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Particle emissions study from tire sample with nano-silver tracer from different steps of its life cycle. A new approach to trace emissions of tire microparticles

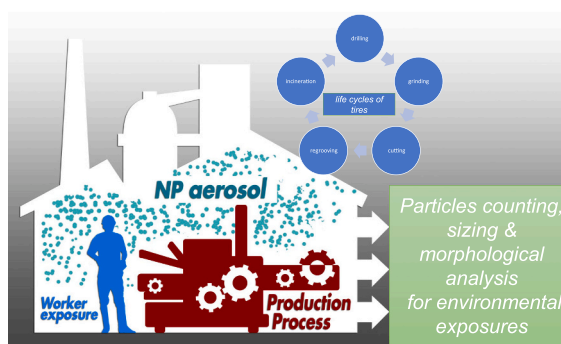
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HIGHLIGHTS

- Laboratory simulations of different steps of tire life cycle was performed.
- The characterization methods are focused on μ plastic particle emissions from tire's workplace.
- Nano-Ag tracer was developed to identify the main exposure steps.
- Increasing average counting is observed as follows: regrooving, drilling, grinding and cutting.

GRAPHICAL ABSTRACT



ARTICLE INFO

Editor: Jianmin Chen

ABSTRACT

Emissions due to tires retread/repair and incineration are a cause of concern owing to the presence of nanoparticles in the products. The assessment exposure to humans hereto related is a challenge in an environmental context. The first object of this work is to develop a method to characterize the emission sources using online (counting and sizing) and offline measurements. The second objective of this work is to apply this new metrological strategy to two tire samples. One of these samples contains nanoparticles made of silver to trace the nanoparticle emissions and assess the performance of the characterization method presented here.

Retread/repair testing involves different tasks *i.e.*, drilling, grinding, cutting and regrooving. Testing evidence that emissions adding dried droplets or carbon films and irregular-shaped particles are discernible in microscopy. Carbon and oxygen, and to a lesser extent silicon and sulphur are the most common chemical elements. The compound containing nano-Ag emitted the same amount or sometimes less nanoparticles (*i.e.*, drilling, cutting and regrooving) than the compound having no nano-Ag. Silver embedded in the rubber matrix was found at low concentration in irregular-shaped particles originating from one of these two samples and highlights the approach interest.

Incineration testing were carried out in a laboratory scale tubular furnace. The generated aerosol is dominated by soot whatever the sample. Few particles containing ZnO or silver are identified in residue samples. A STEM/

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EDX mapping analysis exhibits few nanoscale objects from incineration residue containing free nanosized silver particles and separated from the large silver object.

1. Introduction

Due to a broad evolution in societal sustainability requirements, with respect to non-exhaust car emissions (Philippe et al., 2020) tire producers need to modify their formulations. The collaborative report by OECD (Organization for Economic Co-operation and Development) “Nanotechnology and Tyres – Greening Industry and Transport”, highlights benefits of development and use of new nanomaterials in tires and the interest of implementing best practices to use new nanomaterials in the tire life cycle (OECD, 2014). The evolution of the formulations involves mainly the change of tire compound and specially the nanoparticle fillers (OECD, 2014). These modifications are occasionally inspired by powder industry (García-Triñanes et al., 2010; Morgeneyer et al., 2008; Shandilya et al., 2014a). On one hand this substitution could lead to improved performances such as reducing tire wear and rolling resistance. The use of new nanomaterial fillers will so allow reaching new tire types with lower mass and reducing greenhouse gas (GHG). On the other hand, the risks associated with new nanoparticle could implicate harmful releases for workers or environment and need to be investigated.

A large range of chemicals can be utilized in vehicle tires, many different materials can be found like synthetic or natural rubber, carbon black, steel cord for belts, polyester and nylon fiber, steel bead wire and many different chemicals, waxes, oils, pigments, silica and clays (Evans and Evans, 2006). For commercial reasons it is unusual for tire manufacturers to release exact details of tire. The compounds of the emitted particles are mainly driven by the tire compositions. But, compared to the original tire tread, the chemical composition and characteristics of the particles generated are often modified because of the forces engaged during the stress but also the contact able to alter the emitted particles (Panko et al., 2013).

