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CHROMIUM AND NICKEL FROM SOME THERMAL
SPRAYING OPERATIONS

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CHARACTERISTICS OF AEROSOLS CONTAINING CHROMIUM AND NICKEL FROM SOME THERMAL SPRAYING OPERATIONS

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Abstract

Characterizations of aerosols, emitted from five methods of thermal spraying with materials containing chromium and nickel, were carried out. The characterization procedure includes the determinations of the metal content, the particle size distribution, the oxidation state of chromium and a measure of the solubility of chromium. Three different kinds of samplers and the analytical methods PIXE (Particle Induced X-ray Emission analysis), ESCA (Electron Spectroscopy for Chemical Analysis), AAS (Atomic Absorption Spectrophotometry), spectrophotometry with DPC-reagent (sym-diphenylcarbazide) and TEM (Transmission Electron Microscopy) were used. The relative concentrations of the major elements in the aerosols agree with the relative abundances in the spraying material. A considerable part of the aerosol mass was found in the respirable fraction. Mass median aerodynamic diameters in the respirable fractions were below 0.5 μm . Hexavalent chromium, which is the oxidation state of chromium with the highest health hazard potential, was determined. Less soluble hexavalent chromium was detected in some of the spraying methods.

Introduction

Thermal spraying is a technique for coating objects with a surface layer of material with high resistance to e.g. wear and effects of chemical agents (3). The technique is often used for repairing machine details. The material which is to be deposited is heated by either an oxygen-acetylene flame (flame spraying), an electric arc (electric arc spraying) or in a plasma (plasma spraying). The heated material is brought to the object by either the expanded gas (flame spraying and plasma spraying) or by using a jet of compressed air (flame spraying and electric arc spraying). The usage of a large variety of materials with different properties occurs. Metals such as molybdenum, aluminum, copper, zinc, chromium and nickel are often components of the spraying materials, which can be in the form of wires (all methods) or powders (flame and plasma spraying).

The use of this technique give rise to various environmental problems such as high noise levels, UV-radiation and the emission of toxic gases and aerosols.

When carrying out epidemiological research on occupational hazards of exposure to airborne particles containing chromium and nickel, there is a lack of knowledge about the character of the aerosols (11) and hence the objective of this study was to characterize the aerosol emission from methods of thermal spraying with materials containing chromium and nickel. Chromium and nickel are among the most toxic metals used in thermal spraying materials. There is extensive documentation of the toxicity of different chromium and nickel compounds (9,10). Several compounds of these metals have been shown to be carcinogenic. For chromium the oxidation state is of major importance. Cr(VI)-compounds have much higher health hazard potential than Cr(III)-compounds. There is no evidence of carcinogenicity for extra cellular Cr(III)-compounds. For Cr(VI)-compounds water solubility is also a parameter of considerable interest. Indications exist that the less soluble compounds are those of highest carcinogenicity (11).

The toxicity of an aerosol is strongly dependent on the particle size

distribution and the chemical composition of the particles. The characterization procedure used in the present work includes the determination of the particle size distribution, the elemental composition, the oxidation state of chromium and a measure of the solubility of chromium.

Materials and methods

Thermal spraying methods

Five common methods of thermal spraying with materials containing chromium and nickel were chosen. The techniques flame spraying, electric arc spraying and plasma spraying were included. Table 1 describes the methods.

SPRAYING METHOD	SPRAYING MATERIAL	SPRAYING EQUIPMENT	PERCENTAGE OF THE MAJOR METALS IN THE MATERIAL (% of weight)	DIAMETER OF WIRE (mm)	SPRAYING CAPACITY (kg/h)
1a. flame spraying	wire METCOLOY 2	METCO 10E	Fe: 85 Cr: 13 Ni: 0.5	3.17	5.9
1b. flame spraying	wire METCOLOY 2	METCO 3K (3 spraying guns)	Fe: 85 Cr: 13 Ni: 0.5	4.76	3 x 7.26
2. flame spraying	powder METCO 44	METCO 5P	Ni: 76 Cr: 16 Fe: 8	-	8.2
3. flame spraying	wire METCOLCY 33	METCO 10E	Ni: 60 Cr: 16 Fe: 22.5	3.17	5.9
4. electric arc spraying	wire METCOLOY 2	METCO RG (32V, 320A)	Fe: 85 Cr: 13 Ni: 0.5	2.31	14.5
5. plasma spraying	powder METCO 44	METCO 3 MB	Ni: 76 Cr: 16 Fe: 8	-	9.1

Table 1

The spraying methods used. Spraying equipment and spraying materials were manufactured by METCO AB. Settings of gas mixtures, gas flows etc. and distance between the nozzle of the spray-gun and the object according to the recommendations of the manufacturer. Method 1 b was used during sampling under normal production conditions.

Sampling

The spraying and the sampling of aerosols from the five methods were performed in a room sealed off from other activities in a thermal spraying workshop. For one of the methods the characterization was also performed during normal production, where a paper-mill roll was coated with a chromium containing material by flame spraying. A higher spraying capacity, than for the "sealed room" measurements of the same method, was used.

In conventional sampling personal monitors are used. So-called total filter samplers for a "total fraction", or collection on filters after a pre-collection stage for the respirable fraction. Since the main purpose of this study was to explore the emission of aerosols from thermal spraying operations we used more well-defined sampling procedures. For sampling during the normal production, however, personal monitoring was also performed.

The definition of the "total" fraction is often dependent on a specific sampling system and specific sampling conditions. The upper limit of particle size collected depends on the design of the sampling inlet and the air flow rate. Speed and direction of the air movement around the inlet also strongly influence the particle size characteristics of the aerosol collection. Criteria for perfect sampling have been suggested by Agarwal and Liu (2) and Davis (5). The purpose of these criteria is to secure a complete sampling of an aerosol with particles below a given size. For purposes of health effect assessment Vincent and Armbruster (14) suggest sampling procedures with particle size collection efficiencies simulating the collection efficiency of the human respiratory system as determined by experimental studies.

