Boelter & Yates, Inc.
Environmental Engineers & Scientists

ULTRASONICATION OF 7M CHRYSOTILE

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BYI Project #1418A-4943

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ULTRASONICATION OF 7M CHRYSOTILE

SUMMARY

Objectives

The purpose of the study was to determine the effect of ultrasonication on 7M chrysotile when performed according to the procedures specified by ASTM Method D-5755[1].

Background

Air samples are to be prepared for analysis by the technique known as "*direct* preparation". The purpose of this study was to determine the effect of ultrasonication on 7M chrysotile when an "*indirect* preparation" method is used.

Testing Results

The ASTM "indirect preparation" method causes dispersal of clusters and bundles of asbestos into individual fibers. The effect is a significantly higher fiber count than had direct preparation techniques been used.

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1 D5755-95 Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentration, American Society for Testing and Materials, Section 5. Significance and Use, Paragraph 5.1

METHODOLOGY

Ultrasonication Method

Particle-free water was used for the study. The ultrasonication device was a 100W Fisher Scientific Tabletop Ultrasonicator. The ultrasonicator was calibrated prior to use and was operated at equilibrium temperature.

7M Chrysotile

A finished commercial grade 7M chrysotile was used in the study. The chrysotile is a Quebec, Canada mine product which was obtained in 1995 by the author from a U.S. manufacturer of roofing products. It is typical of the chrysotile raw ingredient used in manufactured products which would contain encapsulated asbestos.

Microscopy Techniques

Two different microscopy techniques were employed. Observation of the solution was performed under a stereo binocular trinocular scope or directly through the lens of the video camera. Observation of the filtered specimen and the fiber type identification was performed through a polarizing light trinocular microscope.

Recording Techniques

Video recording of the specimens and ultrasonication bath was performed with a Sony Digital Handycam DCR-TRV11 (SN 44385). Video recording of the magnified images was accomplished with trinocular mounted cameras transmitting their signals through a VCR.

Equipment List

A list of laboratory and recording equipment used in the study are shown below:

1. Mac-HEPA filtered fume hood (SN1126389)
2. Baxter Scientific Ameri-Sorb Fume Hood
3. Particle free water
4. Acetic acid
5. Various size (100ml to 1000ml) glassware, beakers, flasks.
6. Fisher Scientific Vacuum Filtration Assembly (CN 09-753-1C) with Stainless Steel Support, 1000ml flask, vacuum tubing and High Volume Pump
7. Fisher Scientific Tabletop Ultrasonicator (CN 001 15 335 30) 120V 50/60 Hz, 1.3 A, 100W
8. Various sample preparation instruments: micro-spatulas, scissors, scalpels, probes, forceps, brush: objective centering wrenches, 80A blue daylight filter, green (540nm) filter, and color compensating filters, P/A orientation slide, 1 mm (in 100 divisions) stage micrometer, 1x3 microscope slides and 18mm (#1.5) coverslips
9. 25 mm filter cassette with MCE, (0.45 to 0.8um pore size) millipore filters
10. MacVap acetone vaporizer; acetone, triacetin (reagent grade), nail polish
11. PLM - Olympus BHS Polarizing Microscope with Trinocular Head, rotatable analyzer with focusable Bertrand lens, Pol Condenser, rotatable bb stage, 4,10, 20, 40X objectives (all Olympus planachomats) 10X DS objective, 530nm compensator, 10X WHK ocular, 10X WK ocular with cross-lined graticule. MTV-3 adaptor with, 2.5X & 5X NFK photoeyepieces

with cross-lined graticule. 12V 100W quartz halogen illuminator.

