

ANALYSIS OF RESPIRABLE AND SEDIMENTED DUST SAMPLES

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Summary

Determination of phase and metal content of dust samples collected from different workplaces was carried out. Qualitative and semi-quantitative measurements were taken by X-ray powder diffraction, X-ray fluorescence spectrometry, IR-spectroscopy, emission spectrography and SEM-EDAX. Amount of chrysotile in asbestos cement dust and quartz in total and respirable enamel dust was determined by IR spectroscopy.

Introduction

Despite the safety devices there is still a rise in the dust emission into the air. However, public awareness has called for tighter standards and more monitoring programmes. The measurement of dust emission usually contains the weighing of the amount of dust only. On the analytical side, there is an ever increasing demand for determination of the metal and phase composition of the dust samples.

In this work, sedimented asbestos cement dusts and total, as well as, respirable dusts were investigated. Qualitative and semi-quantitative determination of metal and phase content of the samples was carried out by X-ray powder diffraction, infrared spectroscopy, X-ray fluorescence spectrometry, emission spectrography and scanning electron microscopy.

Experimental

Total and respirable dust samples were collected by personal sampler on Microsorban membrane filter in an enamel factory. Amount of the samples ranged between 1.2–22 mg. Recovery of the dust from the membrane filter was carried out by dissolving the membrane in benzene (analytical grade, Reanal).

Samples of sediments from various sampling sites and standard asbestos cement were obtained from the Central Research and Design Institute for Silicate Industries. Samples were ground in a tungsten carbide swing-grinding mill (Model HSM 100, Herzog, FRG). IR-spectra were recorded by a Specord

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75 IR-spectrophotometer (Carl Zeiss, Jena, GDR), X-ray powder diffractograms and SEM pictures were obtained by a Model 1051 Philips diffractometer and JSM-U3 (Jeol, Japan), respectively. Particle size distribution in the pellet was measured by a VIDIMAT Image Analyser (Research Institute for Ferrous Metallurgy). Qualitative analysis of metals was carried out by PGS emission spectrograph (Carl Zeiss, Jena, GDR) and Philips PW 1410 X-ray spectrometer, respectively.

Pellets were prepared by dilution method using ethanol to avoid agglomeration of the primary particles [1]. The mean particle size in the pellet was determined as described recently [2].

Results and discussion

Asbestos cement sediments

Phase analysis of the dust samples was carried out by X-ray powder diffraction. To avoid the orientation effects, the samples were placed in a shaken sample holder. From the diffractograms phases were found as follows: chrysotile, tricalcium-silicate, dicalcium-silicate, tricalcium-aluminate, brown-millerite, calcit, quartz and hematite. Along with the chrysotile, no other asbestos were found in the samples. Furthermore, the samples did not contain clay minerals. It means that the IR-spectroscopic method developed recently can be used for determination of chrysotile content of asbestos cement dusts [1]. O—H stretching vibration of chrysotile at 3660 cm^{-1} was used for quantitative measurements. All samples were ground for 15 min in a tungsten carbid swing-grinding mill. The optimal grinding time was determined by SEM method [3]. Standard samples were prepared from portland cement and chrysotile according to the technology of the factory. Calibration curve reads: $A = 5.65 \cdot 10^{-3} + 5.21 \cdot 10^{-3} C_k$, where A = absorbances at 3660 cm^{-1} and C_k = amount of chrysotile. $S_{\text{reg}} = \pm 7.52 \cdot 10^{-3}$, $r = 0.995$. The average particle diameter in the pellet, $d = 2.34 \pm 0.086\text{ }\mu\text{m}$, relative error, r.e. = $\pm 3.67\%$. Chrysotile content of some asbestos cement dusts is shown in Table 1.

Table 1

Chrysotile content of asbestos cement dusts

Samples	Amount of chrysotile [%]	Ave. particle diameter, d , [μm]
1	40.5 ± 4.7	2.16
2	37.9 ± 4.6	2.12
3	15.7 ± 4.5	2.08
4	9.6 ± 4.9	1.91
5	8.8 ± 4.9	1.95
6	6.7 ± 5.1	1.92

Total and respirable dusts

Metal content of the total and respirable dust samples were investigated by emission spectrograph and X-ray fluorescence spectrometry. Personal samplers were attached to the clothes of the workers in different workplaces. Results of some measurements are summarized in Table 2.

Table 2

Metal content of total and respirable dust samples collected in an enamel factory

Samples	Main components (>3%)	Impurity (0.1–3%)	Trace (<0.1%)
Enamel spraying	Si, Fe, Al	Ca, B, Co, Zn, Ti	Ba, Mn, Pb, Cr, V, Ni, Na, Sr, K, Zr, Mg, As, Be, Sb, Ag, Cu, Pd
Enamel mill	Si, Al, Mg, Ca	Ba, Ni, Co, Pb, B, Ti, Cu, Mn, Sn, V, Fe, K	As, Na, Zn, Sb, Ag, Cr, Sr, Zr
Welder	Si, Al, Mg, Pb, Mn	Be, Ba, Ca, Na, Zn, Ti, Ni, Cu, Fe, V	Sb, Sn, Mo, Cr, B, Zr, Co, As, Pd, Ag, K, Sr
Turner	Si, Mg, B, Al, Ca	Na, Co, Ti, Zr, Cu, Ba, Fe, V	Ni, Zn, Ag, Pb, Mn, Sn, P, K, As, Be, Sb, Cr, Sr, Mo

Table 3

Metal content of enamel dusts

Samples	Metals
Membrane filter	Ca > Ti > Fe > Zn > Ni > Mn > Ga
Enamel furnace	Ti > Ca > Fe > Zn > Sb > K > Ni > Mn > Ga
Enameling	Ti > Ca > Ba > Fe > Co > Zn > Zr > Sb > Ni > Mn > Ga
Grinder	Ti > Ca > Fe > Zn > Co > Ba > Ni > Mn > Zr > Ga
Plasticizer	Ti > Ca > Fe > Zn > Ba > Sb > Ni > Mn > Ga > Co

Some samples were investigated directly on the membrane filter by X-ray fluorescence spectrometry. Amount of the dust collected ranged between 2–20 mg. Results are shown on Table 3.

It can be seen from the data that the samples contain Ba, Co, Zr and Sb in the amount of detection limit.

Quartz content of the dust samples was determined by IR-spectroscopy. Pellets were prepared by means of alcoholic treatment. Absorbances at 800 cm^{-1} (ν_a Si—O—Si) were measured from the spectra. Calibration curve

Table 4
Quartz content of total and respirable dusts

Samples	Quartz [%]	Average particle size [μm]
Basal enamel	13.3 ± 3.3	2.25
Basal enamel mud	9.5 ± 3.3	2.21
Enamel mill	12.9 ± 3.3	1.97
Enamel spraying	10.7 ± 3.3	2.01
Enamel spraying	7.6 ± 3.4	2.16
Enamel application	2.8 ± 3.5	1.92

reads: $A = 1.11 \cdot 10^{-2} + 3.69 \cdot 10^{-1} C_q$, $S_{\text{reg}} = \pm 2.71 \cdot 10^{-3}$ and $r = 0.999$.
Average particle size measured in pellets was found as $d = 1.48 \pm 0.045 \mu\text{m}$.

Quartz content of some dust samples is listed in Table 4.

Confidence limit of the determination was found to be $\pm 3.4\%$ in average.

References

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