Final Report

In Vitro Mordenite Fiber Dissolution at Acidic pH

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Abstract

Mordenite is an aluminosilicate zeolite mineral similar to asbestos. The unique structural and chemical properties make mordenite favorable to a wide variety of commercial applications ranging from waste-effluent treatment to paper production. Interest in mordenite, as an occupational inhalation hazard, arose when it was discovered that the fibrous form of the mineral exists in the subsurface of Yucca Mountain, NV, the site of a federally proposed nuclear waste repository. During preliminary geologic investigations at Yucca Mountain, workers performing dry-drilling operations were potentially being exposed to aerosols of mordenite. In addition, environmental exposures to zeolites (including mordenite) were brought to the world's attention when a reported outbreak of mesothelioma was documented in the Cappadocia region of Turkey.

While the precise mechanism for the production of an adverse biological response is yet unknown, increased scrutiny is being placed on a fiber's dimensional characteristics and biopersistence (durability) and the role they play in the induction of pulmonary disease. Although the morphology of aerosolized mordenite has been reported by at least one researcher, what has yet to be investigated is the biopersistence of mordenite fibers upon deposition in the pulmonary environment. Research studies have shown that a fiber's *in-vivo* biopersistence is related to the degree of its *in-vitro* dissolution. Methods for measuring *in-vitro* fiber dissolution for durable fibers have been developed using acidic solvents, representing the environment within alveolar macrophages (pH of 4.5-5.0).

This research acquired and aerosolized mordenite minerals deposited in different regions of the United States. Each mordenite mineral was analyzed for it major mineral and chemical constituents. Fiber samples from each specimen were collected and evaluated for their respective morphologic characteristics and subjected to an acidic simulated lung fluid assay in an effort to gain a better understanding of their pulmonary biopersistence.

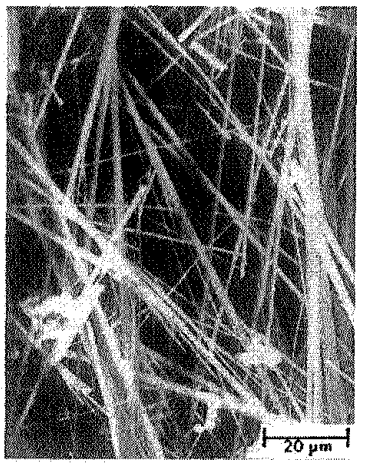
Results of the aerosolization phase of this research suggest that mechanical aerosolization of a fibrous mordenite minerals can cause an environmental release of fibers having small diameters (<1 μm) and large aspect ratios. Performance of the dissolution phase of this study identified an optical method to evaluate fiber dissolution and suggests that mordenite fibers are insoluble at acidic pH.

It is anticipated that duplication of methods performed in this study will facilitate the estimation of fiber biopersistence at both intra-cellular and extra-cellular pH. In addition, the study results will also provide epidemiologists conducting health hazard studies on fibers a mechanistic basis for the bioavailability of mordenite fibers in the lung.

Highlights/Significant Findings

Mechanical Aerosolization of Mordenite Mineral and Collection of Fibrous Material

Significant to this study are the visual features found on photomicrographs taken of the aerosolized mordenite specimens. Inspection of these photomicrographs revealed the presence of many long, acicular fibers (see photomicrograph below):



As can be seen using the scale in the photomicrograph, the fibers have small diameters, yet large aspect. A number of the fibers are smaller in diameter than can be resolved by light microscopy. Thus, electron microscopy is a more robust method for examination of the fibers.

Development of a Method to Evaluate Fiber Solubility at Acidic pH

Traditionally, dissolution studies have relied on a combination of direct mass-loss data and analysis of a dissolution system's effluent solution to determine fiber dissolution rate. However, as fiber insolubility increases, the ion concentrations in the effluent solution often fall below detectable levels. However, the performance of this research has identified a method that can be used to directly measure a fiber's diameter using an environmental scanning electron microscope (ESEM).

Translation of Findings

The performance of this study identified a method to characterize the morphology and dissolution of mineral fibers using laboratory-scale aerosolization and environmental scanning electron microscopy. It is anticipated that duplication of this method using zeolites and other fibrous minerals will facilitate the estimation of the dissolution rate at both intra-cellular and extra-cellular pH. The results of this study provide a method to reveal the ability of deposited mordenite fibers to persist in the solution of the pulmonary region of the lungs at both intra- and extra-cellular pH. The results obtained from the use of this method will also provide epidemiologists conducting health hazard studies on fibers a mechanistic basis for the bioavailability of mordenite fibers in the lung.

Outcomes/Relevance/Impact

This research provided meaningful scientific information to assist in developing methodology to more accurately identify the role biopersistence plays in a fiber's ability to induce a carcinogenic response.

Scientific Report

Background

Zeolites are aluminosilicate minerals similar to asbestos having unique structural and chemical properties that make them favorable to a wide variety of commercial applications ranging from waste-effluent treatment to paper production (Mumpton). Interest in zeolites, as an occupational inhalation hazard, arose when it was discovered that the fibrous form of these minerals exists in the subsurface of Yucca Mountain, NV, the site of a federally proposed nuclear waste repository. During preliminary geologic investigations at Yucca Mountain, workers performing dry-drilling operations were potentially being exposed to zeolite aerosols.

