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Libby Amphibole Contamination in Tree Bark Surrounding Historical Vermiculite Processing Facilities

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ABSTRACT

Over a 70-year period, a mine near Libby, MT supplied nearly 80% of the world's vermiculite. Raw vermiculite, which was contaminated with naturally occurring amphibole in veins throughout the deposit, was shipped to processing sites throughout the United States for exfoliation. In this pilot study, tree bark samples were collected near processing facilities in Spokane, WA, Santa Ana, CA, Newark, CA, and Phoenix, AZ in an effort to determine if areas surrounding these facilities are today contaminated with Libby amphibole asbestos (AA). From areas surrounding each of the four historical processing sites, Libby AA was detected in a subset of the bark samples. At the Santa Ana, Newark and Phoenix facilities, actinolite-tremolite and other high Fe Ca-bearing amphibole were also measured from the bark samples. In addition, chrysotile was frequently measured in samples collected from each of the sites. From the results of this pilot study, it is evident that tree bark can serve as reservoirs of asbestos, and indicators of past and current contamination. These data also suggest that areas outside of these historical processing facilities may today have some level of existing contamination resulting from the operation of these facilities.

***Keywords*:** *Vermiculite, Asbestos, Amphibole, Libby, Exfoliation, Tree Bark*

1. Introduction

Prior to 1990, up to 80% of the world's vermiculite was derived from a mine near Libby, Montana [1]. The vermiculite ore mined from Zonolite Mountain seven miles northeast of Libby was contaminated with fibrous and asbestiform amphibole in veins throughout the deposit [2], containing a combination of winchite (84%), richterite (11%) and tremolite (6%) [3]. As a result of this contamination, occupational exposure to Libby amphibole asbestos (AA) has led to a significant increase of serious respiratory diseases such as lung cancer, pleural cancer and asbestosis among the former mine workers [4-6]. In addition, pleural abnormalities have been defined in 17.8% of the 6668 participants who lived or worked in the Libby area prior to 1991 [7]. In October 2002, Libby was added to the Environmental Protection Agency's (EPA) National Priorities List, and in June 2009 the town of Libby was designated a public health emergency. This is the only time EPA has made such a declaration.

Libby AA has been measured outside of Libby as well. Between the 1920s and 1990s, vermiculite mined from Libby (estimated in the millions of tons) was shipped by railroad to 245 facilities within the US for processing via exfoliation [13]. Exfoliation refers to a commercial process where vermiculite is rapidly heated to expand it into low-density, accordion-like nuggets [13]. At the facilities, the raw vermiculite was typically unloaded manually by workers using shovels and front loaders, and stored on site until transferred to an exfoliation furnace where it was heated to a temperature between 1500 and 2000 °F [14]. The processed vermiculite was then stored on site until packaged in various forms for commercial use such as attic insulation. In facilities such as the Western Mineral Products Site in Minneapolis, Minnesota additional products such as Monokote (a fire proofing material that combined vermiculite and chrysotile asbestos) were also produced [15].

In 2008, ATSDR released a study in which 28 out of

245 sites were selected for detailed evaluation for on-site Libby AA contamination. At many of these facilities, Libby AA was found in exterior soil and indoor dust in areas where vermiculite and waste rock were unloaded or stored. At the present time, many of these former exfoliation sites are occupied with commercial and industrial operations not related to the original exfoliation processes. In addition to on-site contamination, we hypothesize that airborne emissions of Libby amphibole fibers from exhaust stacks and fugitive emissions from vermiculite storage sites may have been dispersed into the areas surrounding these locations.

In this manuscript, we report on a study in which tree bark samples were collected surrounding historical Libby vermiculite processing facilities located in Spokane (WA), Newark (CA), Santa Ana (CA), and Phoenix (AZ). The goal of this research project was to determine if Libby AA emanated from the industrial sites during the periods of operation of these facilities, and if trees surrounding these facilities are today contaminated with Libby AA.

