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Characterization of Wood Dust from Furniture by Scanning Electron Microscopy and Energy-dispersive X-ray Analysis

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Abstract: Study characterized and analyzed form factor, elementary composition and particle size of wood dust, in order to understand its harmful health effects on carpenters in Quindío (Colombia). Once particle characteristics (size distributions, aerodynamic equivalent diameter (D_a), elemental composition and shape factors) were analyzed, particles were then characterized via scanning electron microscopy (SEM) in conjunction with energy dispersive X-ray analysis (EDXRA). SEM analysis of particulate matter showed: 1) cone-shaped particle ranged from 2.09 to 48.79 μm D_a; 2) rectangular prism-shaped particle from 2.47 to 72.9 μm D_a; 3) cylindrically-shaped particle from 2.5 to 48.79 μm D_a; and 4) spherically-shaped particle from 2.61 to 51.93 μm D_a. EDXRA reveals presence of chemical elements from paints and varnishes such as Ca, K, Na and Cr. SEM/EDXRA contributes in a significant manner to the morphological characterization of wood dust. It is obvious that the type of particles sampled is a complex function of shapes and sizes of particles. Thus, it is important to investigate the influence of particles characteristics, morphology, shapes and D_a that may affect the health of carpenters in Quindío.

Key words: Aerodynamic diameter, Shape factor, Softwoods hardwoods, Wood dust

Introduction

In the mid-1990s, the International Organization for Standardization (ISO), the European Committee for Standardization (CEN), and the American Conference of Governmental Industrial Hygienists (ACGIH) agreed definitions for inhalable, thoracic and respirable particles that penetrate and/or settle in various compartments of the human respiratory tract[1]. In the following, the particulate material is referred to wood dust from the process of making wood furniture.

Particulate matter from wood dust is a complex mixture of cellulose, mainly, polyoses and lignin. Wood used in the manufacture of furniture can be hard (from non-conifers) and soft (from conifers). During the manufacturing process and handling of timber, plywood and chipboard panels sawdust is generated, which contains tiny particles also known as chips. A chip is a piece of waste material in the form of a spiral or curved blade that is drawn through brush, sander or other tools when working on wood or metal in the form of dispersion. Powder formed by dispersion tends to be more irregular in shape and size, than aerosols formed for instance by condensation of a gas, because nucleation, coagulation and growth occurs in small regions of fluid under similar conditions. Machine tools emit particles, whose sizes vary depending on the tool from particles larger than 100 μm to some millimeters and particles below 10 μm, with a speed of tens of meters per second. Wood particles with aerodynamic diameters ranging between 10–100 μm are the greatest concern in the study of dis-

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eases of upper respiratory tract, causing nasal cancer or diseases associated with wood dust exposure[2–4].

There are several methods for the characterization of particulate matter. The principal method is based on the gravimetric approach of measuring the mass gain of a given particulate size fraction collected on a filter per unit volume of air sampled. Electron beam techniques such as SEM and X-ray microanalysis with wavelength- or energy dispersive X-ray analyzers are ideally suited for the analysis of such samples. The analytical information of SEM/EDXRA can be combined with image processing and analysis techniques to provide statistical description of particles with respect to their chemical composition and morphology[5].

Soft woods from conifers are irritants, allergens, and with time they may give rise to occupational asthma and chronic obstructive pulmonary disease (COPD)[6]. Hardwood dust has been associated with several types of cancer, including the nasal cavity, lung and gastrointestinal tract cancers, and Hodgkin's disease[6, 7]. Long particle with pores are typical from conifers. The majority of non-coniferous particle are short and thick-walled. It is suspected that morphological characteristics of the particle from conifers make them more aggressive when deposited at the soft tissues of the nose and lungs that particle from non-conifers, although the effects of the latter are more lethal[6, 7].