Documented cases of human exposure to zeolite minerals have been reported in literature. An exposure to environmental zeolites (including mordenite) was brought to the world's attention when Baris et al. reported an outbreak of mesothelioma in the Cappadocia region of Turkey (Baris et al.). In another study, Casey et al. reported treating a patient with extensive parenchymal and pleural fibrosis who resided and worked in an area rich in zeolite deposits (Casey et al.). Suzuki injected Swiss Albino male mice with fibrous erionite and a mixture of fibrous and granular mordenite (Suzuki). In this study, malignant peritoneal tumors were produced in 6 out of 10 mice treated with erionite and a fibrotic effect was seen in the mordenite-treated mice. The precise mechanism for the production of an adverse biological response is yet unknown, but increased scrutiny is being placed on a fiber's dimensional characteristics and biopersistence (durability) and the role they play in the induction of pulmonary disease.

Stanton et al. have formulated a hypothesis stating that the carcinogenicity of all fibers depends upon dimension and durability rather than physicochemical properties (Stanton, Layard, and Tegeris et al.). Stanton and Layard devised size categories which correlate specific fiber dimensions with a higher probability of pleural mesothelioma in rats (Stanton and Layard). The morphology of mordenite aerosols has been investigated by Stephenson, et al. where they reported large percentages of aerosolized mordenite fibers achieving size dimensions consistent with Stanton and Layards' high risk categories for mesothelioma induction (Stephenson et al.). This evidence suggests that, based on fiber morphology, mordenite is a potential inhalation health hazard. What has yet to be investigated is the biopersistence of mordenite fibers upon deposition in the pulmonary environment.

There is intense interest within the scientific community today in the pulmonary biopersistence of fibers and the role that dissolution (the rate at which fiber mass is removed per unit surface area of the fiber (measured in nm/day)] plays in lung clearance. This interest is, in part, related to a fiber's enhanced ability to initiate a pathogenic response if it exhibits a sufficient biopersistence in distal airways such that it will not degrade to the extent where it can be adequately cleared from susceptible lung tissues. Many questions, like those offered below, remain unanswered.

- $ Are sufficiently short fibers, those completely engulfed by a macrophage, effectively phagocytized and cleared if there is minimal fiber dissolution in their acidic environment?
- $ Do some fibers undergo differential dissolution allowing for longer fibers to be split in half, enhancing their ability to clear, or do they cleave length wise, effectively doubling their number?

$ Does pulmonary dissolution alter the physical dimensions of fibers to the extent that their risk of inducing mesothelial carcinogenicity is reduced per the Stanton Hypothesis?

Thus, further scientific investigation of pulmonary biopersistence of fibrous material through dissolution measurement is needed to narrow this current gap in knowledge.

Recent research has suggested that a fiber's *in-vivo* biopersistence is related to the degree of its *in-vitro* dissolution.[8] Prior *in-vitro* studies have had success in measuring fiber dissolution by flowing simulated lung fluid (SLF) across a mat of contained fibers to identify changes in the fiber's chemical and physical characteristics (Bernstein et al.) (Christensen et al.).

No comprehensive studies are apparent in literature measuring fiber dissolution using a wide variety of fibrous zeolite minerals. Most fiber dissolution research has been performed using man-made vitreous fibers (MMVF) and crocidolite asbestos (a natural mineral fiber similar to zeolites) (Zoitos et al.) (De Meringo et al.) (Guildberg et al.). Results of these *in-vitro* studies have shown that the pulmonary dissolution behavior of crocidolite asbestos is best described using an acidic SLF (pH 4.5-5.0), representing the environment within an alveolar macrophage (De Meringo et al.) (Guildberg et al.) (Christensen, Eastes et al.)

Of notable importance to the potential health implications of exposure to fibrous zeolite aerosols, is that *in-vitro* research results indicate that the fibers of crocidolite asbestos are extremely durable, having almost no dissolution in neutral SLF and only minimal dissolution in acidic SLF. Due to their physical and chemical similarities to the asbestos mineral, this finding suggests a similar outcome for zeolite fibers. The research hypothesis of concern in this project is whether there exists any difference in the chemical or physical characteristics of zeolite fibers before and after exposure to an *in-vitro* dissolution assay at an acidic pH. The dissolution model that needs to be tested is similar in nature to the dissolution model described in a previous published study measuring the dissolution of crocidolite asbestos using an acidic SLF (Christensen, Eastes et al.) The end result needed is the calculation of a fiber's dissolution rate and extrapolation to that result to the pulmonary biopersistence of similar fibers deposited *in-vivo*.

Specific Aims

The pulmonary biopersistence (durability) of mordenite fibers plays a crucial role in mesothelial carcinogenicity and other lung diseases. The degree of biopersistence for this fiber type is poorly understood, but is enhanced by investigation of *in vitro* fiber dissolution rates in solutions mimicking pulmonary fluids. The long term goal of this research is the determination of risk associated with human exposures to fibrous mordenite aerosols and other similar airborne contaminants based on an understanding of the fiber's biopersistence in the human lung.

The pathogenic response of a fiber is in part dependent on a sufficient biopersistence in distal airways. This suggests that more durable fibers may not degrade to the extent where they are adequately cleared from susceptible lung tissues. Thus, an evaluation of biopersistence plays an important role in determining the risk of producing an adverse health effect resulting from pulmonary fiber deposition. It has been experimentally shown that a fiber's *in-vivo* biopersistence is related to the degree of its *in-vitro* dissolution. Methods for measuring *in-vitro* fiber dissolution for durable fibers have been developed using an acidic simulated lung fluid,

representing the environment within pulmonary alveolar macrophages (pH of 4.5-5.0). This research will test the following hypothesis:

- There exists no difference in chemical or physical mordenite fiber characteristics before and after an *in-vitro* dissolution assay at an acidic pH.

