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A computer program has been set up in Fortran language to calculate pore size distribution using the Cranston-Inkley method of calculation. Nitrogen adsorption isotherms were determined on amosite and chrysotile asbestos at -195°C . Size reduction had a major effect on increasing pore volumes of both asbestos materials.

The porosity of amosite and chrysotile asbestos

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Introduction

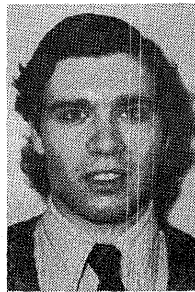
In a recent study,¹ two of the authors made a series of 56 adsorption-desorption isotherms of argon and nitrogen on amosite and chrysotile asbestos in three states of subdivision. This work showed the chrysotile to have a larger surface area and greater porosity than the amosite, and more hysteresis on desorption. A limited number of Cranston-Inkley² calculations and Sing³ plots confirmed this. In the present work, Cranston-Inkley calculations were made from nitrogen isotherms obtained at -195°C to determine the effects of precon-

ditioning temperature and size reduction on pore size distribution and total porosity.

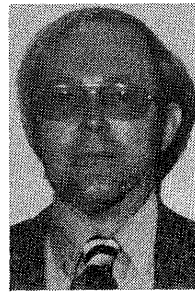
Asbestos is a generic term applied to certain fibrous, inorganic minerals. Chrysotile is a serpentine mineral with an idealized empirical formula of $3\text{MgO}\cdot 2\text{SiO}_2\cdot 2\text{H}_2\text{O}$. Chrysotile fibers are currently considered to be tubular with material of lower electron density than the fiber walls filling the tubes.⁴ Amosite is a member of the amphibole group of minerals and is monoclinic in shape. Chemically, it is a magnesium iron silicate ($\text{Fe}^{2+} +_{5.5}\text{Mg}_{1.5}\text{Si}_3\text{O}_{22}(\text{OH})_2$). Amosite fibers are made of laths which appear



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Lloyd E. Stettler received B.S. in Chemistry from Case Western Reserve University, Cleveland, Ohio; M.S. and Ph.D. in Physical Chemistry from University of Cincinnati, Ohio. Previously with the U.S. Public Health Service, Division of Air Pollution from 1964-1968. Employed at USPHS, NIOSH, Cincinnati, Ohio since June 1971.

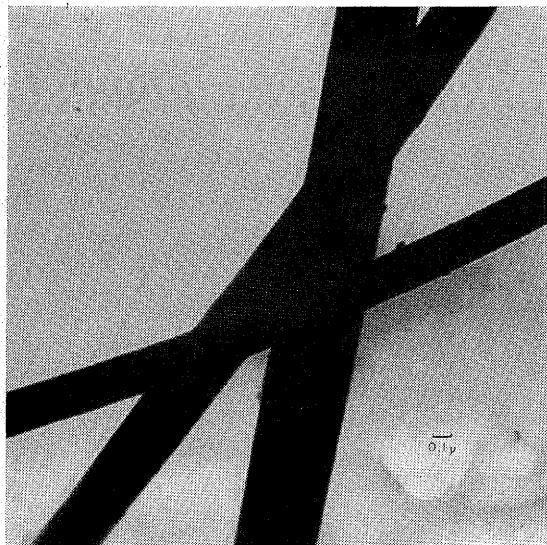


Figure 1—Electron micrograph of amosite.

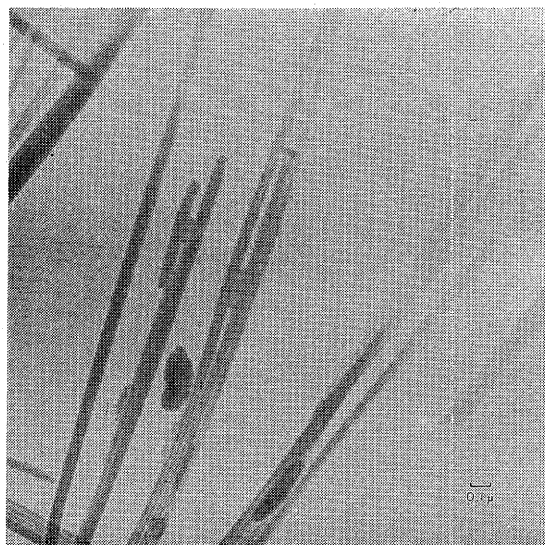


Figure 2—Electron micrograph of chrysotile.

to be made of layer upon layer of thin, sheet-like material.⁵

Electron micrographs were made of the individual fibers of both materials at total magnifications of approximately 65,000X. These are shown in Figures 1 and 2. They confirm the tubular structure of chrysotile and show the amosite fibers to be solid and considerably greater in diameter than the chrysotile.