Of all these diseases, the most serious epidemiological evidence is cancer of the nasal cavity. In 1964, IARC stated that wood dust is carcinogenic[7–10]. In 1995, it again confirmed to wood dust as a human carcinogen and reported increased nasal cavity cancer, paranasal sinus cancer and lung cancer in workers exposed to dust wood in the U.S.[11–13]. A case study in the U.S. carried out by the National Cancer Institute following the SEER programme reported that occupational exposure to formaldehyde (but not to wood dust) increases the risk of nasopharyngeal cancer which, coupled with exposure to hardwood dust and unhealthy habits such as tobacco consumption, contribute to the development of cancer faster. The lapse of time between first occupational exposure to wood dust and the development of nasal adenocarcinoma is 40 yr (in a range of 7–70 yr)[13]. TLV for wood dust should not exceed 5 mg/m^3 [14]. Carcinogenic potential of wood dust is biologically plausible because of the components that enter the lung[15, 16].

The respiratory tract (RT) is divided into three main regions: the first upper region includes the nose, mouth, nasopharynx, oropharynx, epiglottis and larynx; the second region includes the lower airways of the trachea through the terminal bronchioles, and the third region includes the pulmonary respiratory bronchioles, alveolar ducts, alveolar sacs and alveoli. Probability of deposition of particles in the RT depends on the aerodynamic diameter. Sizes of particles deposited correspond to 10–50 μm in the upper region, 5–10 μm in the lower region, 2.5–5 μm in lungs and < 2.5 μm in the alveolar region[1].

In Colombia, wood dust is considered one of the most frequent causes generating asthma[17].

Among the most important economic activities of the department of Quindío (Colombia) include agriculture, livestock, mining, tourism and exploitation of wood. In 2007 there have been approximately 84 companies with about 6,500 workers from manufacture to sale of furniture and other wood products. Most of the furniture made by these companies is handmade[18].

Main goal of this work is to characterize the elemental composition and morphology of the particulate matter collected at furniture workshops in Quindío (Colombia), through SEM/EDXRA analysis.

This work aims to contribute to knowledge of the morphological characteristics of particulate matter from wood dust, which may allow to relate it to the influence of wood dust on the health of workers in manufacturing wooden furniture, as well as to introduce improvements to their working conditions and respiratory protection devices.

Methods

The present study is based on descriptive statistics[19]. Expressions commonly used as measures of dispersion of a distribution of particles are: the standard deviation (σ) and geometric standard deviation (DGE or σg). For computing these statistical functions we have used the program Microsoft Excel®.

Companies selected and sampling site

The authors used a convenience sample of ten companies in Quindío identified upon recommendations from members of the Chemical Risks research line at the University of Quindío[18]. The selected companies had similar tasks in the manufacture of a variety of wood products, including store fixture, parts for doors and windows, cabinets, beds and home furniture.

Two workplaces were selected for air sampling (sanding and brushing) in each of the carpenter's shops that agreed to participate. In these two work areas is where the smallest particles are supposedly generated by sanding wood[20, 21]. Each was visited first to ascertain the work conditions, job types, types of wood and likely exposure levels, and to introduce the factory manager or safety supervisor to the type of equipment that would be used in the study.

Sample size and sampling strategy

It was decided to mount the sampling equipment (pumps and sampling heads) according to the analytical method used (standard NIOSH 500 "Particulates not otherwise regulated, total[22]"). Because the method of analysis by observation of individual particles, only a small quantity of dust can be collected to ensure that individual particles do not overlap. Four samples were taken in each company (two in sanding and two in brushing areas). Forty air samples with particulate matter were collected in total.

One personal sample per worker was taken during at least 70 min and a flow rate of 1.5 l/min. In order to characterize the particles in a wide range of sizes, we used an air sampler pump brand GilAir5 Gilian Multi Fol. No. 800519 with Sensidyne gauge type TM 2 803024B Filian Gilibrator, Bubble Generator Rang 20-CC 6LPM P7N 800,286. Hydrophobic filters used with a pore size of the membrane between 2 to 5.0 μm of 37 mm polyvinyl chloride (PVC) side open. Additionally, we used a desiccator with silica gel and an analytical balance Gibertini E50 S-calibrated for a minimum weight of 0.001 g.