2. Materials and Methods

Tree bark samples were collected in areas surrounding the former vermiculite processing facility in Spokane, WA, on March 9, 2009. Between June 8 and June 10, 2010, tree bark samples were collected in areas surrounding three other former facilities in Newark, CA, Santa Ana, CA and Phoenix, AZ. Bark samples were also collected in Missoula, MT to serve as control samples. The Spokane site (located approximately 160 kilometers from Libby) was selected because it is one of the closest former processing facilities to Libby. The remaining three sites were selected based on a ranking system, considering 1) the tonnage of raw vermiculite processed, 2) the year that exfoliation work was terminated at the site, 3) the population density within one mile of the site and 4) the total duration of site operation. **Table 1** presents the characteristics of each site.

2.1. Tree Bark Sampling

At each location, bark was collected from several tree species native to the area. A pry-bar or spatula was used to collect a ~ 200-gram piece (with surface area between 50 - 150 cm^2) of bark from approximately four feet above the base of each tree. These were placed into labeled plastic bags. The spatula/pry-bar was wiped down after each sample collection with isopropyl alcohol and laboratory tissues. In total, 22 samples were collected from around the Spokane facility. At the Santa Ana, Newark, and Phoenix facilities, 40, 22 and 25 samples, respectively, were collected in proximity to the facilities. Because asbestos fibers can become airborne and easily dispersed, the majority of samples were collected in areas predominantly downwind of the facilities.

2.2. Tree Bark Analyses

Following the MD Webber method [8], samples of approximately 1 gram (normalized to 10 cm^2 surface area) were weighed, dried to stable mass at 60 to 100˚C, ashed at 450˚C for ~16 hours, and re-weighed to determine percentage loss of organic material. Residue, typically 5% of original mass, was suspended in filtered deionized water, thoroughly mixed, and filtered through 0.4-um polycarbonate filters before being prepared for transmission electron microscopy (TEM) analysis using carbon coating and ethylene-diamine dissolution onto TEM grids.

All 22 samples from the Spokane site were analyzed whereas funding constraints limited us to only five samples per site from the Santa Ana, Newark, and Phoenix sites. As the goal of this project was to detect Libby amphibole in the areas surrounding the historical facilities, the five samples from the latter three sites were chosen

Table 1. Description of Vermiculite processing facilities in Spokane, WA, Santa Ana, CA, Newark, CA and Phoenix, AZ.

Factor/Site	Spokane, WA[A]	Santa Ana, CA[B]	Newark, CA[C]	Phoenix, AZ[D]
Years of Operation	1951-1973	1971-1993	1966-1993	1964-1992
Tons Processed	10,317 (between 1967 and 1973)	453,000	337,100	254,900
Pop. Within 1 mile	17,214	35,832	10,183	12,915
Surrounding area use	Commercial and residential	Light industrial and commercial, elementary school (1950-present) 200 yards away	Mixed commercial, industrial and residential	Industrial, commercial and residential
Prevailing wind	Southwest	Southwest	Northwest	Variable, west in the daytime and east in the evening
Sample collection direction from facility	All directions, primarily north and northeast	All directions, primarily north and east	All directions, primarily northeast and southeast	All directions, primarily east

[A]**ATSDR,** Vermiculite northwest, Spokane, WA [16]; [B]**ATSDR,** Fact sheet, Santa Ana, CA [17]; [C]**ATSDR,** Fact sheet, Newark, CA [18]; [D]**ATSDR,** Fact sheet, Phoenix, CA [19].

from trees close to and downwind of the facility. For each sample, four different dilutions were prepared from the ashed bark in an effort to eliminate both over and under-loading of TEM grids.

Once the grids were prepared, they were sent to ALS Laboratories (Cincinnati, OH) for TEM analysis. TEM analysis was performed at a screen magnification of at least 15,000× on a Philips CM-12 TEM with EDAX Genesis System providing Energy-dispersive X-ray analysis (EDXA) capabilities. Identification and measurement of asbestos structures were conducted according to AHERA protocol [20].

3. Results

Table 2 presents the summary of findings from the Spokane site, while **Table 3** presents the results from

Table 2. Summary of tree bark results from the Spokane site.

	Asbestos Concentration (s/cm^2)				Type of tree	Distance from Facility (meters)
	Total asbestos <5 μm		Total asbestos >5 μm			
Sample ID	AA	Chrysotile	AA	Chrysotile		
SPK_1	ND	939,506	ND	ND	American Elm	122
SPK_2	2,400,640	ND	800,213	ND	Douglas fir	61

Note: AA: Libby Amphibole Asbestos; ND: None detected.

