Length-Reduction Method for Man-Made Mineral Fibers for Biological Experiments

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Received October 21, 1996 and accepted November 7, 1996

Abstract: A simple fiber length-reduction method was developed to obtain a large amount of fiber samples with different length distributions for use in various biological experiments. This press method is only to press a raw fiber sample charged in a stainless cylinder at an adequate pressure, and is effective for man-made mineral fibers (MMMF) such as glass wool, rock wool and refractory fibers (ceramic fibers, mullite fibers) and some brittle natural mineral fibers such as fibrous brucite and wollastonite. The mean fiber-length of man-made mineral fibers became shorter with the increase in the pressure applied without diameter change. We could obtain a length-reduced fiber sample with a suitable length distribution by this method. This press method is therefore a size-selective method able to produce a large amount of pulverized fiber sample depending on the press cylinder size for biological experiments. A very small amount of non-fibrous particles with aspect ratios (length vs. diameter) of under 3 was seen in the pulverized fiber samples. To eliminate such non-fibrous particles as well as too long fibers from the sample, separation by sedimentation in water was somewhat effective.

Key words: Length-reduction, MMMF, Glass fiber, Rock wool, Ceramic fiber, Biological experiments, Press method, Size distribution

Introduction

Since the carcinogenicity of asbestos was confirmed, various types of fibrous materials have been explored and developed, and widely used as asbestos substitutes in industrialized countries. At the end of the 1970s, several researchers had suggested that not only asbestos but also some other mineral fibers have carcinogenicity based on animal experiments on many kinds of mineral fibers and proposed the idea that “the carcinogenicity of mineral fibers is due to the morphology of long and thin fibers, and long durability in the lungs, and not due to the chemistry and crystal structure”^{1–4)}, which is the so-called “Stanton and Pott’s hypothesis.” Since then, this hypothesis has become an important working hypothesis for many researchers concerned with the study of mechanisms of fiber carcinogenicity.

By contrast to Stanton and Pott’s hypothesis, some recent biological studies suggested that the fiber type is also an

important factor in fiber carcinogenicity. That is, fiber carcinogenicity seems to vary not only with fiber size and durability but also the differences in fiber type, such as chemical composition, crystal structure and/or surface physico-chemical properties. For example, the potency of carcinogenicity of asbestos in the human body varies with the type, that is, crocidolite is the strongest, amosite is next, and chrysotile is weakest, as Doll (1989) summarized them⁵⁾. On the other hand, chrysotile showed signs of stronger carcinogenicity (higher incidence of mesothelioma) than crocidolite and amosite in animal experiments on rats⁶⁾. In addition, erionite, a fibrous zeolite, caused a higher incidence of mesothelioma than asbestos (chrysotile, crocidolite and amosite) in experiments on mice⁷⁾, and rutile (TiO₂) whisker in asbestos size did not show any sign of carcinogenicity (incidence of mesothelioma) in the experiment of intraperitoneal injection in rats⁸⁾.

There are therefore still many unsolved problems related to fiber carcinogenicity, and further biological studies have

been needed. To solve these problems and to clarify the mechanism of adverse health effects, biological experiments with fiber samples of which the properties well defined are very important. Assessment of the adverse health effects, showing which fibers have strong carcinogenicity and which fibers are safer, based on biological experiments with well defined fiber samples as well as epidemiological studies, is now urgently requested by society.

Fiber samples of well defined lengths and diameters were not available due to difficulties in experimental selection by size (length and/or diameter). Various methods have been applied to separate fibers of certain lengths from a raw sample. Stanton *et al.*¹⁾ obtained fiber samples of various lengths and diameters by varying the grinding time of their grinding machines such as ball mills and separating the fibers by sedimentation in water. Spurny *et al.*⁹⁾ made asbestos and glass fibers into fibers of certain lengths by a combination of a vibrating sieve procedure and a slow sedimentation in water. They concluded that the size-selective method had problems in reproducibility and not obtaining enough fiber samples. Myojo and Kohyama¹⁰⁾ reported an improved

separation method for asbestos (chrysotile) fibers by length, in which aerosolized fibers were separated by wire screens and a virtual impactor by length, but it has been difficult with these methods to obtain fibers of well defined length in a quantity sufficient for many biological experiments to be performed in different laboratories. Koshi *et al.*¹¹⁾ obtained a short micro glass fiber sample by an impact method with a long vertical cylinder. After it was filled with raw micro glass wool and the air in the cylinder was evacuated, a heavy piston at the top was allowed to drop. The strength of the impact pulverized the raw micro glass wool into short fibers, but it was difficult to control fiber length.

This paper describes a newly developed technique to produce a few hundred grams of fiber samples of well controlled lengths from raw fiber materials for use in biological experiments. This technique works for man-made mineral fibers of glass wool, rock wool (sludge wool), refractory fibers (ceramic fibers, mullite fibers) and some brittle mineral fibers, such as brucite fibers and wollastonite.