Once collected, samples were sent from the University of Quindío to the UPC (Spain) for analyzing the particles. Four samples per company were taken (two of them in belt sanding and the other two in manual brushing areas), i.e., 40 samples in total. The average occupational exposure (AOE) at each company was estimated. Then, those samples that exceeded the theoretical TLV (5 mg/m^3) were chosen five companies (they corresponded to 50% of companies). The selected samples were analyzed by electron microscopy and we discarded those samples exceeding 50 μm particle size. Only two samples had particles of less than 50 μm. Henceforth, the two samples selected are called #1 and #2. These two samples had been taken from the belt sanding (#1) and manual brushing (#2) areas.

Sample preparation for SEM/EDXRA Analysis

The samples were prepared with gold coating under vacuum to ensure image quality (due to the excellent electrical conductivity of gold) and examined by digital image scanning microscopy using a JMS microscope.

SEM/EDXRA analysis

The characterization of particle morphology and composition has been analyzed by SEM. A scanning microscope JEOL JSM 6400 (JMS) was used to observe shapes of particles. Moreover, an Energy-Dispersive X-Ray Analysis (EDXRA) was applied to analyze their surface.

Characterization of wood dust

Morphology: Shape, size, texture and appearance are differentiating elements in the microscopic world that are intuitively assumed by humans. In the case of wood dust, particles are generally large and rectangular-prism shaped[2], although in the present study percentage of predominant shapes was not computed. The descriptive statistical analysis of the particles of the two group of samples to quantify the average occupational exposure concentrations was expressed by the following statistical variables: the geometric mean (GM), standard deviation (SD) and geometric standard deviation (GSD)[19].

Shape factor and aerodynamic equivalent diameter (D_a): The parameter conventionally used to quantify the particle shape is the shape factor, defined as the ratio between the surface of the sphere of same volume as the particle and the particle surface. This value can lie between 0 and 1, and gives a idea of the sphericity of the particles. Wood particles are irregularly shaped and can be classified as non-spherical. Their shape corresponds to, for instance, particle, coils, or chips, so it can be deduced that these systems are rarely fluidized[23].

The form factor for individual particles was calculated by the following equation[2, 24]:

$$K_D = 0.864\left[\frac{1}{3}+\frac{2}{3}\left(\frac{d_s}{d_n}\right)+0.0739\left(\frac{d_{\max}}{d_n}\right)+0.0108(AR)\right] \quad (1)$$

where K_D is the dynamic shape factor, d_s is the diameter of a sphere with surface area equal to that of the object, d_n is the diameter of a sphere with projected area equal to that of the object, d_{max} is the maximum dimension of the object measured in the direction of motion, AR is the aspect ratio, the ratio of the longest to the shortest dimension of the object in the area projected normal to the direction of motion[24].

The equivalent volume diameters were converted to aerodynamic equivalent diameters by correcting for density and shape factor using equation 2[2, 25]:

$$\mathrm{D_a} = \mathrm{D_s}\sqrt{(\rho\overline{\mathrm{K}})} \quad (2)$$

where D_a is the aerodynamic equivalent diameter, D_s is the volume equivalent diameter, ρ is the particle density, and $\overline{K}$ is the dynamic shape factor averaged for all orientations. Densities were obtained from *The Particle Atlas* averaged a density of both types of wood as 1.5[25]. This value has been taken as reference of density in the analysis of D_a. The geometric expressions (cylinder, cone, rectangular prism and sphere) are taken from *The Particle Atlas*[26].

Particle shapes and D_s were obtained from SEM images. Characteristic geometrical dimensions of each particle shape (cylinder, cone, rectangular prism and sphere) determining D_s were measured. Then, D_a values were estimated by using equation (2). Measuring instrument was connected to a personal computer with the software Omnimet Enterprise to visualize and store the results on screen as they were getting the data. Average grain size was determined by the intercept method according to ASTME-112[27]. This test method is used to estimate the average size of grain of glass particles, cells, metallic and nonmetallic materials with a unimodal lognormal distribution of grain areas, diameters and lengths. The volume can not be determined in this test. The comparison method can be used if the structure of the material approaches the appearance of one of the standard comparison charts. The intercept and planimetric methods (or Jeffries) are always applicable for determining average grain size. However, comparison charts can not be used for the measurement of individual grains.