Materials and methods

The amosite used was from the H. K. Porter Company* operation in South Africa and was designated 202-901. The chrysotile consisted of loose fibers obtained from the Bell mines in Canada and was marked Grade 3T.

Adsorption-desorption isotherms were made using the surface properties equipment described by Scheel, *et. al.*⁶ This system consists of a Cahn RH electrobalance for measuring weight change of the sample during adsorption, a Wallace and Tiernan Model FA 145 Bourdon gauge for measuring pressures from 1 to 800 torr, and a Bendix GT-340 thermistor gauge for measuring pressures from 0 to 1 torr.

Experiments were made with both asbestos samples in three states of subdivision, namely, as received, mechanically dispersed, and impact milled at liquid nitrogen temperature. The mechanically dispersed asbestos was prepared by mixing 5 grams of sample and 1,000 grams of distilled water in a Ver-Tis mixer for 20 minutes at 20,000 RPM, using a tantalum impeller. This method has been used in the National Institute for Occupational Safety and Health laboratory for preparing asbestos samples for animal exposure tests. The impact milled material was prepared by grinding in a Spex mill cooled with a liquid nitrogen bath. This effectively broke up the fibers so that their geometric mean sizes were 0.8 μ in length and 0.31 μ in diameter for amosite, and 0.61 μ in length and 0.15 μ in diameter for chrysotile.

Nitrogen was obtained from the Air Reduction Company. It contained 5.3 ppm argon, 0.5 ppm hydrocarbons, and it had a dewpoint of -105°C .

A series of preconditioning experiments was made at 25, 150, 250, and 400 $^{\circ}\text{C}$ to determine the effect of temperature on surface area. Since there was no difference in surface area at 25 and 150 $^{\circ}\text{C}$, no further tests were made at 150 $^{\circ}\text{C}$. Preconditioning was thus done at 25, 250, and 400 $^{\circ}\text{C}$ for a minimum time of 16 hours while pumping to a final pressure of 0.4-1.5 $\times 10^{-7}$ torr.

*Mention of commercial names or concerns does not constitute endorsement by the Department of Health, Education, and Welfare.