12. PCM - Olympus BHS Polarizing Microscope with Trinocular Head, rotatable analyzer with focusable Bertrand lens, Abbe Condenser with 40X phase annulus, rotatable bb stage with mechanical stage 40X phase contrast objective (achomat) 10X WHK ocular, 10X WK ocular with Walton-Beckett graticule. MTV-3 adaptor with 2.5X, 3.3X and 5X photoeyepieces with Walton-Beckett graticules. Spare 12V 100W illuminator with quartz halogen filaments.
13. Olympus SZ 60 Stereo Microscope with Trinocular Head, SZ-PT photo adaptor, 1-6.3 zoom lense, GSWH 10X oculars, MTV-3 adaptor with 2.5X, 3.3X and 5X photoeyepieces.
14. Olympus SZH Stereo Microscope with Binocular Head, 1.5X Achromat Objectives, Bright Field/ Darkfield Base, GSWH 10X-H oculars.
15. Javelin CCD Color Chromachip III Camera (SN 34500177) wth 24V 410mA power source and cord
16. Javelin CCD Color SmartCam Camera (SN 371700) with 12V 500mA power source and cord
17. Sony CCD Color Hyper HAD (ser# 107404) with 13V 1,3A power supply and cord
18. Sony VHS Recorder Model SLV-696 HF HQ (SN RV 705OR)
19. Sony Digital Handycam DCR-TRV11 (SN 44385).
20. Dolan Jenner Fiberlite (fiber optic light source) Model 3100 (3797)
21. Olympus Highlight (fiber optic light source) 3000 (typical 8500N)
22. Fiber Optic Unlimited (fiber optic light source) (TA 300S)
23. 150 H Universal (fiber optic light source) (08131)
24. Fiber Optic Specialties (fiber optic light source)
25. Kaiser Pro Copy Stand with four 5100K light sources (quartz halogen filaments) filaments; with camera mounts

DISCUSSION OF FINDINGS

Study Location

The study was performed on October 19, 2001 in the laboratory of Mr. Peter Cooke of Microscopy Instruction, Consultation and Analysis (MICA) in Chicago, Illinois.[2] Also participating the study was Mr. Fred Boelter and Dr. Eric J. Chatfield of Chatfield Technical Consulting, Ltd. of Mississauga, Ontario, Canada.[3]

Study Sequence

The study sequence shows a series of orientation views to gain a sense of the scale. The opening view is of chrysolite ore against a $1 bill (Figure 1). The bill was selected since most everyone is familiar with its size and the images depicted on the obverse and reverse.

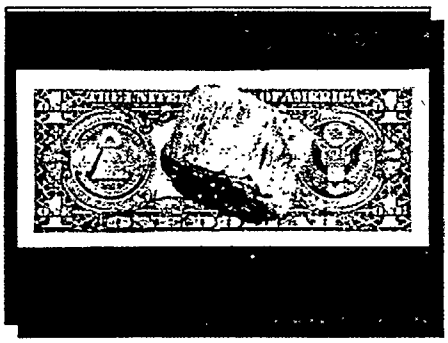

Figure 1 - Chrysotile ore and a $1 bill.

In Figure 2, the camera focuses on the eye above the pyramid in comparison to a millimeter scale. There are twenty-five millimeters (25.4mm) to the inch.

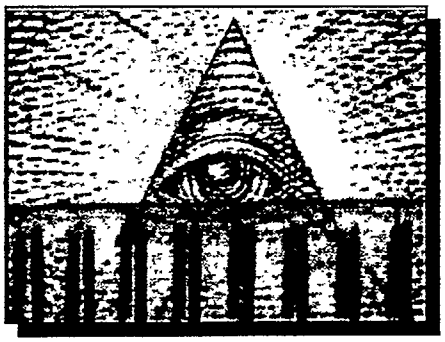

Figure 2 - Millimeter scale laid across the eye above th pyramid on a $1 bill.

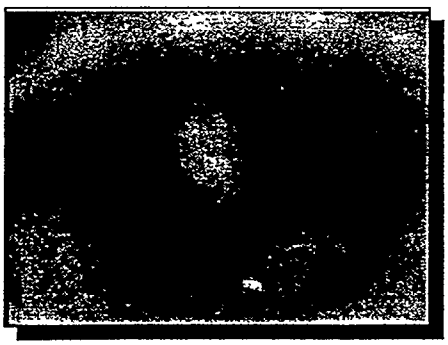

Figure 3 - A micron scale laid across the pupil of the eye on a $1 bill.

Since most of the discussion about fibers of health risk interest revolves around the scale of microns, the pyramids' eye is enlarged further in reference to a micron scale (Figure 3). The image shows the center portion of the pupil of the eye to be about 500 microns across. There are 1 million microns in a meter or 1,000 microns in a millimeter or 25,400 microns in an inch. Note that because of the depth of field, in Figure 3 the pupil is out of focus as the micron scale is in focus.