The Omnimet Enterprise software was used for the implementation of the standard. This software is an image analysis program that also includes the option to view and capture digital images. It is mainly used to determine the grain size and percentage of individual entities identified. The calculations are based on the amount of pixels at the original image that are painted in red as shown in Fig. 1. From the micrographs taken, it selects particles trapped in the mesh and transform

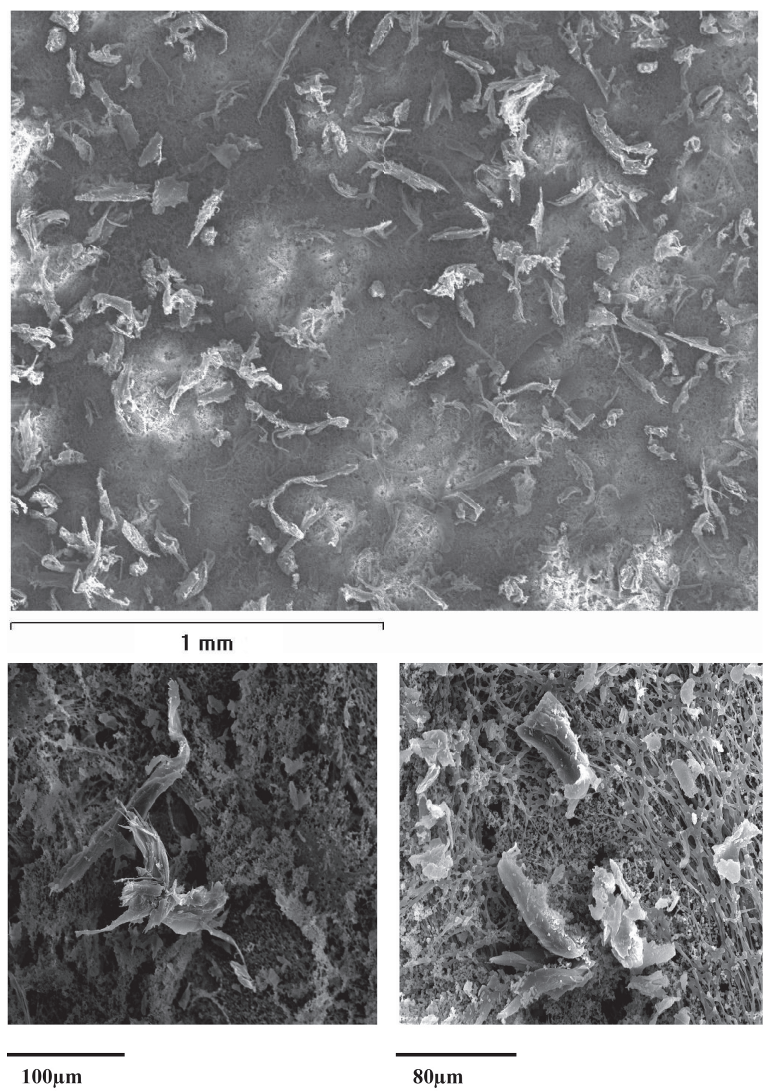

Fig. 1. Series of photomicrographs of particles of wood dust collected by electron microscopy of 1 mm, 100 and 80 μm.

It can be observed some long, woody or torn, thin and spiral particle, which are typical from conifers, and short, thick and compact particle, typical from non-conifers.

them into a bitmap (shaded in red), and determines the particle size using the ASTM-E112 routine[27)].

Concentration of particles: Both mass concentration, i.e. mass of particulate matter per unit volume of aerosol, usually expressed as g/m^3, mg/m^3 or μg/m^3, and number of particles per mm^2 settled on the surface of a PVC filter, were estimated[9)].

Elemental chemical composition of particles (Elemental analysis EDXRA): Analysis of the elemental components of paints and varnishes of particles in the paint section. Among these substances, there is chromium (VI) oxide (CrO_3). Compounds of chromium (VI) are oxidants and carcinogens[28, 29)]. Most of chromium (VI) irritate eyes, skin and mucous membranes.