To test this hypothesis, experiments with the following specific aims were needed:

1. To aerosolize and collect test fibers for inclusion into an *in-vitro* dissolution assay;
2. To characterize test fiber samples with respect to the following:
   - Mineral composition using Reference Intensity Method of Chung and quantitative analysis by the Rietveld Method, and;
   - Chemical composition using quantitative x-ray diffraction analysis.
3. To provide a method to estimate dissolution rates of test fibers based on observed changes in fiber size in an acidic *in-vitro* dissolution assay.

Methodology

Acquisition of Mordenite Mineral Specimens

For the performance of this study three mordenite mineral specimens were logged and their visual texture documented. The log number of each specimen and a brief description of their visual texture are provided in Table 1.

Table 1. Mordenite Mineral and Control Specimens

ID #	Deposition Location	Where Obtained	Color and Visual Texture
1M	Bucoda, WA.	Ward's Natural Science Establishment	Macroscopic analysis of fractured mineral reveals the presence of white, cobwebbed fibrous material
2M	Challis, Idaho	USGS, Denver CO.	Macroscopic analysis of fractured mineral reveals the presence of white, cobwebbed fibrous material
3M	Yucca Mountain, NV.	Los Alamos National Laboratory, NM	No macroscopic evidence of fibrous material present on fractured mineral surface

Aerosolization and Nucleopore Collection of Mordenite Mineral Specimens

Each mordenite mineral specimen was aerosolized by mechanical dispersion of the fibrous material using a Dremel, model 270, high RPM, moto-tool drill and collected on Nucleopore Corporation, 0.2 μm, 25 mm nucleopore filters in the aerosolization chamber shown in Figure 1.

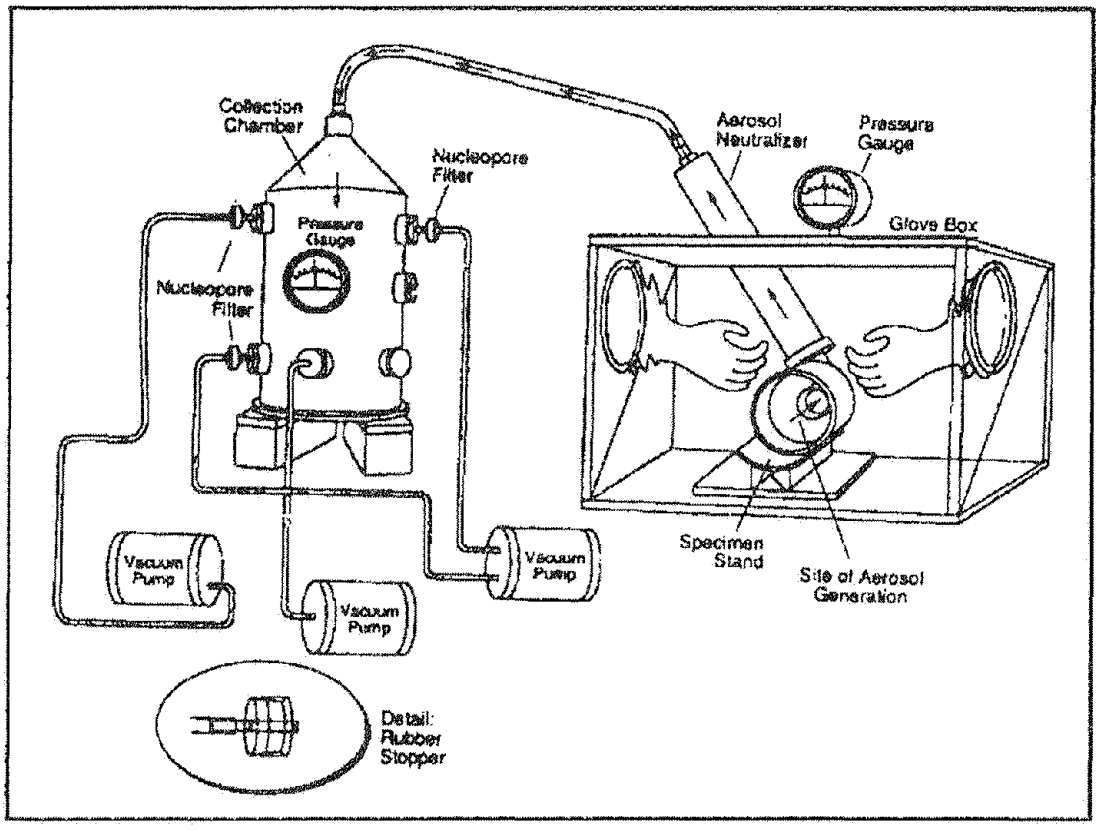


Figure 1. Aerosolization Chamber

The aerosolization process for the mordenite minerals involved the following steps a) The operator's hands were inserted into the gloves connected to the glove box, b) the moto-tool drill was grasped in the operator's right hand while the mineral to be aerosolized was held firmly in his left hand, c) the power switch for the drill, located on the side of the drill, was then moved to the "on" position, d) simultaneous with the activation of the moto-tool drill, three vacuum pumps, supplying negative pressure to the system, were activated, and e) the tip of the drill bit was applied directly to the mineral. This process generated an aerosol, which moved out of the generation chamber, up through a krypton-85 aerosol neutralizer, and down into the collection chamber.

The movement of the aerosol was initiated by the presence of increasing negative pressure from the site of generation to the collection site. Negative pressure was created by three vacuum pumps located downstream of the collection chamber. Makeup air was provided through the drilling of three holes (2 cm in diameter) in one side of the glove box.