The use of carbon black and silica nanomaterials as fillers in tires is well documented (Vilgis et al., 2009). Their replacement by new nanomaterial fillers such as CNT (Iijima, 1991) or graphene (Novoselov et al., 2005) or bio-based compounds (Bélanger et al., 2023) is under study. In this framework, the characterization of releases in the tire life cycle is an aspect which has been poorly examined due to the meteorological challenge of emitted nanoparticle emissions.

Many studies focus on nanoparticle releases of nanoparticle embedded in coating matrix when performing abrasion (Shandilya et al., 2014b; Shandilya et al., 2015a; Shandilya et al., 2015b; Vorbau et al., 2009), sanding (Gohler et al., 2010; Koponen et al., 2011) or aging (Clar et al., 2019; Olabarrieta et al., 2012; Sung et al., 2015). Some authors proposed a list of releases from nanomaterials under stress (Koivisto et al., 2017). However, they could not succeed to establish a global rule able to describe the nature and the amount of the nanoparticle releases due to the diverse stresses observed on nanomaterials (Koivisto et al., 2017). There is information which has been deduced by experimental results. The literature on exposure assessments highlights that free releases from nanomaterial solicitations are considered as unusual (Koivisto et al., 2017; Schlagenhauf et al., 2015). A consensus on emissivity studies of nanomaterial considers essential to mimic the stress scenario under well controlled conditions and sample released fragments using harmonized protocols (Shandilya et al., 2014c).

To our knowledge, study of Waquier et al. is the unique work devoted to simulation of repair / retread tasks by tire abrasion (Waquier et al., 2020). This situation is due to 1/ the composite nature of the nanomaterial, 2/ the problems caused by simulating the operations involved in the recycling process, 3/ the difficulties to characterize nanoparticle emissions from solicited material.

The present study on the tire production is focused on recycling

approach related with repair/retread and incineration of tires. Indeed, the part of the tire life cycle have been poorly studied and need to be more documented. The nanosafety must consider the different tasks involved in the tire workplace framework. The two most crucial characteristics of a nanoparticle exposure are the amount and the nature of the releases. The concentration of nanoparticles emitted during the repair/retread and incineration of tires is complicated by the type of material used in the tire manufacturing. Tires are made of composites which are mainly constituted by rubber and nanoparticles and will certainly produce nanocomposites under stress with similar composition.

The retread / repair process *i.e.*, retread (affixing a new tread)/ and repair (all processes involving drilling, grinding and regrooving of tread) involves tasks leading to particle emissions when drilling, grinding and regrooving of tread used in tire maintenance. Testing was carried out at laboratory to facilitate particle characterizations when operating test protocols. The handling for each task is carefully drawn from analyses of the behavior of professionals in the investigated tasks.








Different metrologies have been used to characterize particle emissions in repair workplace or incineration units related to the industry using nanomaterial composite. Composite cutting and abrasion were examined using complex equipment such as particle counters and sizers (Cena and Peters, 2011; Wohlleben and Neubauer, 2016). Microscopy was operated to describe emitted particles in size, morphology and nature (Bressot et al., 2017; Bressot et al., 2015; Lee et al., 2011; Morgeneyer et al., 2018). A metrology combining counting and microscopy enables characterization of the emitted particles but without distinguishing nanoparticles from conventional particles (Waquier et al., 2020).

A robust metrological approach including counting, microscopy analysis but also sizing is therefore necessary to characterize the nature and number of particles under stress. Regarding the challenge that represents these measurements, the most reasonable approach consists in simulating the tasks in a confined place *i.e.*, a glove box or isolated furnace respectively for the repair/retread tasks and incineration. The confinements will prevent any pollutants in ambient air from flowing in.

But no studies deal with an approach on tire life cycle to estimate a nanoparticle assessment able to evaluate the most emissive task for workers or the environment driving to possible mitigations in production process.

A new metrological approach strategy is needed in order to improve the knowledge of aerosol emission when introducing new nanoparticle in the tire formulations. A comparative strategy between one formulation of standard rubber as controlled sample and the same standard formulation containing nanoparticles acting as tracer will be implemented. The controlled sample is made of usual compounds used in usual car tires and can contain different types of chemicals, as synthetic and natural rubbers, carbon black, synthetic polymers materials including silica and steel (ETRMA, 2008). Both sorts of samples are solicited to simulate repair / retread and incineration work using an identical testing protocol and set up. The final objective of the project is to understand environmental and health potential exposure due to new nanoparticle introduction in the future formulations for having a more sustainable tire industry. To reach these objectives, two aspects of tire lifecycle are examined. Particle emission characterization using real-time and differed analysis is made for retread / repair and incineration of two tire samples. A control sample and a nano-Ag containing sample, Ag acting as a tracer, are introduced to assess the emissions. Silver has been chosen for it is not part of the usual formulation of tires and could be employed in practical application as a tracer. The obtained results are presented and discussed.