Results

Sampling

Work areas with the highest measured dust concentrations have been those for sanding that use belt, stationary or hand tools, and also those for sawing, assembly, and manual or vacuum cleaning of dust. Levels of wood dust increased when operated belt sanders[16)]. The samples were taken in these areas with air sampling pumps, by following the above-mentioned analytical method 0500 of NIOSH.

Average occupational exposures of the areas visited were: planning and sanding: 62.8 mg/m^3; immunization: 17.1 mg/m^3; painting: 26.7 mg/m^3; cutting: 31.9 mg/m^3, compared to TLV: 5 mg/m^3 [14)].

Particle concentration for #1 was 77.14 mg/m^3 (SD=13.05, GM=0.60, GSD=1.81) and for #2 was 12.38 mg/m^3 (SD=14.77, GM=0.73, GSD=2.08). Statistics data were taken from the images taken to the filters: in total, 87 images, 9 spectra and 37 images treated through ASTM. A filter area of 105 mm^2 was used for analysis by SEM in both samples and, then, the approximate number, size, shape and D_a of particles were estimated. For sample #1: approx. 118 particles (35 images, 14 ASTM images), For sample #2: approx. 338 particles (52 images, 23 ASTM images).

Particles of #1 had their D_a comprised between 4.34 and 63.43 μm; D_a range for those of #2 was 2.47–72.90 μm. The average D_a was 22.7 and 14.3 μm, for #1 and #2, respectively. To calculate the density of particles in the filter, first, pictures were taken through SEM in some areas of the filter, randomly selected from those areas containing particles. Second, numbers of particles in those areas were computed by means of the ASTM-E112 method. Finally, the amount of particles in the whole filter area was calculated extrapolating these numbers linearly. Taking into account that each filter had a total area of 1,075.2 mm^2 and by assuming a constant density, the number of particles in the #1 was approximately 336,310 particles per cm^2 and in the #2 was around 275,700 particles/cm^2.

Characterization of particulate matter using SEM/EDXRA

The particles collected of both samples (#1 and #2) had different shapes and aerodynamic diameters ranging from 2.47 to 72.90 μm. Larger sizes usually belong to particles having pointed, conical, spherical, rectangular prism, or slender cylindrical shape, i.e. streamlined shapes. Figure 1 shows a series of photomicrographs at a scale of 1 mm that allows to see a wide variety of irregular particles in the sample: long and short, flat, taped, torn or frayed particle (it is noticeable the tearing of the particle allegedly due to the mechanical friction of belt sanders). To the naked eye can not see what type of timber is, although it is sure that several types of wood were used in furniture making. By increasing the scale between 80 and 100 μm it is a little clearer what type of timber may be made of, in which some long, irregular, spiral and tear particle can be seen. They are typical from conifers. In addition, there are short, flat and compact particle, which are characteristic of non-conifers.

From the data obtained from scanning images with the JMS microscope and the grain size method ASTM E-112, a descriptive statistical analysis of the particles was carried out. Figure 2 shows the technique used to determine the grain size of a wood particle.

In this study, the shapes selected to find the form factor and D_a of particles have been: rectangular prisms, spheres, cones and cylinders, as shown in Fig. 3.

Rectangular prisms: particle size was between 32.92 and 72.90 μm D_a (#1), and from 2.47 to 25.10 μm D_a (#2).

Spheres: D_a ranged between 9.08 and 51.93 μm (#1), and from 2.61 to 10.40 μm (#2).

Cones: D_a ranged between 32.92 and 48.79 μm (#1), and from 2.09 to 16.25 μm (#2).

Cylinders: D_a ranged between 32.92 and 48.79 μm (#1), and from 2.5 to 16.25 μm (#2).