There has been an extensive discussion during the last few years about methods and criteria for fractionating aerosols according to particle size for health effect evaluations. In a recent suggestion for an ISO-standard (12) the total suspended particles are divided into fractions which should reflect probable deposition sites in the human

respiratory system based on the available data from lung deposition studies. An "inspirable" fraction is defined as the part of the total suspended particles which reaches the human airway. The inspirable fraction consists of an extra-thoracic and a thoracic fraction. The thoracic fraction is further divided into two sub-fractions; respirable and tracheo-bronchial. For the respirable fraction there are two definitions; The BMRC-definition (British Medical Research Council) and the ACGIH-definition (American Conference of Governmental Industrial Hygienists). Approved instruments for respirable sampling according to the British and American standards can be used, after a precollection stage according to the definition of the inspirable fraction, for sampling according to the suggested standard. However even if the instruments are used without any precollection stage the differences in collection efficiencies are below the permissible error laid down by the ad hoc working group of ISO (12).

Techniques for "total" sampling and respirable sampling were utilized in the present work. The choice of sampling techniques was determined by the demands on them for well defined particle size fractionations and for generating samples suitable for various analytical techniques. Our intentions were further to collect fractions which could be compared with samples obtained by personal monitoring (total and respirable). However, since personal sampling with "total" samplers does not constitute well defined sampling with respect to particle size, only rough comparisons can be performed. Orientation of the sampler and local air movements around the bearer and the sampler can strongly influence the particle size characteristics of the sampling. The definition of "total" fraction in this work is given in figure 1, which also shows the respirable fraction and the fractions according to the suggested ISO-standard. The "total" sampling was performed for estimation of the relative fraction of the respirable fraction in conventional total filter measurements (idealized case) and for estimation of the relative abundances of elements in different size fractions. Care should however be taken when drawing conclusions about the health hazard potential from information on the magnitude of the coarse fraction. The relevance of sampling different particle size fractions to what actually is inhaled by humans in different situations was recently discussed by Vincent and Mark (16).

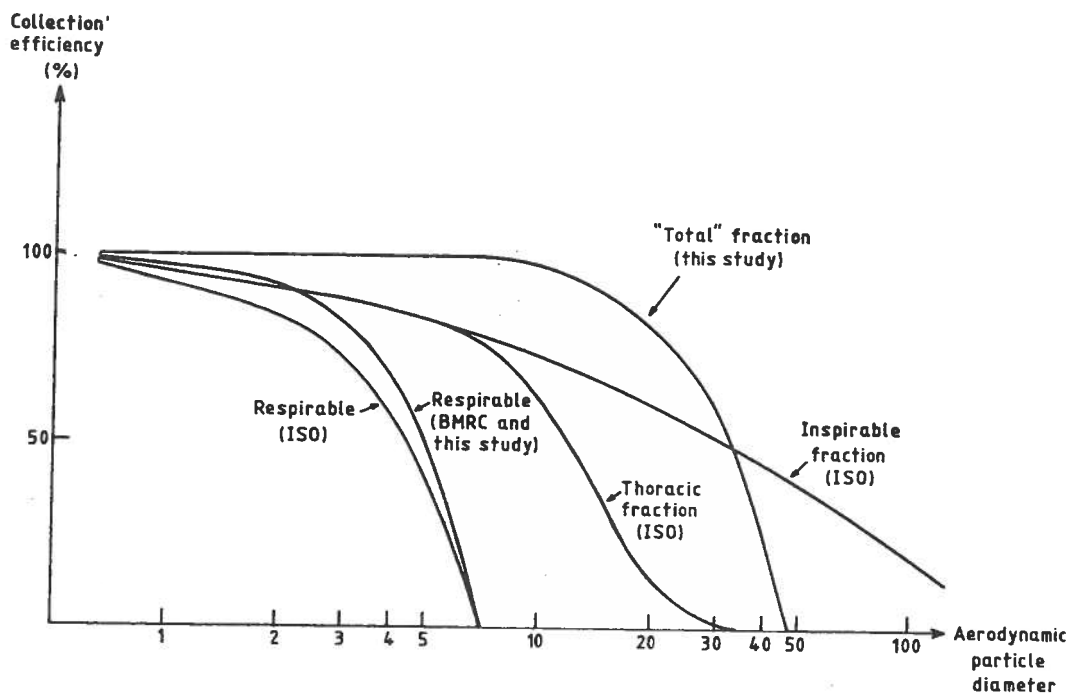


Figure 1. Theoretical collection efficiencies for the multi-filter sampler as a function of aerodynamic particle diameter. For comparison the suggested ISO-standard for thoracic fraction and inspirable fraction are given. The respirable fraction used is that of the BMRC standard for respirable dust.

Three types of samplers were used: 1) a multi-filter sampler for total and respirable fractions, 2) a modified Battelle impactor and 3) a sampler with two parallel filters prepared for transmission electron microscopy studies.

Figure 2 shows the multi-filter sampler. It consists of four filters (Millipore GSWP, 37 mm diameter, 0.22 μm pore size), two of them with pre-collectors (cyclone pre-collector; Casella Personal Sampler) (A and B) and two without (C and D). A cylindrical common inlet with a length of 300 mm and a diameter of 50 mm was used to avoid the effects of turbulence which would affect the collection efficiency. The inner surface was of aluminum, electrically ground, in order to minimize losses due to electrostatic effects. The air flow through each filter was 1.9 l/min which is the nominal flow of the pre-collectors used.