Table 3. Summary of tree bark analysis results of Santa Ana, Newark and Phoenix sites.

		Asbestos Concentration (s/cm^2)								
		Total Asbestos < 5 μm			Total Asbestos > 5 μm					
Sample ID	Dilution Prep	AA	A-T	Chrysotile	AA	A-T	Chrysotile	Other Conc. (s/cm^2)	Type of tree	Distance from Facility (meters)
A. Santa Ana Site										
SA_1	2	1,704,683	ND	ND	1,136,456	568,228	ND	ND	Eastern cottonwood	366
	2.5	ND	ND	ND	463,192	ND	ND	ND		
	3.5	ND	ND	333,559	ND	ND	ND	ND		
SA_2	6	469,716	ND	ND	ND	ND	ND	ND	***Blue gum Eucalyptus***	***457***
SA_3	1.5	ND	ND	744,062	ND	ND	ND	744,062	American Elm	610
	2.5	ND	ND	428,992	ND	ND	ND	ND		
	3	ND	ND	713,842	ND	ND	ND	ND		
SA_4	3	ND	ND	ND	ND	ND	649,360	ND	American Elm	732
	3.5	ND	ND	543,881	ND	ND	ND	ND		
B. Newark Site										
NEW_1	4	ND	ND	1,266,526	ND	ND	ND	ND	***Blue gum Eucalyptus***	***701***
NEW_2	1.5	ND	807,883	ND	ND	ND	ND	ND	***Eastern cottonwood***	***152***
	2	ND	ND	634,753	ND	ND	ND	ND		
	2.5	ND	ND	1,485,125	ND	ND	ND	ND		
	3	ND	ND	842,188	ND	ND	ND	ND		
NEW_3	2	530,103	530,103	2,120,412	ND	ND	ND	ND	***California redwood***	***61***
	2.5	ND	ND	ND	ND	ND	ND	429,191		
NEW_4	2.5	ND	ND	1,599,152	ND	ND	ND	ND	Australian pine	213
	3	ND	ND	2,013,383	ND	ND	ND	671,128		
	3.5	ND	ND	1,137,822	ND	ND	ND	ND		
NEW_5	3	ND	ND	ND	ND	ND	ND	645,265	Australian pine	305
	4	ND	ND	480,088	ND	ND	ND	ND		
	5	ND	ND	776,296	ND	ND	ND	ND		
C. Phoenix Site										
PHX_1	3	ND	ND	924,072	ND	ND	ND	ND	Snow Gum eucalyptus	274
	6	ND	473,892	ND	ND	ND	ND	ND		
PHX_2	4	ND	ND	681,027	ND	ND	ND	681,027	Snow Gum eucalyptus	152
PHX_3	3	ND	ND	1,371,575	ND	ND	ND	685,787	Ash-leaf Maple	610
	4	ND	ND	ND	479,941	ND	ND	479,941		

Note: A-T: Actinolite-Tremolite; AA: Libby Amphibole Asbestos; Other fibers: refers to fibers that may be a high Fe Ca-bearing amphibole; ND: None detected.

Santa Ana, Newark, and Phoenix, respectively. Chrysotile and Libby AA were detected in samples collected from trees surrounding the Spokane facility. At the Santa Ana, Newark and Phoenix facilities, actinolite-tremolite (A-T) and other asbestos structures were identified in addition to chrysotile and Libby AA. The term "other fibers" is used in this paper to refer to fibers that appear to be a high Fe Ca-bearing amphibole.

3.1. Spokane Site

Libby AA did not predominate in tree bark samples collected surrounding the Spokane site. Of the 22 bark samples collected and analyzed, only one sample yielded Libby AA (2,400,640 s/cm^2 < 5 μm in length, and 800,213 s/m^2 > 5 μm in length). Another sample revealed chrysotile structures, with a concentration of 939,506 s/cm^2 (<5 μm in length).

3.2. Santa Ana Site

Four of the 40 samples collected were analyzed, on the basis of their location predominantly downwind and near the facility. Two samples yielded AA structures with concentrations ranging from 463,192 to 1,704,683 s/cm^2. Actinolite-tremolite structures (568,228 s/cm^2) were detected in a third sample, while another sample contained Fe Ca-bearing amphibole (concentration of 744,062 s/cm^2). In addition to AA, chrysotile structures were found in three of the four samples with concentrations ranging from 333,559 s/cm^2 to 744,062 s/cm^2 (**Table 3**).