Some particles with crystal shape were also observed, maybe due to the deck of paint or other chemicals that are added to the timber. The spectrum ininin Fig. 4 shows the presence of C and O in the scale of k_α 4.4000 and 2.3708, respectively, which are the main elements of the cellulose (wood). The Au peak (k_α 9.711) that can be seen in the spectrum is the cover of the sample for EDXRA scanning and the presence of Cl (k_α 2.622) is maybe due to the PVC filter or to an immunizing organochlorine compound. Figure 4 shows a particle taken in the sector of second sanding (furnitures

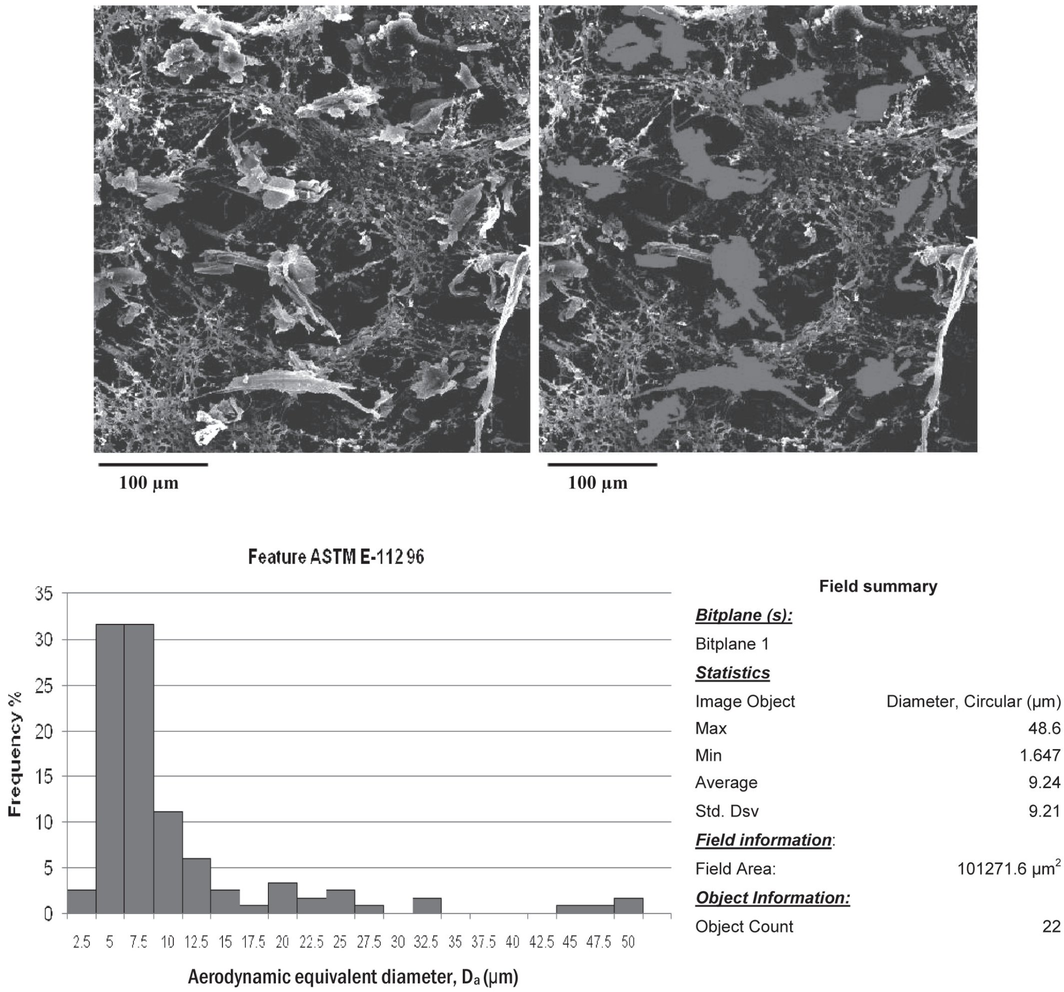

Fig. 2. Series of photomicrographs of particles of wood dust collected by electron microscopy JEM of 100 μm. It can be observed that the photomicrograph on the left was treated with the help of OmniMet Enterprise software, calibration is 0.5525 μm/pixel. The particles that were trapped in the mesh are shaded in red into a bitmap; their particle size was determined following the ASTM-E112 routine.

are sanded and painted again for a finished painting). Spectrum establishes the presence of Ca, K and Cr, which may be characteristics of paints and varnishes, since the samples were taken after a second sanding and were already painted. The SEM analysis was done by a SEM expert. He checked more than 12 points to check the homogeneity of the analysis.