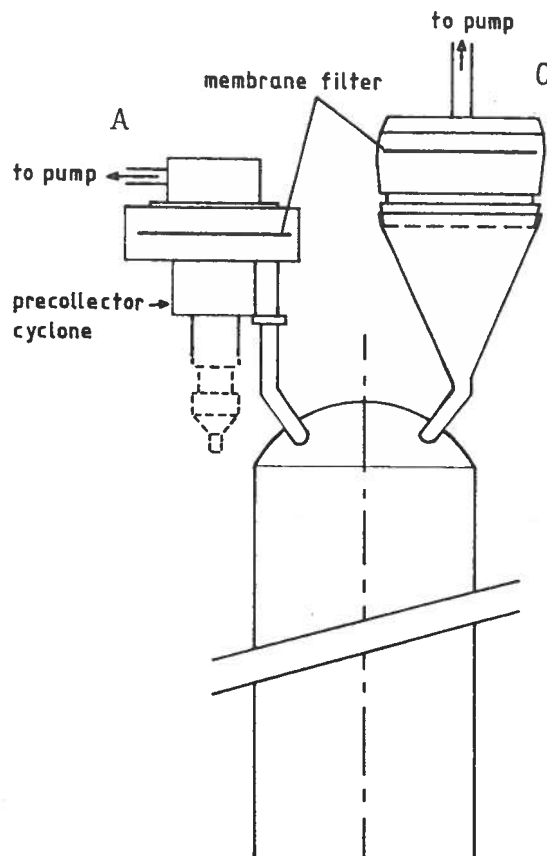


Figure 2. The multi-filter sampler. The figure shows two (A and C) of the four (A,B,C and D) filter holders. They are symmetrically connected to the top of the sampling tube. The air flow through each filter is 1.9 l/min. The filters after the pre-collector cyclones (A and B) collect particles according to the Johannesburg convention (BMRC-curve).

The sampler was intended to give samples for a) gravimetric determination of the ratio between respirable and total fraction, b) analysis of the elemental composition of particles in the two fractions and c) determination of the oxidation state of chromium. The multi-filter sampler was designed for sampling a large fraction of the total aerosol. The upper limit of particle size depends mainly on the sedimentation rate in the inlet tube. Figure 1 shows the theoretical inlet characteristics for sampling in calm air. The mean of the M_A/M_B and M_C/M_D -ratios, where M_A , M_B , M_C and M_D denote the gravimetrically determined mass of particles collected on the filters, were 1.04 ± 0.10 and 0.97 ± 0.14 respectively for 25 collections (the uncertainties are given as one standard deviation of the distribution).

The modified Battelle impactor (8) was used for obtaining a more differentiated determination of the particle size distribution within the respirable fraction. The impactor consists of six impaction stages and one filtration stage. The collection substrate of each impaction stage is a glass plate with a thin ($\sim 30 \mu\text{g}/\text{cm}^2$) polystyrene foil. The foil is coated with a thin layer of paraffine to obtain a sticky surface for reducing bounce-off and re-entrainment effects. The impactor divides the particles into seven particle size fractions with the aerodynamic cut-off diameters 8, 4, 2, 1, 0.5 and $0.25 \mu\text{m}$ respectively (stages 1, 2, 3, 4, 5, 6 and a filter collecting particles with diameters below $0.25 \mu\text{m}$). The impactor was modified by giving an excentric rotation to the collection plates of stages 4 and 5 as described in a personal communication by K.G. Malmqvist, Lund Institute of Technology. Using this modification a thin deposit of particles is achieved, thus minimizing collection losses. The substrates of stages 4 and 5 were the most heavily loaded for all spraying methods used. After sampling the polystyrene foils are floated off the glass plates onto water and collected on aluminum frames for subsequent irradiation with protons in the PIXE -analysis (7). As the impactor in this study was intended for fractionation of the respirable particles, the inlet collection characteristics for coarse particles were not taken into consideration. The first stage, collecting particles larger than $8 \mu\text{m}$ aerodynamic diameter, was used as a pre-collector stage removing coarse particles.

For studying individual particles with transmission electron microscopy, a double filter sampler was used for particle collection. Before sampling the filters are coated with a carbon layer by evaporation. Before examination the filters are again coated with a carbon layer and the filter matrix dissolved in ethylacetate, thus achieving a thin sandwich of carbon with particles in between, suitable for transmission electron microscopy studies.

The samplers were placed 50 to 150 cm above and 50 to 100 cm behind the spraying object with the inlets facing downwards during sampling. The distance between the nozzle of the spray-gun and the object were between 5 and 20 cm. Five collections with each sampler were performed for each spraying method.

The same characterization procedure was also applied to one of the methods during normal production. The samplers used for characterization were placed at different places in the spraying hall. Personal monitoring equipment was carried by two of the operators during spraying. "Millipore monitors" with Millipore filters (Millipore AAWP, 37 mm diameter, 0.8 μm pore size) were carried for two working days during continuous spraying. The filters were changed every other hour. There were other activities in the hall during the personal sampling, however none of them emitting chromium-containing aerosols. The personal sampling was complemented with a stationary "Millipore monitor".

Analytical methods

To gain maximum information from the samples several analytical methods were used. Gravimetical analysis was applied to the filters from the multi-filter sampler by using a Sartorius balance with 1 μg precision and accuracy. Particle Induced X-ray Analysis (PIXE) was used for elemental analysis at the Lund PIXE facility (7). This method gives fast and reliable results for elements heavier than phosphorus. The detection limits are typically below 1 ng. One of the respirable and one of the total samples (B and D) from the multi-filter sampler were washed in distilled water buffered to pH 7.4. A procedure for analysis of chromium described by Bohgard et al. (4) including PIXE, ESCA (13) and spectrophotometry with DPC reagent (1) was used. In the present study AAS, with Perkin-Elmer 403, was used as a complement to the procedure for determining the total fraction of soluble chromium. The sample treatment is described in figure 3. Knowledge about the structure of individual particles is essential for evaluating the results from ESCA, since the results from ESCA are valid only for a thin surface layer (~2 nm) of the sample. Transmission electron microscopy was used to study individual particles before and after washing. It should be noted that "solubility" in this study is defined as the relative amount which is leached out by washing a filter with 25 ml distilled water (at 37°C) buffered to pH 7.4 according to the procedure described by Bohgard et al. (4).