TABLE I
Program for Determining Porosity at Various Pore Sizes

DIMENSION L(37),T(37),R(36),C(36),F(35,35),D(36),V(36),S(36),	PSD1 10
1DESC(12),VNTP(37)	PSD1 20
DATA L/300,290,280,270,260,250,240,230,220,210,200,190,180,170,160	PSD1 30
1,150,140,130,120,110,100,90,80,70,60,50,45,40,35,30,25,20,18,16,14	PSD1 40
2,12,10/	PSD1 50
DATA T/13.52,13.52,13.43,13.32,13.17,13.05,12.89,12.74,12.59,12.45	PSD1 60
1,12.29,12.12,11.93,11.71,11.50,11.26,11.03,10.78,10.51,10.20,9.88	PSD1 70
2,9.530,9.150,8.705,8.215,7.638,7.243,6.858,6.427,5.962,5.443,4.819	PSD1 80
3,4.333,4.008,3.606,3.116,2.472/	PSD1 90
DATA C/0...5,.52,.54,.56,.58,.60,.62,.65,.68,.71,.75,.79,.84,.89,	PSD1 100
1.95,1.02,1.1,1.19,1.3,1.44,1.6,1.8,2.08,2.44,1.4,1.56,1.76,2.,2.34	PSD1 110
2,2.86,1.34,1.49,1.68,1.93,2.26/	PSD1 120
DO 30 I=1,36	PSD1 130
J= I+1	PSD1 140
IF (I.EQ.1)GO TO 20	PSD1 150
TIJ= T(I)+T(J)	PSD1 160
K= I-1	PSD1 170
DO 10 N=1,K	PSD1 180
10 F(K,N)= (D(N)-TIJ)/D(N)**2	PSD1 190
20 O(I)= (L(I)+L(J))/2.	PSD1 200
30 R(I)= (D(I)/(D(I)-2.*T(J)))**2	PSD1 210
C *** READ NUMBER OF ADSORPTION ISOTHERM DATA SETS *****	PSD1 220
READ 1,NRUN	PSD1 230
DO 90 ISET=1,NRUN	PSD1 240
C *** READ SAMPLE # ;DESCRIPTION, STEP VOLUMES & B.E.T. SPECIFIC SURFACE	PSD1 250
READ 1,NSET,DESC,(VNTP(I),I=1,37),SBET	PSD1 260
1 FORMAT(2X,I3,2X,12A4/10F8.3/10F8.3/10F8.3/7F8.3/F8.3)	PSD1 270
CUMV= 0.	PSD1 280
CUMS= 0.	PSD1 290
DO 60 I=1,36	PSD1 300
SUM= 0.	PSD1 310
IF (I.EQ.1)GO TO 50	PSD1 320
K= I-1	PSD1 330
DO 40 N=1,K	PSD1 340
40 SUM= SUM+F(K,N)*V(N)	PSD1 350
50 V(I)= R(I)*(VNTP(I)-VNTP(I+1))-C(I)*SUM	PSD1 360
IF (V(I).LT.0.)GO TO 70	PSD1 370
CUMV= CUMV+V(I)	PSD1 380
S(I)= 63.36*V(I)/D(I)	PSD1 390
60 CUMS= CUMS+S(I)	PSD1 400
K= I	PSD1 410
70 PRINT 2,NSET,DESC	PSD1 420
2 FORMAT(1H1/25H PORE SIZE DISTRIBUTION--/9H SAMPLE #,I3,2H :,12A4//	PSD1 430
15X,59H LAYER EST. PORE MEAN SURFACE PERCENT CUMULATIVE/	PSD1 440
25X,57HDIAMETER VOLUME DIAMETER AREA OF TOTAL PERCENT/62	PSD1 450
3H N (A) (ML-NTP/G) (A) (M*M/G) AREA(%) AREA(%) /62	PSD1 460
4H -- -----) PSD1 470	
CPCT= 0.	PSD1 480
DO 80 N=1,K	PSD1 490
PCT= 100.*S(N)/CUMS	PSD1 500
CPCT= CPCT+PCT	PSD1 510
80 PRINT 3,N,L(N),L(N+1),V(N),D(N),S(N),PCT,CPCT	PSD1 520
3 FORMAT(I3,2X,I3,1H-,I3,F10.3,F10.1,F10.3,2F10.2)	PSD1 530
PRINT 4,I,L(I),L(I+1),V(I)	PSD1 540
4 FORMAT(22H LAST VALUE-----/I3,2X,I3,1H-,I3,F10.3/1X,61(1H-))	PSD1 550
PCTV= 100.*(CUMV-VNTP(1))/VNTP(1)	PSD1 560
PCTS= 100.*(CUMS-SBET)/SBET	PSD1 570
AVGD= 63.36*VNTP(1)/SBET	PSD1 580
90 PRINT 5,CUMV,CUMS,VNTP(1),SBET,PCTV,PCTS,AVGD	PSD1 590
5 FORMAT(15H TOTALS V'=,F7.3, 13H S'=,F7.3//	PSD1 600
1 15H PORES<300A: V=,F7.3, 13H B.E.T.: S=,F7.3//	PSD1 610
2 16H 100(V'-V)/V=,F5.1,15H% 100(S'-S)/S=,F5.1,1H%//	PSD1 620
3 44H AVERAGE PORE DIAMETER= 4(.001584V/S)*10000=,F6.2,3H A.)	PSD1 630
END	PSD1 640

TABLE II
Summary of Results from Cranston-Inkley Calculations

SAMPLE	PRECONDITIONING TEMPERATURE, °C.	BET SURFACE AREA SQ. M./G.	AVG. PORE DIAM., A	TOTAL PORE VOLUME ML./G.
Amosite, As Received	25	1.3	58.5	0.0019
	250	2.2	63.6	0.0035
	400	2.8	55.1	0.0039
Amosite, Mech. Dispersed	25	5.9	68.0	0.0100
	250	6.9	61.0	0.0105
	400	7.0	54.4	0.0095
Amosite, Impact Milled	250	11.9	85.7	0.0255
Chrysotile, As Received	25	15.3	56.4	.0216
	250	19.3	62.9	.0296
	400	22.1	61.9	.0342
Chrysotile, Mech. Dispersed	25	30.6	78.1	.0598
	250	36.1	64.1	.0578
	400	36.3	66.4	.0602
Chrysotile, Impact Milled	250	48.4	80.6	.0975

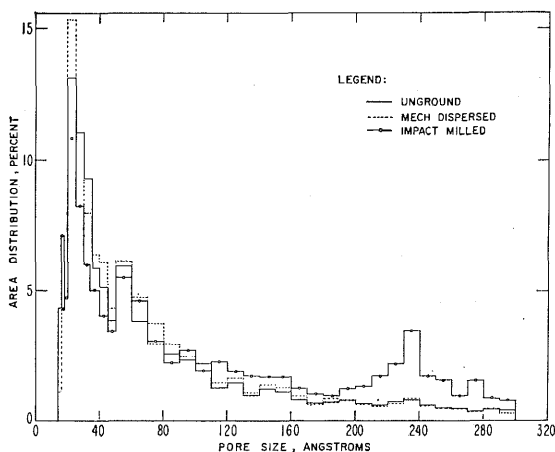


Figure 3—Effect of grinding on amosite pore size.