Four aerosol collection devices were used during each run, two cascade impactors and two nucleopore filter samplers. A total of three vacuum pumps pressurized the aerosol generation system. Two vacuum pumps pressurized all impaction and filter samplers. An additional vacuum pump provided appropriate aerosol mixing within the collection chamber. Vacuum pumps and collection devices are described below.

A vacuum pump, equipped with two needle valves, was connected, by plastic tubing, to two Anderson 1 ACFM Ambient Particle Sizing Samplers. The impactor samplers allowed for the determination of aerosol mass median aerodynamic diameter. The impaction samplers were attached to the collection chamber using brass tubing. The brass tubing was inserted into holes bored into rubber stoppers, which were fitted into sampling ports located on the collection chamber (Figure 1.). The collection filters used in the Anderson samplers were Gelman, 76mm polypropylene filters.

A vacuum pump, equipped with two toggle switches, was connected, by tygon tubing, to the nucleopore filter samplers. The samplers were attached to the collection chamber in the same method as described for the impactor samplers. The filters were housed in Gelman, 25 mm Delrin in-line filter holders. The type of filters used was Nucleopore Corporation, 0.2 μm, 25 mm nucleopore filters.

An additional vacuum pump was connected, via plastic tubing, to the collection chamber. It was located near the bottom of the chamber. Its purpose was to provide appropriate aerosol mixing throughout the length of the chamber.

The four collection devices were located strategically, two high and two low, on the collection chamber, to monitor any fluctuations in aerosol distribution. Near equal aerosol concentrations on all four devices ensured a uniform aerosol distribution within the chamber.

Magnehelic pressure monitoring devices were located on the aerosol generation and collection chambers. Constant observation of these devices let the operator know if sufficient negative pressure was being generated throughout the aerosol generation system. Also, it was mandatory that the pressure inside the generation chamber, during an aerosol run, was negative, relative to the ambient air. Positive pressure at this site would mean that loss of aerosol into room atmosphere was possible.

In order to ensure a tight system, with no pressure losses, all fittings between the glove box, aerosol neutralizer, brass tubing, and collection chamber were sealed with Dow Corning 732, multi-purpose silicon sealant. The same silicon sealant was also used to seal all plexiglass contacts and potential leak sites on the glove box. Between each run the aerosol generation system and impactor samplers were cleaned using Alconox chemical and industrial detergent. All cleaned items were set aside to air dry before reuse. All impactor stages were analyzed between runs using a Wild Heerbrug M8 stereo light microscope. Impactor stage analysis was performed to ensure that no significant interstage fiber losses were occurring.

Mineral Composition Analysis

Based on the results of the morphologic analysis described above, specimens 1M (Bucoda, Washington) and 2M (Challis, Idaho) were submitted to the Earth and Environmental Sciences Division at Los Alamos National Laboratory for x-ray diffraction (XRD) analysis of the specimen's major mineral phases. Specimen 3M (Yucca Mountain) was not submitted for analysis as no fibers from this sample were acquired during aerosolization.

Chemical Composition Analysis

Similar to the mineral composition analysis discussed above, Specimens 1M (Bucoda, Washington) and 2M (Challis, Idaho) were submitted to the Earth and Environmental Sciences Division at Los Alamos National Laboratory where spectrographic analysis of was performed to

ascertain each specimen's major elemental constituents. Specimen 3M (Yucca Mountain) was not submitted for analysis as no fibers from this sample were acquired during aerosolization.

Dissolution Solvent

The acidic simulated lung fluid solution used in this study was a modified Kanapilly solution using phthalate as a buffer. The composition (mmol/L) of the solution is as follows:

Sodium	116.8
Potassium	24.0
Calcium	0.2
Chloride	114.2
Phosphate	1.0
Sulfate	0.5
Glycine	6.0

The following reagents (g/5L) were used to prepare the simulated lung fluid solution:

Sodium phosphate dibasic anhydrous	0.70
Sodium chloride	33.25
Sodium sulfate anhydrous	0.36
Calcium chloride dihydrate	0.15
Glycine	2.25
Potassium hydrogen phthalate	20.42

Preparation of the acidic simulated lung fluid contained the following steps:

(Step 1) Calibration of a laboratory-scale microbalance;

(Step 2) Addition of 4.75 L deionized and distilled water to a 5 L carboy;

(Step 3) Recording of the room temperature;

(Step 4) Weighing out of all of the fluid reagents listed (except the potassium hydrogen phthalate) onto separate tared weighing dishes;

(Step 5) Addition of each reagent (individually);

(Step 6) Placement of a stir bar into the carboy and a vigorously stir on a magnetic stir plate (allow solution to stir for 12 hours)

(Step 7) Once the solution has stirred, weighing out of the potassium hydrogen phthalate and adding it to the solution;

(Step 8) Placement of a stir bar into the carboy and a slowly stir on a magnetic stir plate (allow solution to stir for 1 hour);

(Step 9) Calibration of a pH meter;

(Step 10) Warming of the solution to 37±1°C using a hotplate;

(Step 12) Addition of 0.1 M potassium hydroxide (KOH) until a pH of 4.50 is reached;

(Step 13) Documentation of the total volume of KOH added to confirm the final potassium ion concentration;

(Step 14) Documentation of the final pH and temperature.

Mordenite Fiber Dissolution

A typical dissolution assay can be performed by circulating simulated lung fluid through the fiber-containing cassette with the outlet-side up. The placement of the cassette in this manner reduced the likelihood of an air bubble forming between the planchette and the inlet. The cassette can be drained by clamping it to a ring stand and placing the outlet-side down. The peristaltic pump can be left at the preset flow rate during draining until the cassette was empty of fluid. After draining, the planchette can be removed and placed in a vacuum dessicator to dry.