3.3. Newark Site

One of the five samples revealed Libby AA with a concentration of 530,103 s/cm^2 (**Table 3**). The majority of samples yielded chrysotile structures with concentrations ranging between 480,088 s/cm^2 and 2,120,412 s/cm^2. Actinolite-tremolite structures were detected in two samples with concentrations of 807,883 s/cm^2 and 530,103 s/cm^2, respectively. Fe Ca-bearing amphibole fibers were detected in three samples with concentrations ranging between 429,191 s/cm^2 to 671,128 s/cm^2.

3.4. Phoenix Site

Only one of the three samples analyzed from the Phoenix site indicated the presence of Libby AA (479,941 s/cm^2), while another sample revealed actinolite-tremolite fibers with a concentration of 473,892 s/cm^2 (**Table 3**). Chrysotile was detected in all three samples that were analyzed from the Phoenix site, with concentrations ranging from 681,027 s/cm^2 to 1,371,575 s/cm^2. Fe Ca-bearing amphibole fibers were identified in two samples, with concentrations from 479,941 to 685,787 s/cm^2.

3.5. Control Samples

All 11 control samples used for this study were collected from Douglas fir (*Pseudotsuga menziesii)* trees at The University of Montana campus in Missoula. Control samples were treated with the same analytical protocol as the actual samples. For the Spokane bark analytical program, two control bark samples were analyzed in an effort to detect any potential sources of contamination. No Libby AA were detected in any of the nine control samples that were analyzed when processing the samples from the Santa Ana, Newark and Phoenix sites. However, it should be noted that one chrysotile fiber was measured in one of the control bark samples. This chrysotile fiber could have actually been on the control sample (given the historical ubiquity of chrysotile in the 20th century), or it could have been contamination that occurred either during the sample preparation or during lab analysis. At any rate, we are confident that this single chrysotile fiber does not indicate a contamination problem with the analytical program.

4. Discussion

Tree bark has been used since the late 1980s as biomonitors for both inorganic and organic pollutants [21]. Specifically, polychlorinated dibenzo-p-dioxins and dibenzofurans [22], polyaromatic hydrocarbons [23], polychlorinated bipheynls [24-26], organochlorine pesticides [27-28], radioactive analytes [29-30], trace metals [31-39], and persistent organic pollutants [40] have all been studied.

In the present study, Libby AA was detected at each of the four sites in a subset of the trees surrounding the historical processing facilities. Meeker *et al*. [3] conducted the first comprehensive study on Libby asbestos to determine the mineralogy and morphology of both fibrous and non-fibrous amphiboles, supporting the earlier results of Wylie and Verkouteren [41] and Gunter *et al*. [42]. They described the Libby AA as winchite, richterite, tremolite, and magnesioriebeckite, with the majority of structures displaying a gradient of morphologies between prismatic crystals and asbestiform fibers. Libby amphibole has a standard elemental composition of Si > Mg > Ca > Fe > Na > K, with fibers having a mean length of 4.9 μm, and mean aspect ratio of 17. These characteristics were all used when identifying the Libby AA in bark samples collected surrounding the four historical processing facilities.

The non-Libby AA asbestos structures included actinolite-tremolite (EDXA spectra with just Mg-Ca-Fe-Si peaks present, and occasionally minimal Al) and amphibole fibers that were high in iron and calcium were identified in the bark samples. Chrysotile fibers were also detected in tree bark samples collected around the processing facilities at each of four sites. This finding is not surprising, as chrysotile was widely used in thousands of

commercial products from the 1930s through the 1970s, and is still used in asbestos cement, friction materials, roof coatings and gaskets [43]. It is possible that chrysotile's wide spread usage in industry could contribute to its ubiquity in the ambient environment.