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2 Mr. Cooke's laboratory is equipped with light microscopy and integral microscope mounted cameras.

3 Dr. Chatfield participated in writing the referenced ASTM Method as well as authored numerous other documents on direct and indirect preparation techniques in a addition to documents on electron microscopy analysis.

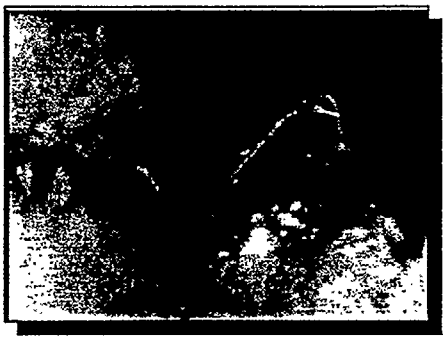

Figure 4 - 7M chrysolite as viewed under the stereo binocular scope.

During the study, commercial grade 7M chrysolite was used. Figure 4 shows the fluffy appearance of 7M chrysolite when enlarged to the same magnification as the pupil on a $1 bill. Refer to Figure 3 for comparison. Note the long fiber in the prominent loop of chrysotile has a diameter of approximately 10 microns.

A 250-ml glass beaker is placed in the ultrasonicator bath (Figure 5). The beaker contains 200ml of particulate free water adjusted to a pH of 3-4. The liquid level in the beaker and ultrasonic bath are identical. A small amount (2x4mm) of 7M chrysolite placed in the beaker.

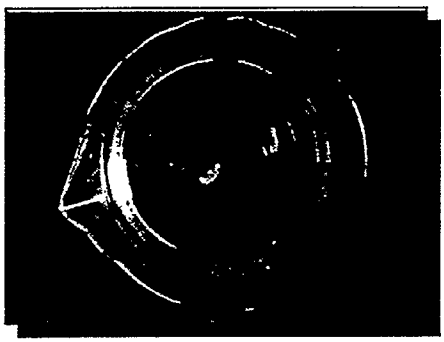

Figure 5 - 7M chrysolite in 3-4pH solution in a 250ml beaker in bath prior to ultrasonication.

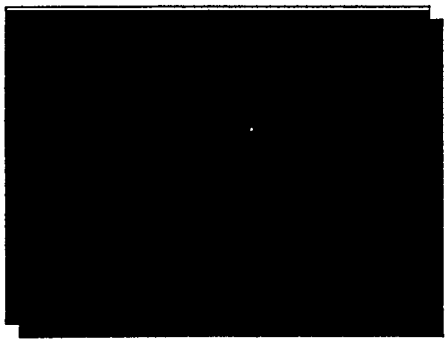

Figure 6 - Solution before ultrasonication.

Prior to starting the ultrasonicator, the beaker solution was viewed under the stereo binocular scope with fiber optic lighting (Figure 6). This viewing was performed at the same magnification as the eye with the millimeter scale shown in Figure 2. Occasionally, a fiber cluster would be visible as it drifted through the field of view but largely nothing was visible in the solution.

Also prior to ultrasonication, 5 milliliters of solution was filtered through a 0.45µ MCE filter and viewed under an Olympus BH phase contrast light microscope at 400x. There were no fibers present in any of the graticule fields similar to the one shown in Figure 7.

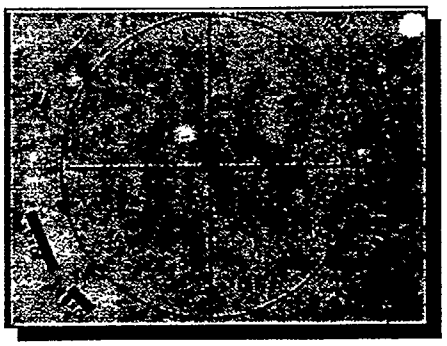

Figure 7 - Filtered solution before ultrasonication.