By comparing the elemental composition of the physiologically relevant solvent (see above) to the estimated concentration of mordenite elements in the solvent as a function of dissolved mordenite mass, it is seen that a traditional dissolution technique involving analysis of dissolved material will not provide useful information about the dissolution rate of mordenite. Therefore, the alternate electron microscopic examination method described in the following section was developed.

Table 2 Estimation of the Solvent Concentration of Elements as a function of the Mass of Dissolved Mordenite (assumed dissolved mass = 1 mg)

molecular formula: $Na_3\ K\ Ca_2\ (Al_8\ Si_{40}\ O_{96}) * 28(H_2O)$

Element	Atomic Mass (amu)	Atoms per Molecule	Mass of Element per Molecule (amu)	Mass Fraction	Mass Dissolved (mg)	Concentration in Effluent (mg/mL)	(ug/mL)
Ca	40.078	2	80.156	0.020	0.020	0.00002	0.020
Na	22.98977	3	68.96931	0.017	0.017	0.00002	0.017
K	39.0983	1	39.0983	0.010	0.010	0.00001	0.010
Al	26.891538	8	215.132304	0.054	0.054	0.00005	0.054
Si	28.0855	40	1123.42	0.282	0.282	0.00028	0.282
O	15.9994	96	1535.9424	0.385	0.385	0.00039	0.385
H	1.00794	28	28.22232	0.007	0.007	0.00001	0.007
O	15.9994	56	895.9664	0.225	0.225	0.00022	0.225
		Total mass:	3986.907034		1	.001	1

INPUT Values for the Calculation:		
	Initial Sample Mass (mg)	100
	Fraction Dissolved	0.01
	Volume of Solvent (mL)	1000
	Total Mass Dissolved (mg)	1

Dissolution Analysis

To provide a method to estimate a mordenite fiber's potential for dissolution rate, a carbon planchette was utilized to serve as a holder for mordenite fibers during exposure to circulation flow of simulated lung fluid and diameter analysis by scanning electron microscopy. To allow for simulated lung fluid flow through a planchette, eight holes were drilled into each carbon planchette using a drill press and a 3/16" bit. A utility knife was then used to cut grooves in the planchette surface to connect the holes of the planchette. Each hole had two grooves that connected it to two other holes (except for the first and last hole which were connected to only one other hole). Locating the holes in this manner facilitates improved scrutiny of the fibers when the planchette is observed under the electron microscope. In addition, a utility knife was used to cut a single groove on the side of the planchette, to assist in standardizing the orientation of the planchette during loading into the scanning electron microscope.

Mounting of the mordenite fibers onto carbon planchettes was accomplished using Hardman Urethane D-85 adhesive. A thin layer of the adhesive was applied to the planchette using a utility knife blade. The holes in the planchette, if covered by adhesive, were cleared using the end of a paper clip. The adhesive was allowed to dry to a tacky stage before application of fibers. The fibers were applied by placing the planchette, adhesive side down, onto the nucleopore filter containing the fibers. The filter was then slowly peeled off, leaving the fibers

adhered to the planchette. The adhesive was allowed to dry and harden in a vacuum dessicator before further use. After the adhesive hardened, a utility knife was used to retrace the earlier cut grooves.

Estimation of pre-dissolution fiber diameter was performed by observation of the mordenite fibers affixed to the planchette under an environmental scanning electron microscope (ESEM). The pre-cut holes were scrutinized for fibers that hung over the edge of a given hole. The fiber's position, diameter, and surroundings were noted.

In summary, estimation of a mordenite fiber's dissolution rate was performed using the optical method outlined below:

(Step 1) A test fiber was mounted onto a carbon scanning electron microscope (SEM) planchette using urethane adhesive. Holes were drilled holes in the SEM planchette to permit circulation of simulated lung fluid.

(Step 2) Test fibers were identified using an environmental SEM and fiber diameters were measured at a set distance away from a drilled planchette hole.

(Step 3) The fiber-containing planchette was mounted in a cassette holder and then placed in a circulation bath containing simulated lung fluid.

(Step 4) The circulation bath was monitored every two hours for maintenance of a consistent pH (4.5±0.1) and temperature (37±1°C).

(Step 5) After 5 days the fiber-containing planchette was removed Step 2 was repeated for determination of fiber dissolution rate.

Fiber dissolution was estimated by exposing mordenite fibers to a circulation bath containing acidic simulated lung fluid over a period of 14 days. The fiber dissolution system consisted of a carboy containing simulated lung fluid connected to a multiple-line peristaltic pump, an inline pH meter, an assay test sample cassette, and an effluent collection bottle. The assay test sample cassette consisted of a three-piece 25mm nucleopore cassette, an O-ring, the sample planchette, and an outer apparatus of washers, screws, and nuts that secured the cassette together. A water bath was used to bring the simulated lung fluid's temperature up to 37±1°C and to maintain the assay test sample cassettes at 37±1°C. A peristaltic pump was used to provide a circulation flow rate is arbitrarily set at 0.05 mL/min. The effluent solution was monitored each day for deviation from the original pH. If the deviation was more than 0.2 pH, the flow rate was increased until the pH was within the prescribed limits of 4.5-5.0. Caution was taken to prevent high flow rates (>1.00 mL/min) due to previous tests, which have shown that erosion of the planchette holes can occur at such flow rates causing artifacts in test results.