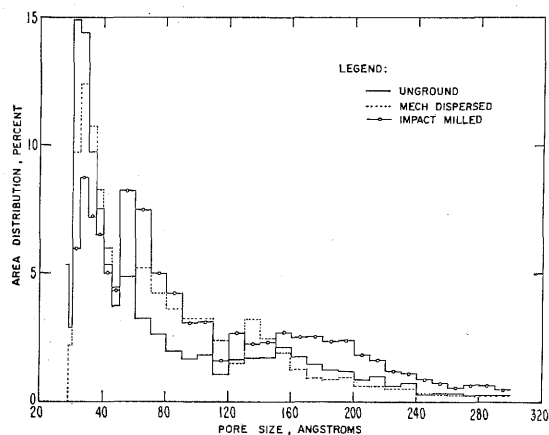


Figure 4—Effect of grinding on chrysotile pore size.

Pore size distribution was calculated using the method of Cranston and Inkley.² This method makes three principal assumptions:⁴

1. The pressure at which a pore of a given radius empties follows the Kelvin equation $\ln(P/P_0) = 2\gamma V/RT_r$ where P = pressure above an interface with radius of curvature, r , P_0 = vapor pressure for temperature, T , γ = surface tension, V = molar volume, R = gas constant.
2. As a pore empties, the wall of the pore retains an adsorbed layer whose thickness varies like that of a flat surface.
3. Pores larger than 300A in diameter contribute little to the total surface area of the specimen.

The method of calculation is given in considerable detail by Cranston and Inkley.² Essentially, the volume of nitrogen in each pore size increment is calculated. This type of calculation is readily performed with a computer. A program has been developed using an IBM 370 computer with Fortran language. The program in a form suitable for keypunching is shown in Table I.

Results

The data are tabulated in Table II. Figures 3-6 are bar graphs showing the effect of grinding on the pore size distribution of amosite and chrysotile, and the effect of preconditioning temperature on the pore size distribution of mechanically dispersed amosite and chrysotile.

As may be seen, chrysotile is much more porous than amosite. However, in most cases similarly processed samples of amosite and chrysotile have similar average pore diameters.

As expected, size reduction has a major effect on the pore volumes of both asbestos materials. Mechanical dispersion increases the total pore volume of untreated amosite by a factor of three and impact milling increases this value approximately seven times. With chrysotile, which is considerably more porous, the increases in total pore volumes, are approximately two times and 3½ times, respectively. Figures 3 and 4 show that mechanical dispersion has very little effect on the pore size distributions of both asbestos samples. Impact milling, in which the samples are subjected to collision with 440C stainless steel rods, gives pore size distributions that contain larger amounts of pores greater than 100A in diameter for amosite and 150A for chrysotile than the unground and mechanically dispersed samples.

For years, investigators at NIOSH and other laboratories have noted that finely ground asbestos fibers lost much of their fibrogenicity.⁷ This has been attributed to shorter fiber length and loss of crystallinity on grinding.⁸ There is a considerable difference of opinion as to the effective median fiber length with values of 2 to 20 μ being reported.⁹⁻¹¹

It would appear that the change in pore size distribution caused by impact milling may have some effect on the lack of toxicity of these fibers. Any material adsorbed in larger pores (>100A) would be more readily desorbed while the smaller pores would tend to retain adsorbate. We doubt that the crystallinity of the impact milled material has been affected since grinding occurred at -195°C, thus preventing local overheating of the fibers.