Table 4 shows a comparison between asbestos fiber dimensions measured from the bark samples collected in Libby, Spokane, Santa Ana, Newark, and Phoenix. From the tree bark samples collected surrounding the abandoned vermiculite mine in Libby, the majority of the AA measured are less than 5 micrometers in length (mean = 3.4 μm), with a mean diameter of 0.39 μm[8]. Libby AA fibers measured in the Spokane, Santa Ana, Newark, and Phoenix samples were comparable to what was measured in Libby, with mean diameters of 0.37 - 0.50 μm and mean lengths of 2.8 - 5.6 μm. Chrysotile fibers that were measured at each of the sites had mean diameters of ~0.1 μm and mean lengths of ~1.6 μm.

There were limitations to this investigative pilot study. We encountered a problem in determining the correct loading for the grids prior to the TEM analysis of the Santa Ana, Newark, and Phoenix bark samples. The protocol we had developed previously for coniferous trees in northwest Montana [8] did not work as well for trees from other parts of the country. Samples collected from the Santa Ana, Newark and Phoenix sites were from several different regional species of trees [Douglas fir (*Pseudotsuga menziesii*), American elm (*Ulmus Americana*), Eastern cottonwood *(Populus deltoids),* Blue gum Eucalyptus *(Eucalyptus globulus)*, California redwood *(Sequoia sempervirens),* Australian pine (*Casuarina equisetifolia*), Snow Gum eucalyptus (*Eucalyptus pauciflora*) and Ash-leaf Maple (*Acer negundo*)]. Hence a series of dilutions were used (for each sample) in preparing the TEM grids before the correct loading was achieved. Overall, this impacted the number of samples we could analyze from each of the sites due to funding constraints.

5. Conclusions

An EPA assessment published in 2009 showed that Libby amphibole was detected in the soil and indoor dust at the Newark, Santa Ana, and Phoenix historical Libby vermiculite processing facilities [44]. The results from tree bark analyses collected near these same areas (also including the Spokane facility) indicate that trees in the residential/commercial areas surrounding these facilities can serve as reservoirs for asbestos fibers. In addition to amphibole asbestos, chrysotile structures were also detected from the tree bark samples. While amphibole asbestos is most likely associated with the historical Libby vermiculite processing facilities, it is difficult to determine the source of chrysotile structures.

Ewing [45] discusses concentrations of surface dust found in a variety of settings and suggests that a concentration of 1,000 s/cm^2 may be considered clean, whereas concentrations >100,000 fibers indicate contamination. Results from the present study revealed concentrations of chrysotile up to 2 million s/cm^2, concentrations of Libby AA from ND to 2.8 million s/cm^2, actinolite-tremolite from ND to 800,000 s/cm^2, and other fibers ranging from ND to 700,000 s/cm^2. Many of these samples were collected in areas near residential areas, and in some cases near schools. For comparison, the levels measured in Libby were a great deal higher than what was measured in the present study. Specifically, bark samples collected in proximity to the abandoned vermiculite mine in Libby measured over 100 million s/cm^2 bark surface.

Adgate *et al*. (2011) estimated potential cumulative asbestos exposures to non-occupational individuals in areas surrounding a historical Libby vermiculite processing facility in Minneapolis, Minnesota [46]. In addition to these findings, the results from our study suggest a potential fiber exposure to persons who perform work activities associated with contaminated trees surrounding these facilities. Surfaces other than trees, such as soil, building structures, etc., may be contaminated in these areas as well. Recommendations for future studies include determining the risk of exposure to persons performing work activities on trees in these areas, as well as determining if there is an elevated health risk to the general public when amphibole-contaminated trees are disturbed.

Table 4. Summary of the average dimensions of asbestos fibers measured from the bark samples in Libby, Spokane, Santa Ana, Newark, and Phoenix.

Site	Amphibole			Chrysotile		
	Avg. diameter (μm)	Avg. Length (μm)	Avg. aspect ratio (AR)	Avg. diameter (μm)	Avg. Length (μm)	Avg. aspect ratio (AR)
Libby, MT	0.39	3.4	11.5	N/A	N/A	N/A
Spokane, WA	0.37	3.6	9.42	N/A	N/A	N/A
Santa Ana, CA	0.49	5.6	11.2	0.10	1.7	16.0
Newark, CA	0.44	2.8	6.52	0.08	1.5	21.6
Phoenix, AZ	0.50	3.7	7.21	0.11	1.6	17.6

Note: Amphibole represents Libby amphibole and actinolite-tremolite fibers.

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