos particles for the regulated phases in air. However, TEM air methods, initially developed to measure asbestos in the built environment, especially in abatement clearance situations (U.S. Environmental Protection Agency USEPA, 1987), where the presence of asbestos can be inferred, do not adequately describe the morphology of bulk mineral powders. In the built environment or in abatement situations, the analyte is more likely to be one of the commercial asbestos types, if asbestos is present, rather than one of the mixed or non-asbestiform non-commercial types. TEM is not used for bulk building materials in the AHERA rule, and although TEM is permitted in the updated 1993 EPA bulk method, aspect ratios of 3:1 and/or 5:1 are not mentioned as definitive identifiers of asbestos.

As size classes documented in air methods are not diagnostic for asbestos in all environments, parameters used in air methods are not

appropriate for the analysis of natural bulk mineral powders. In addition, bulk minerals require significant manipulation during preparation and analysis, so fiber counts may not be relevant. For bulk mineral samples in natural (non-built) environments that contain mixed morphologies, the ISO 22202-1 and ISO 22262-2 methods recommend that further analysis may be necessary to adequately distinguish between asbestos and non-asbestos phases (International Organization for Standardization ISO, 2012; International Organization for Standardization ISO, 2014).

Using techniques that cover the full particle size distribution for a ground mineral mixture will help to fully characterize the material, rather than relying on techniques that predominantly analyze the finest particles. Performing only TEM on bulk mineral powders without complementary techniques that cover all particle sizes may lead to misleading results and improper identification of phases, as the different particle size classes can show vastly different morphological features.

The results of this study confirm that an analysis of coarser particles in bulk mineral powders using methods such as SEM and PLM is more effective than TEM for determination of the mineral habit, as the larger particles are most likely to display classic asbestiform features if they are truly asbestos. This determination is further enhanced when particles are concentrated using the wet sieve technique, which is especially effective for trace amounts. Therefore, techniques described in this study may aid in the conclusive determination of the presence of asbestos in ground bulk mineral powders.

5 Conclusion

This study shows that finely ground mineral products have a larger component that can be isolated and concentrated for SEM and PLM analysis, and if asbestos is present, the larger particles display typical asbestos morphology characteristics for conclusive identification, thereby providing confirmation of the habit in ground mineral powders.

Analysis of the full spectrum of particle size classes for fully characterized materials that contain trace amounts of asbestos ensures that analyses can proceed with limited confounding factors to address health effects of asbestos vs. non-asbestos EMPs of the same regulated mineral, different toxicities of different asbestos types, and different toxicities of different size classes as proposed in the NIOSH Roadmap (National Institute for Occupational Safety and Health NIOSH, 2011).

The following conclusions from this study are summarized:

- Coarser asbestos particles isolated by wet sieving, in spiked mineral mixtures, as well as in a production-scale ultrafine ground talc sample, retain classic asbestiform morphology characteristics, such as occurrence in bundles and presence of long, thin fibers showing curvature, despite aggressive grinding.
- A production-scale ultrafine ground talc sample provides proof of concept that asbestos is effectively concentrated in the coarser size fraction (especially for mixtures with talc) and that coarser particles give the most information about the habit of included mineral phases.

- The morphology of finer particles in a finely ground large-scale milled talc sample containing trace amounts of both asbestos and non-asbestos amphibole is biased toward the non-asbestos component, as measured by TEM, and finer particles are less likely to display classic asbestiform features.
- Isolated coarse asbestos particles in 500 ppm and 100 ppm spiked samples show similar morphology with respect to size and shape of fibers and bundles, although they are fewer in number in lower concentrations, as would be expected. This indicates that detection can be improved by simply scanning more coarse material (i.e., particles ≥5 μm in length) by PLM and/or SEM, and detection of individual fibrils by TEM may not be required.
- Asbestos is confirmed to be resistant to grinding. This is especially true when compared with easier-to-grind talc. Although there are no microscopic procedures to directly assess tensile strength (a parameter associated with asbestos), resistance to grinding is an indirect method for assessing this parameter.
- The ability of PLM to readily detect asbestos particles was unexpected, as it was previously suspected that particles would be below the resolution of the optical microscope, especially for chrysotile. However, both HSL and fine, short-fiber Calidria chrysotile were detected by PLM at 100× magnification and in 100 ppm concentrations.
- Techniques described in this article offer an effective way to determine whether asbestos, or an asbestos component, is present in natural ground mineral powders, even in trace quantities (validated to a detection limit at least as low as 100 ppm by PLM and SEM). This is particularly applicable for studying the health risks of asbestos using accurately characterized materials with minimal interferences.

Data availability statement

The original contributions presented in the study are included in the article/Supplementary Material; further inquiries can be directed to the corresponding author.

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