Apparatus and Method

A conventional oil press machine and a stainless press cylinder are used in this technique (Fig. 1). An example of stainless press cylinder is shown in Figure 2. A cylinder from which air can be evacuated is more desirable if available. The maximum pressure of the oil press machine is required to be at least 2 ton/cm². Consequently, when we use a cylinder

Fig. 1. Oil press machine and stainless press cylinder used in this study

Fig. 2. An example of a medium sized stainless cylinder (60 mm in diameter)

Table 1. Standard data for cylinder size, sample weight and pressure per cylinder

Cylinder size Diameter (mm)/Area (cm ²)	Standard weight of MMMf sample (g)	Example of pressure applied per cylinder (ton/cylinder)		
		0.5 t/cm ²	1 t/cm ²	2 t/cm ²
13/1.3	0.2	0.7	1.3	2.6
40/12.6	5	6	13	25
60/28.3	20	14	28	57
100/78.5	100	40	79	159
160/201	200	100	201	402
200/314	300	157	314	628

60 mm in diameter (28.3 cm²), for example, the maximum pressure of the oil press must be up to 60 ton. The size of the press cylinder is directly correlated with the capability of the oil press machine and the amount of sample to be treated at one time. The relationship between the size of the cylinder, the standard amount of sample and examples of pressure applied per cylinder are shown in Table 1.

A stainless cylinder of which the inner wall had previously been cleaned with ethanol was filled with raw sample of a man-made mineral fiber. The standard amounts to be inserted are shown in Table 1. The pressure applied by the oil press was gradually increased and kept for one minute at the desired pressure which is also indicated in Table 1. After one minute, the pressure was released and the pulverized sample in the cylinder was taken out. The pulverized sample was well mixed, then again put into the cylinder, and the above process was repeated to obtain a fiber sample evenly pressed.

To assess the effects of this method, examination by transmission electron microscopy (TEM) and/or optical microscopy (OM) was done to determine the size distributions of the pulverized fibers. The pulverized fibers were suspended in water by ultrasonic agitation and one droplet of the dispersion was placed on a TEM grid with carbon evaporated collodion film for TEM examination, or few droplets on a glass slide for OM examination. TEM photographs were taken at a low magnification of 400 times, and the images on the negatives were optically enlarged on the print 10 times. OM photographs were taken at a magnification of about 400 times on instant film. On these prints, the size (length and diameter) of each fiber was measured with a micro-rule (length) and a scale magnifier (diameter) for about 200 fibers. The size data for each fiber sample were calculated statistically and plotted on a log-normal distribution chart.

Results and Discussion

Application to micro-glass fiber with a small press cylinder

The effect of this technique was examined for micro-glass fiber (μ GF #104: Manville Co.). The raw micro-glass fibers were observed by SEM, showing very long and thin woolly fibers (Fig. 3). The size of the press cylinder was 13 mm in diameter (a commercial stainless cylinder for making pellet specimens for infrared spectrometry), the sample weights were about 200–300 mg, and the pressures applied to the sample were 1, 1.6, 2, 3, 5 and 10 ton/cylinder (1.3 cm²). The raw fiber sample was pulverized into a powdery sample by being pressed. The TEM photographs of the pulverized samples pressed at different pressure are shown in Figure 4, and the lengths and diameters were plotted on a log-normal distribution chart for each sample as shown in Figure 5. It was recognized that the raw fibers were pulverized into short fibers by the press and the fiber lengths decreased with the increase in pressure. Fibers of one micron meter to a few dozen micro meters in length were achieved at a pressure of 1 to 2 ton/cylinder (1.3 cm²). The mean length and geometrical standard deviation (SD) for each fiber sample pulverized by the press method are listed in Table 2. The mean fiber length of 4.5 μ m at 1 ton/cylinder was decreased with the increase in pressure to 1.8 μ m at 10 ton/cylinder and the SD also seemed to be decreased along with the increase in pressure. On the other hand, the mean fiber diameter and the SD were not changed at about 0.35 μ m and 2.2, respectively, with the increase in pressure. The differences between numbers in Table 2 would indicate experimental errors. The relationship between mean fiber length and the pressure applied is shown in Figure 6, indicating a close correlation between pressure and length with this method.

Through this study it was revealed that this press method can pulverize glass fibers, making them into short fibers, and can control the fiber length distribution by changing

Fig. 3. SEM photographs of micro glass fiber (#104; Manville Co.)

the pressure. The press method is a length-selective technique. On the other hand, it was not clear whether the same fiber length distributions could be obtained with the same pressure when the sample weight was changed, especially when it was increased. Within our limited range of experiments, it seemed that the fiber length distribution was widened with the increase in sample weight, even with the same pressure. The reason is supposed to be the increase in uneven pressure applied to fibers due to the uneven thickness of the sample layer in the cylinder. Therefore, to obtain relatively narrow length distribution in the fiber sample, the press treatment should be repeated on the same sample at least twice. As unwelcome evidence, a very small amount of non-fibrous particles with aspect ratios (length vs. diameter) of under 3 was seen in the final pulverized sample.