Results and Discussion

Morphologic Aspects of Aerosolized Mordenite Fibers

Photomicrographs of specimens 2M and 3M were taken to verify the presence of mordenite mineral fibers on filter media and are shown in Figures 2 and 3 on the following page. Visual Inspection of the photomicrographs reveals that the fibers on the specimen 3M (Yucca Mountain) appear to have smaller lengths and diameters and are less acicular in nature than specimens 1M and 2M. In contrast to mineral specimen 3M, specimens 1M and 2M appear to be rigid and acicular in nature and many have large aspect ratios. Table 2 provided on page 5 below, shows the fiber morphologic characteristics of mineral specimens 1M and 2M. Fiber length, width, and aspect ratio information for mordenite mineral specimen 3M (Yucca Mountain specimen) were not documented due to the inability to measure a statistically significant number of fibers.

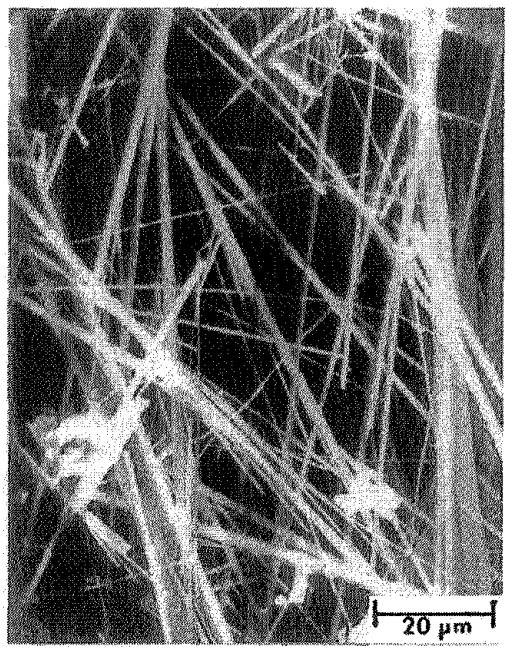


Figure 2 SEM Photomicrograph of Challis, Idaho Mordenite Specimen (Specimen 1M revealed morphologically similar fibers)

Figure 3 SEM Photomicrograph of Yucca Mountain, NV Mordenite Specimen

Table 3. Median and Interquartile Range for Mineral Specimens 2M and 3M

n = 1000 fibers	Mineral Specimen	
	1M – Bucoda, WA	2M – Challis, Idaho
FIBER DIAMETER		
CMD	0.7 µm	0.5 µm
75th Percentile	1.0 µm	0.7 µm
25th percentile	0.5 µm	0.4 µm
Interquartile Range	0.5 µm	0.3 µm
FIBER LENGTH		
CMD	6.7 µm	4.8 µm

75th Percentile	12.5 μm	8.9 μm
25th Percentile	4.0 μm	3.0 μm
Interquartile Range	8.5 μm	5.9 μm
FIBER ASPECT RATIO		
CMD	8.3 μm	8.7 μm
75th Percentile	16.6 μm	15.2 μm
25th Percentile	5.0 μm	5.7 μm
Interquartile Range	11.6 μm	9.5 μm

The results shown in Table 2 suggest that the mordenite minerals used in this study produce respirable fibers upon mechanical degradation. This is significant to human health when exposure occurs during similar conditions. (i.e. mining, vehicle disturbance of unpaved roads, etc.). Also, the fibrous nature of the Yucca Mountain specimen raises concern over human exposure to mordenite mineral aerosols during future activities at the Yucca Mountain nuclear repository site.

Mineral Composition

Table 2 summarizes the quantitative x-ray diffraction (XRD) results for two of the mordenite samples used in this study.

Table 4. Quantitative XRD Analysis

Sample 1M (Bucoda, Washington)		
Mineral	Chung Method (weight %)	Rietveld Method (weight %)
Mordenite	90 ± 16	90.2
Quartz	6 ± 1	9.2
Cristobalite	1 ± 1	0.4
Total	97 ± 16	99.8
Sample 2M (Challis, Idaho)		
Mordenite	75 ± 14	85.5
Quartz	3 ± 1	5.5
Calcite	2 ± 1	3.4
Smectite	2 ± 1	***
Total	82 ± 14	94.4

Chemical Composition

Figures 2 and 3 summarize the chemical composition results for two of the mordenite samples used in this study.