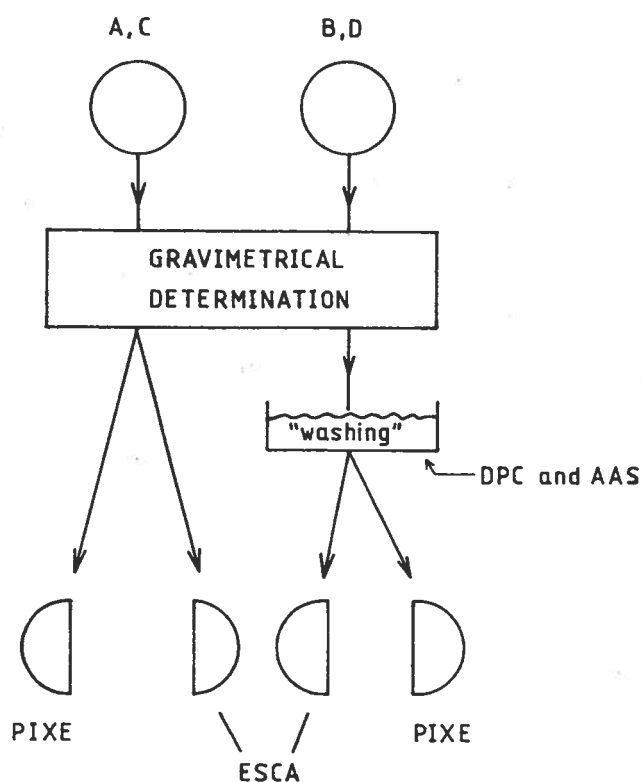


Figure 3. Sample preparation and analytical methods applied to the filters from the multi-filter sampler. A and B are filters with the respirable fraction. C and D are the filters for the "total" aerosol measurements.

Results and discussion

Elemental composition

Table 2 shows the results from the analyses of the filters from the multi-filter sampler. The relative abundancies of the major metals in the aerosols agree with the relative abundancies in the spraying materials. The lower concentrations of the metals in the fume relative to the materials can be explained by the fact that the fumes consist of metal oxides while the spraying materials consist of alloys of the metals. Previous studies of welding aerosols (6) have shown considerable enrichment of certain elements in the fume from the consumable. However, in the present study no such effects were observed for the major elements in thermal spraying operations. The differences in metal concentrations between the total and the

respirable fraction are below 10%. Other elements than those given in table 2 were detected, but due to their low concentrations (<1%), there is a risk that other activities in the workshop may have influenced the results. When the same spraying material was used for different methods (flame spraying and electrical arc spraying with METCOLOY 2, flame spraying and plasma spraying with METCO 44) minor differences in metal composition were seen.

Figure 4 shows the results from the measurements with the personal monitors carried by two workers during the spraying of a paper mill roll. The ratio between chromium and iron concentration is not significantly different from the ratio obtained from the characterization procedure (table 2).

		METHOD					
		1a flame spraying METCOLOY 2	1b flame spraying METCOLOY 2	2 flame spraying METCO 44	3 flame spraying METCOLOY 33	4 electric arc spraying METCOLOY 2	5 plasma spraying METCO 44
METAL % of aerosol mass (total and respirable fraction)	Cr	5.6 ± 0.2	7.3 ± 1	9.4 ± 0.3	7.3 ± 1.7	7.3 ± 0.7	8.2 ± 0.8
	Fe	40 ± 2	44 ± 4	2.8 ± 0.1	6.6 ± 1	41 ± 4	3.2 ± 0.5
	Ni	< 1	< 1	35 ± 2	22.5 ± 3	< 1	30 ± 3
respirable mass/total mass (%)		45 ± 2	39 ± 3	92 ± 1	50 ± 4	83 ± 2	54 ± 2
Cr- fractions in total aerosol	percentage soluble Cr of Cr	7.7 ± 1	3.6 ± 1	3.2 ± 1	13 ± 1	6.4 ± 1	10 ± 1
	percentage soluble Cr(VI) of Cr	6.6 ± 1	3.1 ± 1	1.9 ± 1	10 ± 1	5.1 ± 2	9.7 ± 1
	percentage Cr(VI) of Cr on sample surface	51 ± 5	50 ± 5	< 20	70 ± 5	22 ± 5	63 ± 5
	percentage Cr(VI) of Cr on sample surface after washing	< 15	27 ± 5	< 15	18 ± 5	18 ± 5	20 ± 5
Cr- fractions in res- pirable part of aerosol	percentage soluble Cr of Cr	21 ± 4	6.2 ± 3	2.1 ± 0.2	26 ± 3	9 ± 2	23 ± 2
	percentage soluble Cr(VI) of Cr	18 ± 5	5.8 ± 0.5	1.8 ± 0.2	25 ± 4	9 ± 2	22 ± 2
	percentage Cr(VI) of Cr on sample surface	50 ± 5	52 ± 5	< 20	73 ± 5	22 ± 5	69 ± 5
	percentage Cr(VI) of Cr on sample surface after washing	< 15	22 ± 5	< 15	< 15	17 ± 5	36 ± 5

Table 2

Results from analysis of filters from the multi-filter sampler. Concentrations of metals in the fume, percentage in the respirable fraction and percentage of different fractions of chromium in the total and respirable fraction are given. The uncertainties are given as one standard deviation of the mean from the analysis of five samples.