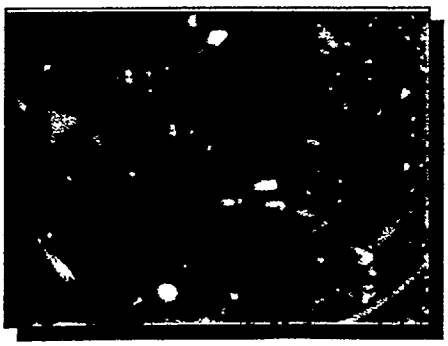

Figure 8 - Ultrasonicator being activated for a period of 3 minutes.

The ultrasonicator was then activated for a period of 3 minutes. During the time the 100W ultrasonicator was operating at its equilibrium temperature, the 7M chrysotile could be seen visibly dissociating (Figure 8).

types can be determine by the TEM method known as NIOSH 7402[7]. The post-abatement clearance method is specified in the USEPA AHERA Post-Abatement Clearance TEM Protocol[8]. All of these above methods are direct preparation techniques not involving the use of ultrasonication. Even the non-health risk base, ambient air quality TEM analysis technique for determining mass known as the Yamate Method recommends the use of direct preparation methods[9].

Ultrasonication is a technique employing acoustical energy which can be used to disperse particulates in solution. The effect which ultrasonication has in the indirect preparation of air samples has been extensively studied by others. It has been concluded that indirect preparation alters the nature of the asbestos fibers on the filter and the results generated by the use of indirect preparation do not represent either the concentration or the form of the asbestos fibers in air.

"The procedure outlined in this test method employs an indirect sample preparation technique. It is intended to disperse aggregate asbestos into fundamental fibrils. However, as with all indirect sample preparation techniques, the asbestos observed for quantification may not represent the physical form of the asbestos sampled... it may alter the physical form of the mineral fibers." [10]

"The AHERA, NIOSH 7402, and Yamate Methods require or recommend direct transfer methods for sample preparation. That is, the original air sampling filter is prepared for analysis and examined in the microscope with minimal disruption of the particles that have collected on the surface. However, indirect transfer may also break up asbestos bundles, clusters, and matrices into smaller units thereby giving a larger structure count." [11]

"When TEM specimens were prepared from the same filter using the indirect-transfer procedure D5755-95, it was found that the calculated concentration of chrysotile structures was much higher than the concentration reported from analysis of specimens prepared by ISO10312 (direct method). Considering chrysotile fibers and bundles only, the (indirect method) exceeded the (direct method) specimens by a factor between approximately 5 to 100." [12]

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7 Manual of Analytical Methods (NMAM) (1989), ASBESTOS by TEM, NIOSH Method 7402, *National Institute for Occupational Safety and Health*, Third Edition.

8 (October 30, 1987), Asbestos-Containing Materials in Schools - Asbestos, Transmission Electron Microscopy, *United States Environmental Protection Agency*, 40CFR Part 763, Appendix A, Section 1.

9 Methodology for the Measurement of Airborne Asbestos by Electron Microscopy, George Yamate, Satish Agarwal, and Robert Gibbons, Draft Report prepared under USEPA Contract No. 68-02-3266, July 1984

10 D5755-95 Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentration, American Society for Testing and Materials, Section 5. Significance and Use, Paragraph 1.4.1

11 A Guide to Monitoring Airborne Asbestos in Buildings, Dale L. Keyes and Jean Chesson, 1989, Library of Congress Catalog Card Number 89-80764, Page 50.

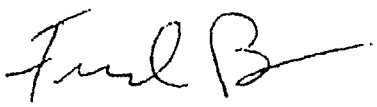
12 Advances in Environmental Measurement Methods for Asbestos, American Society for Testing and Materials, Michael E. Beard and Harry L. Rook, editors, ASTM Stock Number STP 1342, 1999, Chapter entitled Correlated Measurements of Airborne Asbestos-Containing Particles and Surface Dust, E.J. Chatfield, Page 400.

CONCLUSIONS

A 100 Watt ultrasonicator when used in the "indirect preparation" of asbestos samples dissociates clusters and bundles of asbestos. The consequence of this dissociation when applied to air samples leads to a change in the physical form of the asbestos air sample and leads to higher fiber counts. The result would be an elevated fiber count and an erroneous conclusion about the fiber release potential of a product or the significance of the exposure which an individual may receive.

Respectfully submitted,

BOELTER & YATES, INC.



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