Discussion

Presence of particles seen in Fig. 1 suggests that both types of wood (hard and soft) were used in the carpenter's shops. This sample was taken from the section of second sanding (once a piece of furniture has been painted, it is re-sanded and then painted again to give a better finish to wood). Once painted it is difficult to appreciate what type of particle is (conifer or non-conifer), which creates a degree of uncertainty in health risk.

Pictures of samples taken by optical and electronic microscopy show different and complex shapes of particles of wood dust. However, up to four primary shapes have been identified, from which other more complex shapes could be created by combining these elementary geometries. It should be emphasized that D_a obtained for the cylindrical shape (1.5 to 12.08 μm) lies in the range of particle sizes that could affect the RT.

It is worth noting the small size of these particles ($< 20\,\mu$m) which, coupled with chemicals or paint immunizing added, may pose a greater risk to the health of carpenters. Particles whose size is below 3 μm are a high risk for lower airways (lung tissue and alveoli). Moreover, these particles, analyzed by EDXRA, confirmed the presence of toxic substances as chrome.

Due to the large average particle size, it is likely that the deposition of particles in the human respiratory sys-

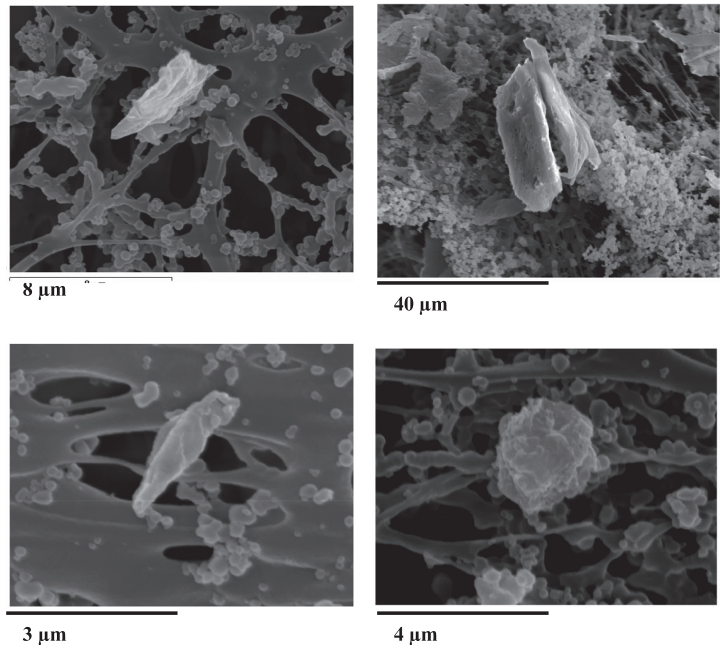

Fig. 3. Series of photomicrographs of wood dust particles obtained by electron microscopy approaching (from left to right and from up to bottom): irregular conical, rectangular, cylindrical and spherical shape.