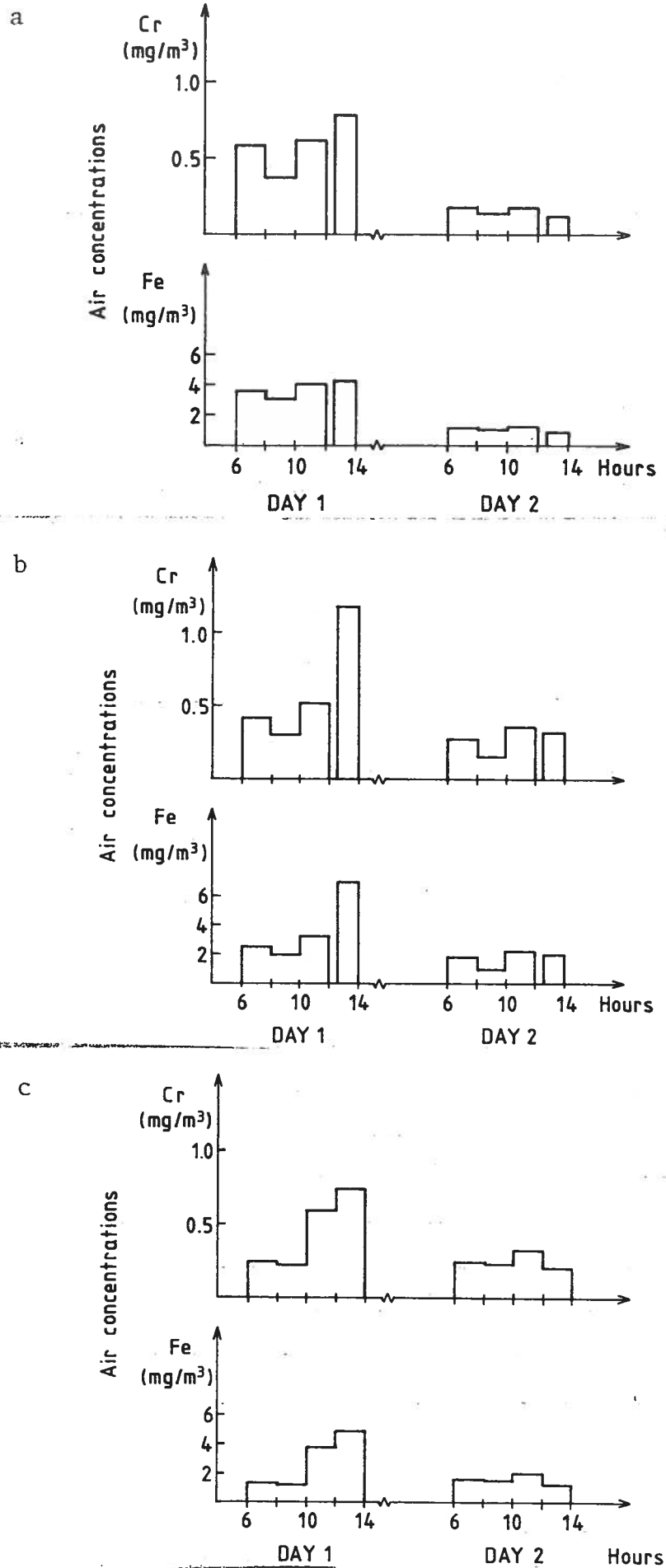


Figure 4. Exposures for chromium and iron obtained from personal sampling (a and b) during two days for two thermal spraying operators coating a paper mill roll by flame spraying with METCOLOY 2. Figure 4c shows the results from a stationary Millipore monitor.

Particle size distribution

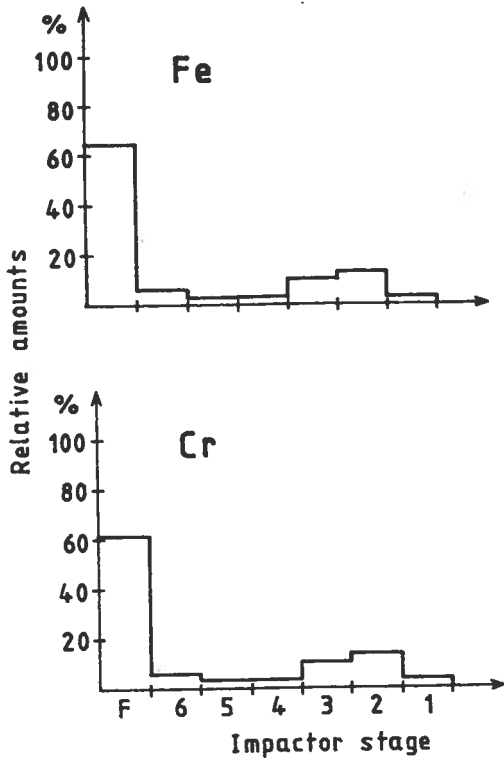
The ratios between the masses of respirable and total fractions are given in table 2. The methods flame spraying with powder and electrical arc spraying give higher percentages in the respirable fraction than the other methods. Figure 5 shows the particle size distributions for fine particles. Significant differences between the methods occur, but for all the five methods the finest particles dominate the mass of the respirable fraction. For method 2 (flame spraying with powder) the mass median aerodynamic diameter of the total aerosol is below $0.5 \mu\text{m}$. For the other methods the mass median aerodynamic diameters of the respirable fractions are below $0.25 \mu\text{m}$.