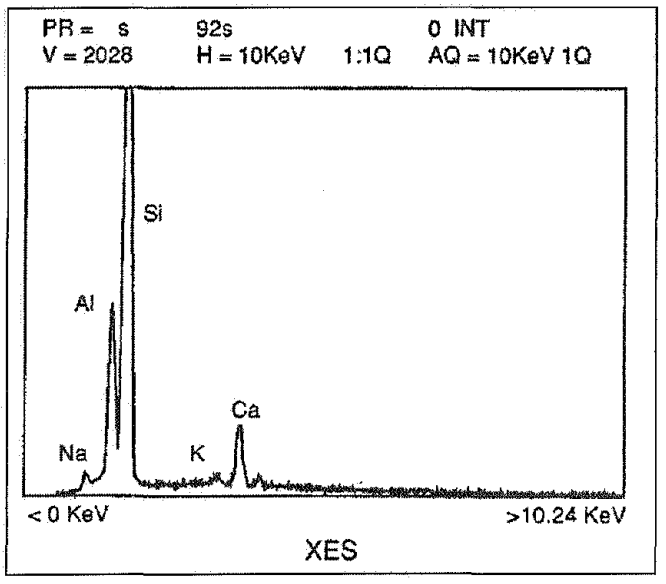


Figure 4 Chemical Composition for Specimen 1M

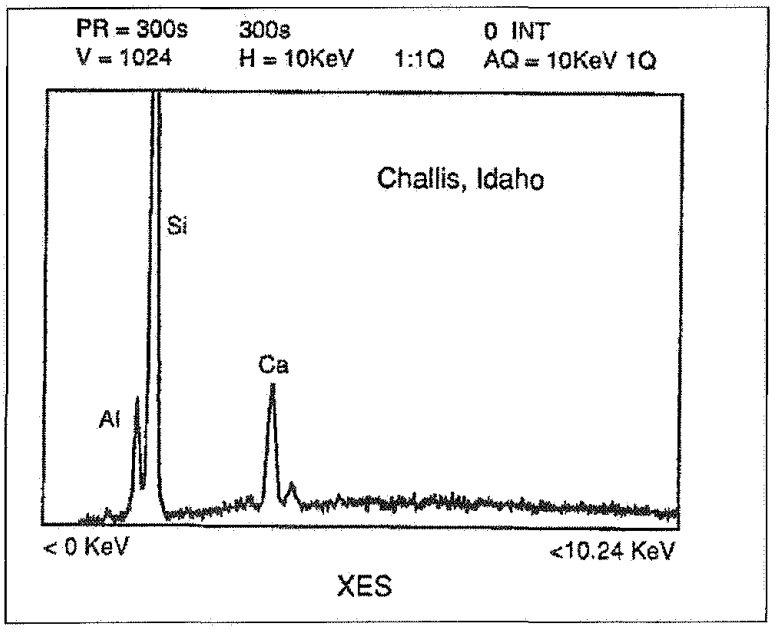


Figure 5 Chemical Composition for Specimen 2M

Aerosol cyclone separation of mordenite fibers

Figures 6, 7, and 8 show scanning electron photomicrographs of fiber samples collected in stages 1, 2, and 3 of the aerosol cyclone. Because the aerodynamic diameter of a fiber is directly proportional to diameter, but only weakly a function of length, the cyclone can be used to separate fibers by diameter for study. This can allow selection of stage materials with a preferential concentration of fiber diameters for mounting onto the ESEM planchettes.

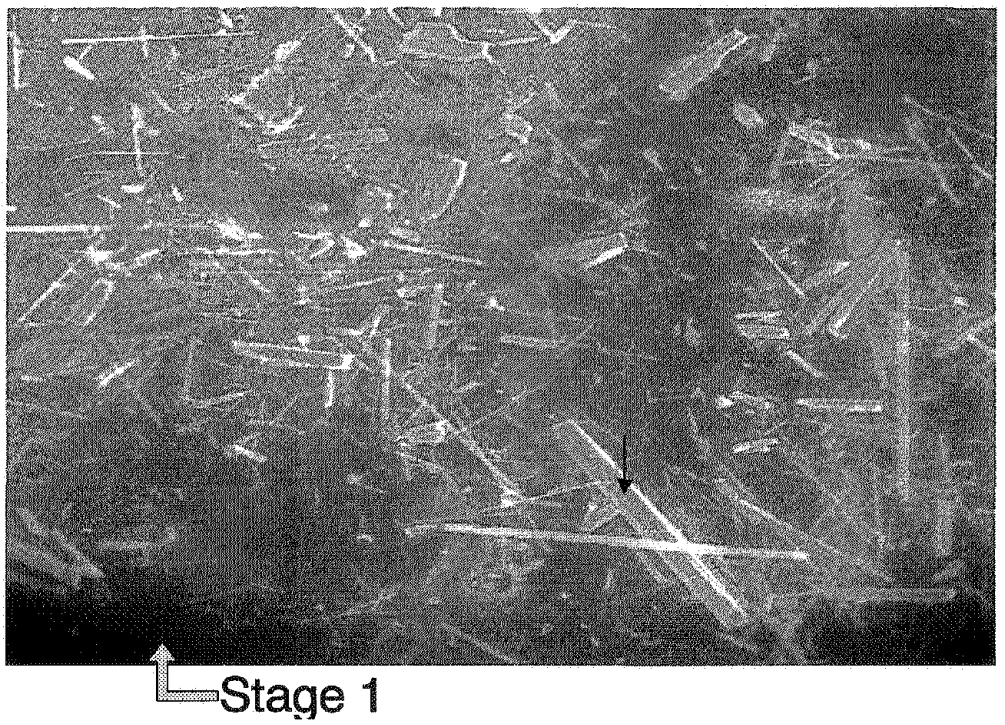


Figure 6 Scanning electron photomicrograph from stage one of the aerosol cyclone. The arrow points to a fiber of length 125 μm

Figure 7 Scanning electron photomicrograph from stage one of the aerosol cyclone. The arrow points to a fiber of length 41 μm