Application to rock wool by using a medium size press cylinder

On the basis of the above experiments, this press method was applied to a rock wool sample by using a relatively large press cylinder 60 mm in diameter (shown in Figs. 1 and 2). The given pressures were about 0.17–0.3 ton/cm² and the sample weights of rock wool were about 20 and 40 g. The OM photographs of pulverized rock wool samples were shown in Figure 7. RW0 was a “shotless” rock wool sample (which means that spherical particles of various sizes coexisting with raw rock wool were removed by an aerodynamic procedure) and already shortened a little as shown. RW1 was pressed at 0.2 ton/cm² and 0.3 ton/cm²

for a 20 g sample of RW0. RW2 and RW3 were pressed at 0.17 ton/cm² and 0.2 ton/cm² twice for 40 g samples of RW0, respectively. The fiber lengths were plotted on a log-normal distribution chart for each sample as shown in Figure 8. It was recognized that the fiber lengths decreased with the increase in pressure as seen in micro glass fibers. The mean fiber length and SD are shown in Table 3.

The same trend observed in micro-glass fiber, that is, the greater the pressure, the shorter the length, was seen in the data for RW2 and RW3 in this experiment applying a relatively large amount of rock wool. Moreover, it was also supposed that the mean fiber length and SD decrease with the increase in the sample weight as comparing the data between RW1 and RW3. As shown here, the press method needs at least two treatments to obtain a relatively narrow length distribution. It was unavoidable that a very small amount of non-fibrous particles was generated and contaminated the final pulverized rock wool sample. The amount, however, was roughly estimated to be under one percent in weight and would be permissible in most of the biological experiments.

To eliminate non-fibrous particles as well as too long fibers from the pressed fiber sample, separation by sedimentation in water was somewhat effective.

In our various experiments it was confirmed that if we use a larger press cylinder such as one 200 mm in diameter (314 cm²), about 200–400 g raw fiber sample can be pulverized at one time, providing similar results to those mentioned here.

Why only pressing can pulverize long fibers into short

Fig. 5. Length and diameter plotted on a log-normal distribution chart for each sample of micro glass fiber pulverized at different pressures

Fig. 4. TEM photographs of micro glass fibers pulverized at different pressures by this press method

(1) 1 t/1.3 cm², (2) 1.6 t/1.3 cm², (3) 2 t/1.3 cm², (4) 3 t/1.3 cm², (5) 5 t/1.3 cm², (6) 10 t/1.3 cm², Bar indicates 25 μm.

fibers without diameter change will be explained by the brittle properties of MMMF due to the amorphous state. We tried to apply this method to asbestos fibers, chrysotile and amosite, and some other natural mineral fibers such as sepiolite and palygorskite, but we could not pulverize them at pressures up to 30 ton/cm² because they were crystalline and had the

flexibility to withstand pressure. On the other hand, although mullite fibers, a kind of MMMF (ceramic fibers), are in a crystalline state, they could be easily pulverized into short fibers without any diameter change by this method. This could be explained by the fact that they were very brittle due to their polycrystalline state. Similarly, as brittle fiber is pulverized by this press method, fibrous brucite and

Table 2. Size parameters for micro glass fiber (#104) pulverized by the press method[§]

Pressure (ton/1.3 cm ²)	Length		Diameter	
	Mean (μm)	SD*	Mean (μm)	SD*
1	4.5	2.7	0.35	2.2
1.6	4.1	2.4	0.38	2.2
2	4.0	2.3	0.29	1.9
3	3.5	2.6	0.35	2.2
5	3.2	2.2	0.37	2.1
10	1.8	2.0	0.30	2.1

[§]: data obtained by TEM examination, *: geometrical standard deviation.

Fig. 6. The relationship of mean fiber lengths and pressure applied to micro glass fibers by the press method

Fig. 7. OM photographs of pulverized rock wool samples under different pressing conditions by the press method. The pressing conditions for each sample are listed in Table 3. Bar indicates 50 μm.

Fig. 8. Fiber lengths for each pulverized sample plotted on a log-normal distribution chart
The pressing conditions for each sample are the same as in Table 3.

Table 3. Size parameters for rock wool pulverized by the press method[#]

Sample Name	Pressing Condition	Length	
		Mean (μm)	SD*
RW0	Shot less by air blow	75	1.82
RW1	20 g, 0.2 t/cm ² + 0.3 t/cm ²	35	2.03
RW2	40 g, 0.17 t/cm ² × 2	33	1.94
RW3	40 g, 0.2 t/cm ² × 2	26	1.86

[#]: data obtained by OM examination for longer than 5 μm in length,

*: geometrical standard deviation.

wollastonite, which are both brittle natural mineral fibers, could also be pulverized into short fibers by this method although the pressures applied were high at about 20–30 ton/cm², and 3–5 ton/cm², respectively.

Conclusions

By using an oil press machine and a stainless cylinder, raw man-made mineral fibers could be pulverized into short fibers. The fiber lengths correlated with the pressure applied, that is, the greater the pressure, the shorter the length. This press method is, therefore, a size-selective method able to produce a large amount of pulverized fiber sample for biological experiments depending on the press cylinder size. We can directly compare the results of various biological experiments in different laboratories or groups by using the same fiber samples but with different length distributions prepared by this method.

Acknowledgments

The author express his sincere thanks to Ms. S. Kurimori

of the National Institute of Industrial Health, Japan, for her help.

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