There are no significant differences ($<5\%$) in metal composition between different particle size fractions. Hence, the metal composition of the collected aerosol is independent of the size characteristics of the sampling inlets.

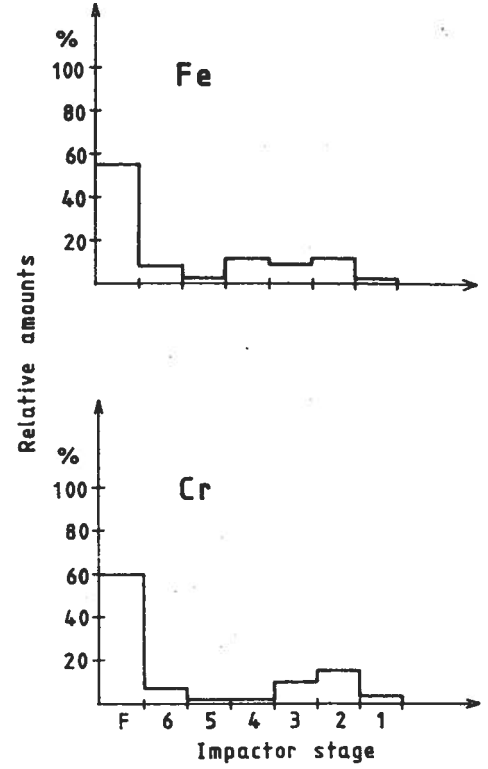
Oxidation state of chromium

Results from the determination of the oxidation state of chromium are given in table 2. There are significant differences in the Cr(VI)-concentration in the respirable and the total fractions. The concentration of Cr(VI) seems to depend on both spraying method and spraying material. The analyses of the soluble part of Cr(VI) were performed within four hours after washing the filters. There is, however, a risk of loss due to the reduction of Cr(VI) to Cr(III). The systematically higher values obtained from the AAS analysis (percentage soluble Cr in table 2) relative to the DPC analysis (percentage soluble Cr(VI) in table 2) may not be significant because of the reduction of Cr(VI) to Cr(III) by other constituents of the aerosol acting as reduction agents during the washing procedure, and the time elapse between washing and analysis,. Hence the soluble fraction of chromium may consist entirely of Cr(VI).

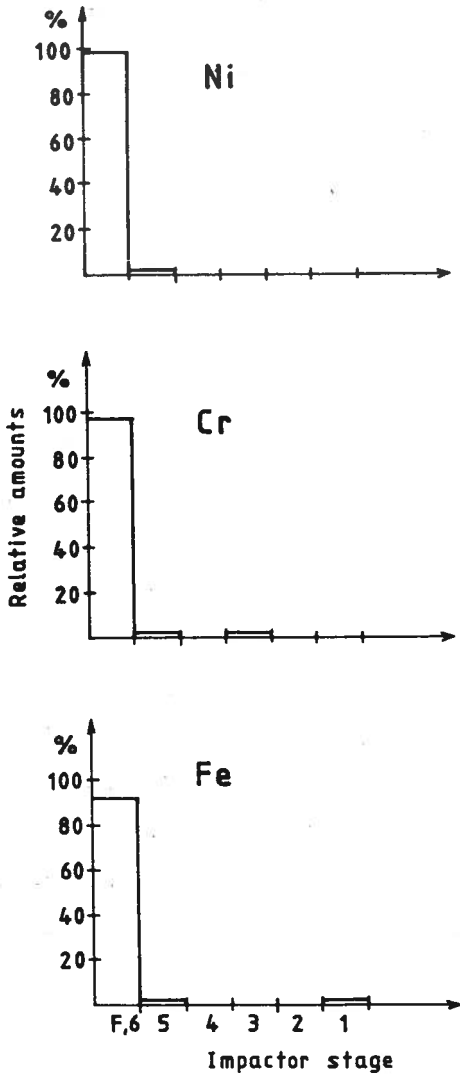
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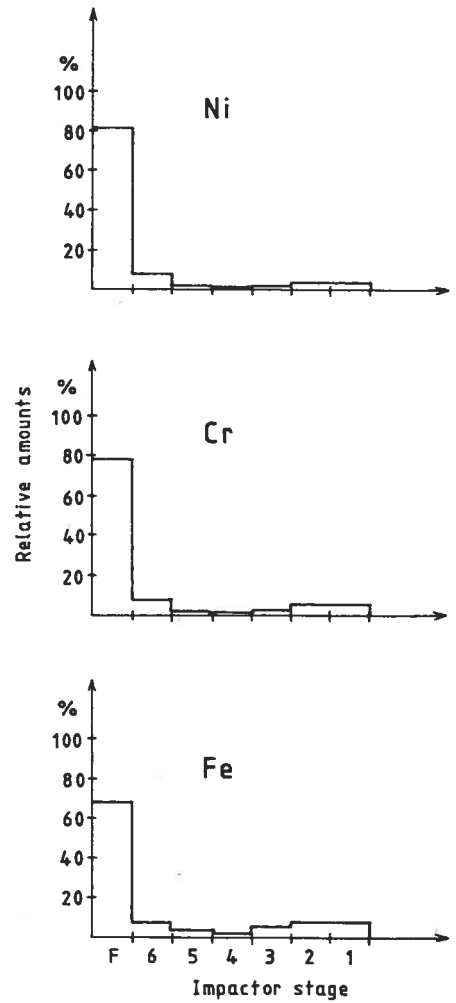
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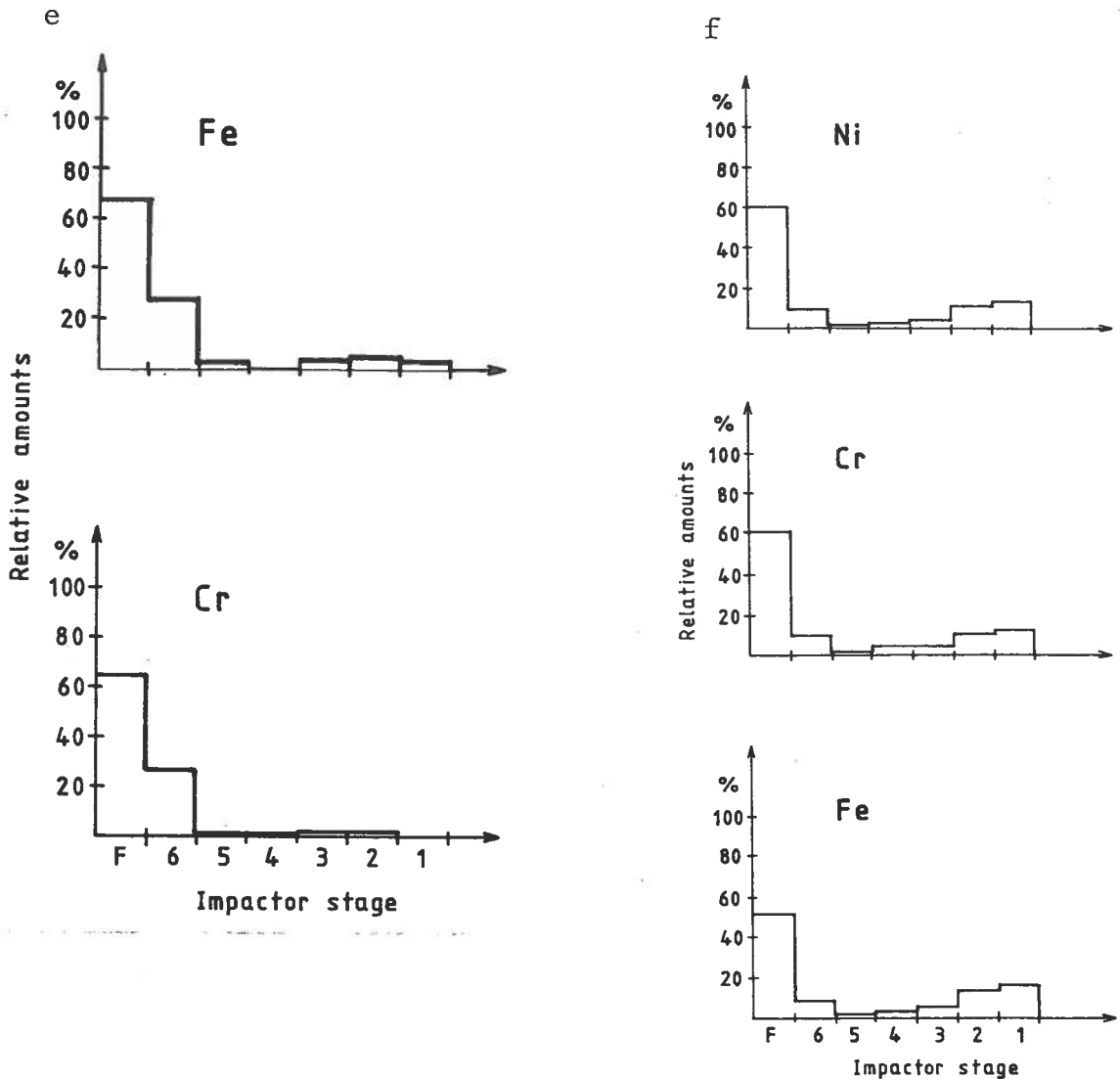