Increasing the preconditioning temperature from 25 to 400°C, has little effect on the total pore volume of mechanically dispersed amosite and chrysotile. However, the samples preconditioned at 250 and 400°C had smaller average pore diameters than the sample preconditioned at 25°C. The data are plotted in Figures 5 and 6 showing the percent area distribution in the various pore size fractions. Evidently, mechanical dispersion broke up many of the fiber bundles, thus creating more

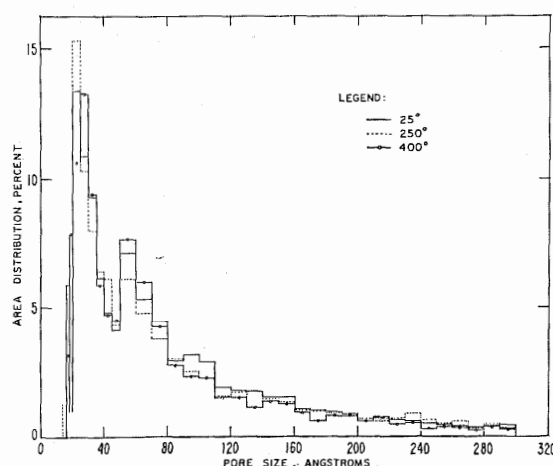


Figure 5—Effect of preconditioning temperature of mechanically dispersed amosite.

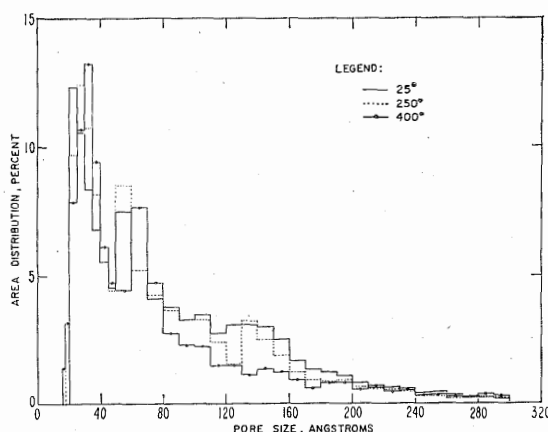


Figure 6—Effect of preconditioning temperature on mechanically dispersed chrysotile.

surface area. Although more water was volatilized at higher preconditioning temperatures as determined by weight loss, there was little effect on the total pore volume, thus indicating that the water was held on the surface or in pores smaller than 10A in diameter which were unavailable to nitrogen.

In the case of unground amosite and chrysotile, increasing the preconditioning temperature increases the total pore volume, thus indicating that volatilizing water must create more pores. The effect on average pore diameter was minor. Examination of the material heated to 400°C, showed no physical difference

between it and the original sample except for a slight darkening in color. Comparison of electron micrographs of the original and pre-conditioned samples showed the materials to be essentially the same.

More surface area was contained in pores between 20 and 30A diameter than in any of the other fractions. This is similar to the results obtained by Naumann and Dresler,⁴ who found a maximum in the pore size distribution at 25A using chrysotile from Cassiar, B.C.

References

1. GORSKI, C. H. and L. E. STETTTLER: The Surface Energetics of Asbestos Minerals. *Am. Ind. Hyg. Assoc. J.* 35:345 (1974).
2. CRANSTON, R. W. and F. A. INKLEY: The Determination of Pore Structures from Nitrogen Adsorption Isotherms. *Adv. Catal.* 9:143 (1957).
3. SING, K. S. W.: Empirical Method for Analysis of Adsorption Isotherms. *Chem. and Ind.* (London), 829 (1967).
4. NAUMANN, A. W. and W. H. DRESHER: The Morphology of Chrysotile Asbestos as Inferred from Nitrogen Adsorption Data, *Am. Mineral.* 51:711 (1966).
5. HUGGINS, C. W.: Bureau of Mines R. I. 5551, Electron Micrographs of Asbestiform Minerals (1959).
6. SCHEEL, L. D., C. H. GORSKI, R. W. VEST, L. E. STETTTLER, W. C. TRIPP and G. P. GRAY: A Gravimetric System to Study Adsorption on Fine Powders. *Am. Ind. Hyg. Assoc. J.* 32:673 (1971).
7. HILSCHER, W., S. SETHI, K. H. FRIEDRICH, and F. POTT: Zusammenhänge zwischen Asbestos und Faserlänge. *Naturwissen.* 57:356 (1970).
8. ORRELLA, E. and G. MADDALON: X-ray Diffraction Characteristics of Some Types of Asbestos in Relation to Different Techniques of Comminution. *Med. Lavoro* 54:628 (1963).
9. VORWALD, A. J., T. M. DURKAN and P. C. PRATT: Experimental Studies of Asbestosis. *Arch. Ind. Hyg. Occup. Med.* 3:1 (1951).
10. WAGNER, J. C.: Asbestosis in Experimental Animals. *Brit. J. Ind. Med.* 20:1 (1963).
11. HOLT, P. F., J. MILLS, and D. K. YOUNG: The Early Effects of Chrysotile Asbestos on the Rat Lung. *J. Pathol. Bacteriol.* 87:15 (1964).

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