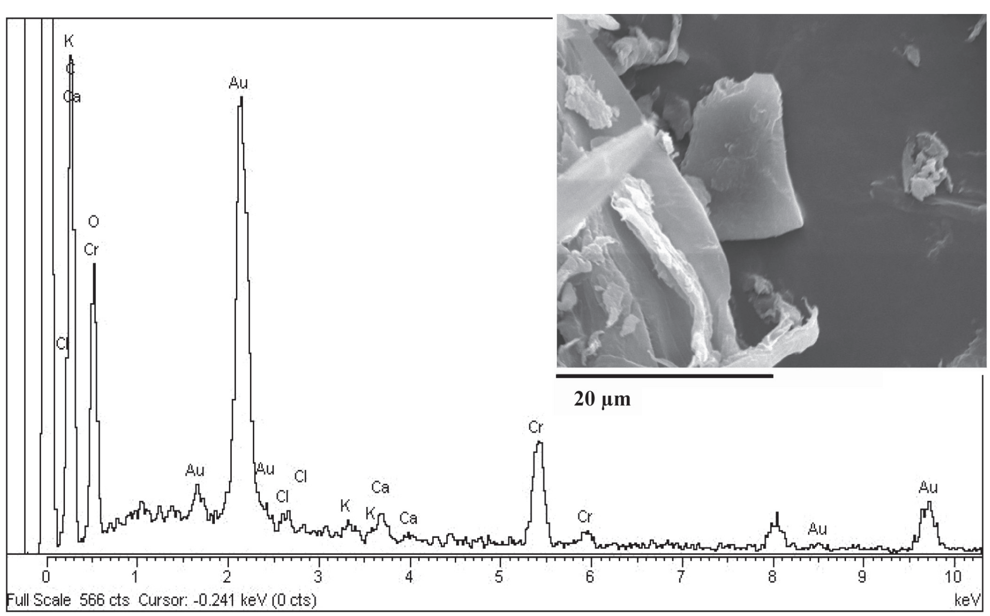

Fig. 4. Microphotography of a particle of wood dust obtained by electron microscopy with its own spectrum indicating the presence of Ca, K and Cr.

tem occurs in the upper respiratory tract.

Deposition of particles in RT, according to the average D_a, could affect to the upper region: nose, mouth, nasopharynx, oropharynx, larynx and epiglottis. Particles with D_a below $5\,\mu$m, could affect the bottom region of the RT, with potential to generate problems or lung diseases.

Results from the SEM/EDXRA analysis show that the sample preparation method developed in this research is applicable to the analysis of wood dust collected. The characterization of wood dust samples yields information that can help epidemiologists and toxicologists to understand the causes of respiratory illnesses. In some cases, the preliminary SEM/EDXRA analysis of particle matter filters can determine whether a more elaborate analysis of highly toxic metals or organic contaminants is necessary or not. In this respect, the spectra showed the presence of relevant chemicals, mainly components of varnishes and paints (such as CrO_3, $PbCrO_4$ or $K_2Cr_2O_7$)[28], most of them dangerous to health. Chromium is considered carcinogenic, especially with valence +6[29]. But in this study we could not confirm that it derives from varnish or paint.

This SEM/EDXRA research contributes to a significant an important analysis with regard to morphological characterization of wood dust. It is obvious that the type of particles sampled is a complex function of shapes and sizes of the particles. Thus, it is important to investigate the influence of particles characteristics; morphology, shape and D_a that can to affect in the health of carpenters at Quindío.

The difference between particle from conifers and from non-conifers is not always easy. Long particle with pores are usually from conifers. Most of non-coniferous particle are short and thick-walled. Wood particles are irregularly shaped and can be classified as non-spherical. Their polymorphic form makes it difficult to focus on a single feature. Morphological characteristics of the particle from conifers make us to think that these are more aggressive after being settled on soft tissues of the nose and lungs than particle from non-conifers.

We found in this study that, depending on the type of tool (in this case, belt sanders), very small particles ($< 20\,\mu$m) can be emitted. This is an issue in protecting the health of workers, since the usual respiratory protective of disposable masks can be ineffective in retaining particles of that size. However, it is important tests as to the removal of particulate conventional disposable masks used by carpenters. An equally serious effect in the finishing of wood is the presence of some chemical elements from paints and varnishes such as Cr considered dangerous to the health of workers.

The concentration of particulate material was above the TLV recommended for wood dust (15 times more in #1 and 2 times more in #2) which confirms the little control of particulate matter in the work environment, increasing considerably the likelihood of becoming ill. This shows the weakness of epidemiological surveillance programs for particulate matter of the companies visited. It also could explain the bad health of workers, because previous studies have pointed to problems of cough, hoarseness, rhinitis and frequent flues, very common after usual exposure to wood dust without proper respiratory protection.

Meeting new form factors of wood particles in the future could help in implementing new innovations on sampling wood dust, in the design of tissues for carpenters' respiratory protection, and above all, to investigate more thoroughly the behavior of particles according to their morphology in the human respiratory tract.

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