Figure 5. Particle size distributions from impactor measurements. The figures 5a and b show the PIXE-results for chromium and iron from flame spraying with METCOLOY 2 measured in the "sealed room" and during normal production respectively (Methods 1a and 1b). The figures 5 c,d,e and f show results from flame spraying with METCO 44, flame spraying with METCOLOY 33, electric arc spraying with METCOLOY 2 and plasma spraying with METCO 44 respectively. Stage 1 was used as a pre-collector stage collecting particles with aerodynamic diameter larger than $8 \mu\text{m}$. On the stages 2, 3, 4, 5, 6 and the filter (F) are the fractions with aerodynamic diameters D ; $4 < D < 8$, $2 < D < 4$, $1 < D < 2$, $0.5 < D < 1$, $0.25 < D < 0.5$ and $D < 0.25$.

The differences between the concentrations of Cr(VI) in the total and respirable part of the aerosols and the determined mass ratios between respirable and total fractions, indicate that the soluble part of Cr(VI) is mainly in the respirable fraction for all methods. The ESCA-results from unwashed samples do not show significant differences between the total and the respirable fraction. Assuming spherical particles, the total particle area of the respirable fraction exceeds 99% of the total aerosol area according to the determined particle size distributions. Hence by using ESCA, for the spraying methods studied, the respirable fraction dominates the particle area and hence the material analyzed by ESCA. After washing Cr(VI) was still detected for four of the spraying methods. The difference between flame spraying with METCOLOY 2 sampled in the "sealed room" and during normal production conditions should be noted and may be due to the fact that different spraying equipment was used on the two occasions.