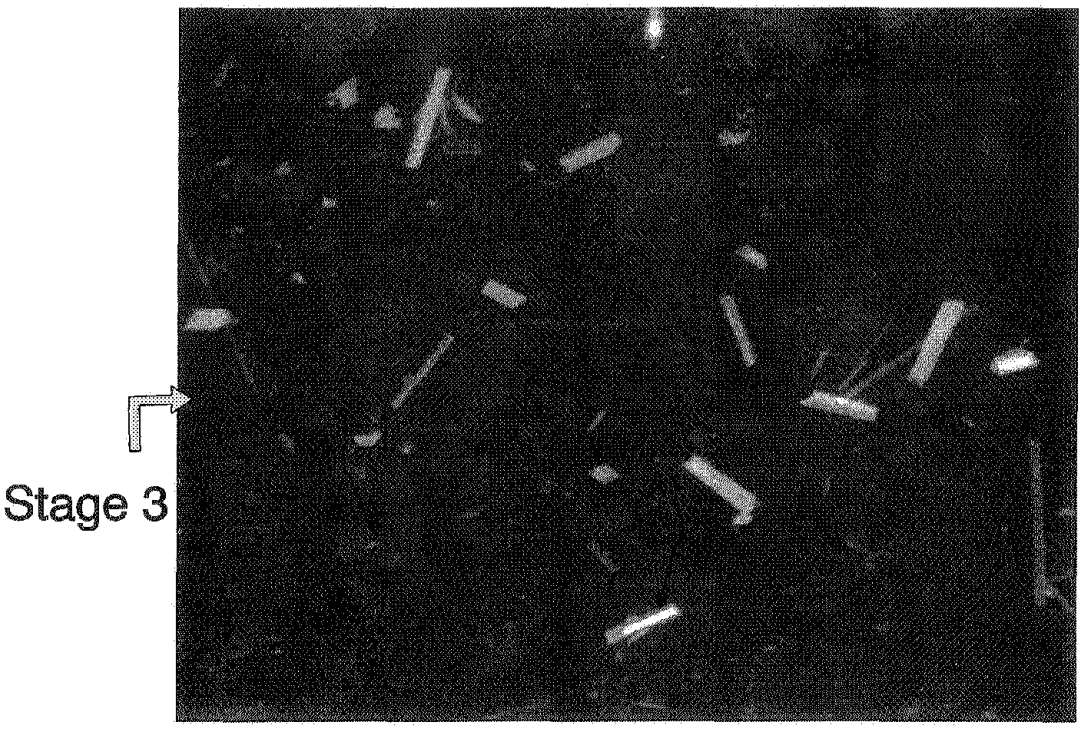


Figure 8 Scanning electron photomicrograph from stage one of the aerosol cyclone. The arrow points to a fiber of length 9 μm

Observation of mordenite fibers as a function of time in an environmental scanning electron micrograph

Figure 9 shows a representative array of fibers mounted on the carbon planchettes. The use of a scoring method to mark the planchettes allows the fiber locations to be recorded for repeated observation.

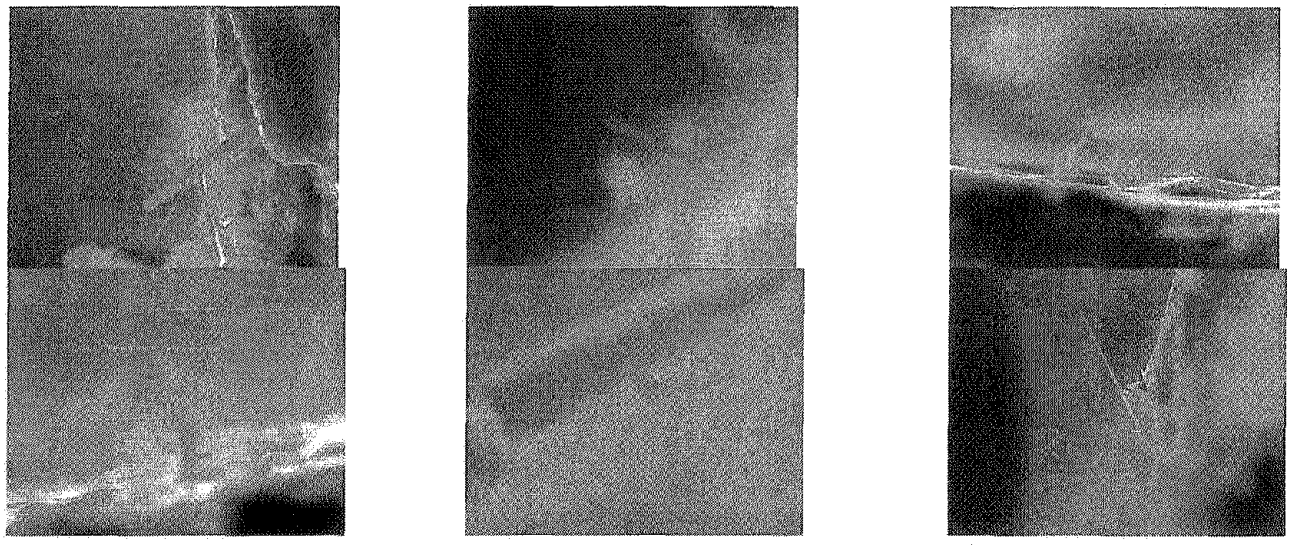


Figure 9 Representative fibers mounted on planchettes and observed with the environmental scanning electron microscope demonstrating that fibers will diameters in the submicrometer range can be successfully mounted and imaged.

Conclusion

The National Institute of Occupational Safety and Health (NIOSH) stated in its description of the purpose of this research grant program that studies are supported to develop methods for measuring exposures to hazards and detecting adverse health effects. The performance of this research will serve that purpose. Further, through its National Occupational Research Agenda (NORA), NIOSH identified research priority areas of cancer research and exposure assessment methodology. The new method for mounting and observing fibers for dissolution studies will assist in more accurately determining the potential biopersistence of mordenite fibers and in further elucidating the role biopersistence plays in a fiber's ability to induce a carcinogenic response. It will also allow health professionals to make a more appropriate exposure assessment of the hazard potential of inhaled fibrous material thereby offering better recommendations to reduce pulmonary disease.

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Publications

None

Materials Available to Other Researchers

The information in this report can be used by other investigators to prepare samples of fibers and examine them in the environmental scanning electron microscope. The in vitro dissolution procedure in this report can be followed to evaluate the effect of the solvent on the fibers.