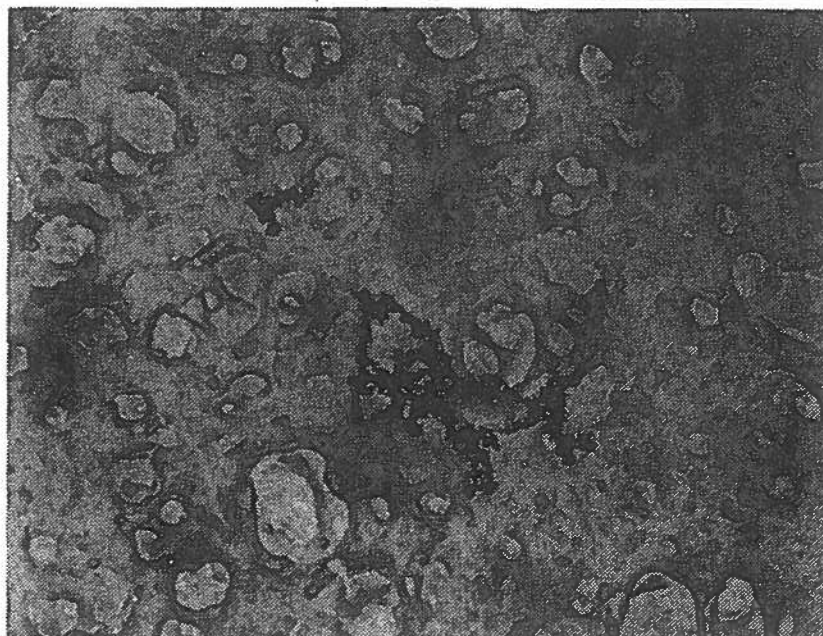
The analytical results of the Cr(VI)-determinations are not unambiguous. There are at least two alternative interpretations of the results; a) Cr(VI) exists only in a surface layer of the particles and is entirely or partly dissolved by the washing procedure. However, our electron microscopy studies do not support the existence of a surface layer with a different composition from that of the bulk material. b) Cr(VI) is homogeneously distributed in the particles. After the washing procedure the surface layer consists of insoluble Cr(III)- and other insoluble metal compounds preventing underlying Cr(VI) from being revealed by washing or by ESCA.

With the current knowledge of the toxicity of Cr(VI)- exposure it is not possible to judge whether the two alternative interpretations are significantly different in terms of health hazard.

Particle structure

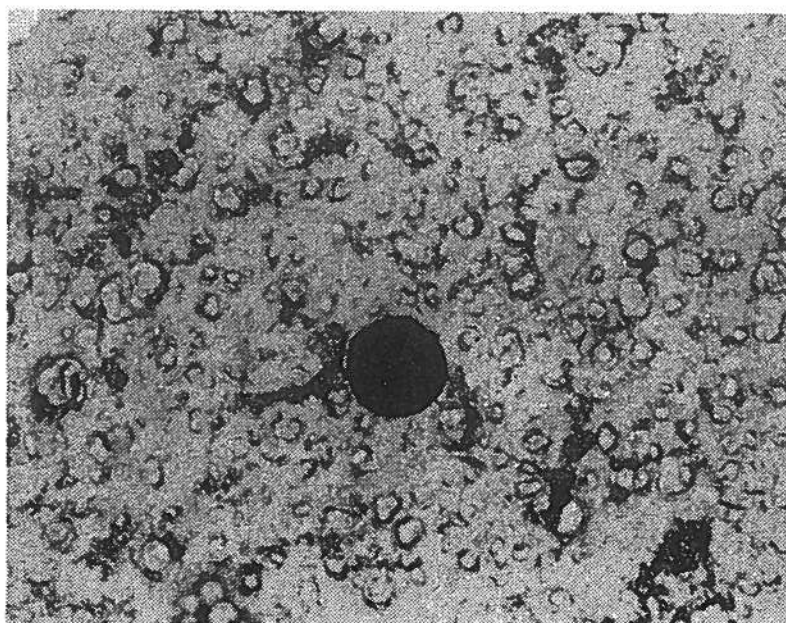
Individual particles have been studied with transmission electron microscopy. Figure 6 shows two micrographs with particles typically seen in these studies. Generally two classes of particles can be seen; coarser spherical particles and finer particles being aggregates of

a)



1 μm

b)



1 μm

Figure 6. Transmission electron micrographs of particles from flame spraying with METCO 44; a) an aggregate of primary particles b) a spherical particle.

small (0.005 - 0.05 μm) primary particles. The aggregates always dominate over the spherical particles in the micrographs. ESCA is valid for 20 to 80% of the volume of the particles in figure 6 a. The observations further support the assumption made above that the total particle area of the respirable fraction exceeds 99% of the total aerosol area.

Conclusions

The relative elemental compositions of the aerosols for the major elements agree with the relative abundances in the spraying materials used and are independent of particle size. Hence for a collected aerosol, the elemental composition is independent of inlet particle size characteristics of the sampler.

Two modes of particles categorized according to aerodynamic diameter can be seen. The respirable fraction consists of 40-90% of the particle mass in the total fraction as defined in this study. The mass median aerodynamic diameter was below 0.5 μm for the respirable fraction, which means that diffusion is the main deposition mechanism of these particles. The particle area in the respirable fraction constitutes more than 99% of the total particle area. A large part of the chromium in a surface layer was found to be hexavalent for four of the methods. Both soluble and less soluble hexavalent chromium were detected. The oxidation state of chromium seems to depend on both spraying method and spraying material.

When the aerosol emission was characterized during normal production, it was found that the aerosol was not significantly different with respect to metal content and particle size distribution.

In thermal spraying, workers may be exposed to high concentrations of aerosols emitted from the spraying process. When using materials containing chromium and nickel, compounds of those metals can constitute the major part of the aerosol. A considerable part of the chromium can be hexavalent. The results imply that a high degree of

precaution has to be taken when working with these processes.

The characterization procedure used in this study has been shown to give detailed data about the aerosol emission from methods of thermal spraying. The knowledge of how the health hazard potential depends on the aerosol characteristics as determined in this work is not sufficient for ranking the five methods studied from a hygienic point of view. However the sampling and analytical techniques used give results of high significance for parameters of considerable interest in health effect assessments and for toxicological and epidemiological studies.

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