Table 1
descriptions of the instruments used in the aerosol characterization. Datamerge program was used to unify and make consistent APS and SMPS sizing.

Instruments	Measurement principle	Size range	Units	Accessible concentration range	Flowrate (l/min)
APS™ Aerodynamic Particle Sizer TSI® 	Time of flight – Aerodynamic size distribution and particle number concentration	0,5 µm to 20 µm	Particle/cm ³	Up to 10 ³ particles/cm ³	5
DMS 500 Fast Aerosol Sizer Cambustion 	Electrical mobility classification – Size distribution and particle number concentration	5 nm to 2,5 µm	Particle/cm ³	Up to 1 × 10 ¹¹ particles/cm ³	1,5
CPC 3007® Condensation Particle Counter TSI® 	Particles are grown by a working fluid (Isopropyl alcohol for instance) so they can be optically counted - Particle number concentration	10 nm to 1 µm	Particle/cm ³	Up to 10 ⁵ particles/cm ³	0,7
CPC 3775® Condensation Particle Counter TSI® 	Particles are grown by a working fluid (butanol for instance) so they can be optically counted - Particle number concentration	4 nm to 3 µm	Particle/cm ³	Up to 10 ⁷ particles/cm ³	0,3
MPS Mini Particle Sampler Ecomesure - Ineris 	Sampling on TEM grids - Morphology, size, and elemental composition using EDX	1 nm to 30 µm	–	–	0,3 à 1,5
SMPS Scanning Mobility Particle Sizer TSI® 	Electrical mobility classification – Size distribution and particle number concentration	14 nm to 740 µm	Particle/cm ³	Up to 10 ⁷ particles/cm ³	–
JEOL 1400 plus Transmission Electron Microscope (TEM) JEOL 	Morphology, size, elemental composition with EDX (Energy Dispersive X-ray)	5 nm to 100 µm	Sizes in nm / µm - Relative Elemental composition %	Size resolution of a few nm	–

2. Materials and methods

2.1. Instruments

Regarding the previous works on tire emissions the concentrations due to tire solicitation in the framework of a retread / repair phase was estimated as relatively high *i.e.* between ten to hundred thousand # / cm³ (Waquier et al., 2020). The cautious measurement strategy to characterize aerosol in these conditions coupled online (real-time particle counters and sizers) and offline (TEM analysis) measurements (Asbach et al., 2014). Measurement strategy has to account for size range of particles to investigate tire emissions. The appropriate size range is expected between few nanometers to 20 µm whatever the particle consistency *i.e.* liquid or solid may be.

The characteristics of all the instruments used in this study are summarized in Table 1. Real time particulate emission measurement was achieved using four instruments CPC 3775, SMPS, APS, and DMS 500. The first three were used for the repair / retread experiments. The CPC 3775 allows counting within a size range of 0.004 to 3 µm with a

maximum number of particles of 10⁷ particles / cm³. Size distributions may be inferred from SMPS and APS measurements. The SMPS operates within the electric mobility size range of 15 nm to 673 nm whereas the APS gives size indications from 500 nm to 20 µm. Aerodynamic diameters are obtained based on the merged size distributions gathering SMPS and APS results and using datamerge® software from TSI® company. These merged sizing enable having an average sizing between 15 nm to 20 µm over 3 testing. Each counting or sizing is diluted by 100 (VKL-100 Palas company) by introducing a diluter between the box outlet and all measuring instruments. This set up makes possible a relevant on-line analysis (counting and sizing) without any device damages. Incineration emissions are commonly high, variable and constituted with submicronic particles. The DMS 500 spectrometer (Cambustion, Fast particle analyzer, Dynamic particle spectrum) was consequently chosen to carry out counting and sizing of abundant particles generated when incinerating tire samples. The DMS 500 (Cambustion) allows sizing ranging from a few nanometers to 1 µm. Maximum measured number concentration (counting) is of around 10¹¹ particles/cm³. A diluter identical to that described above (VKL) was

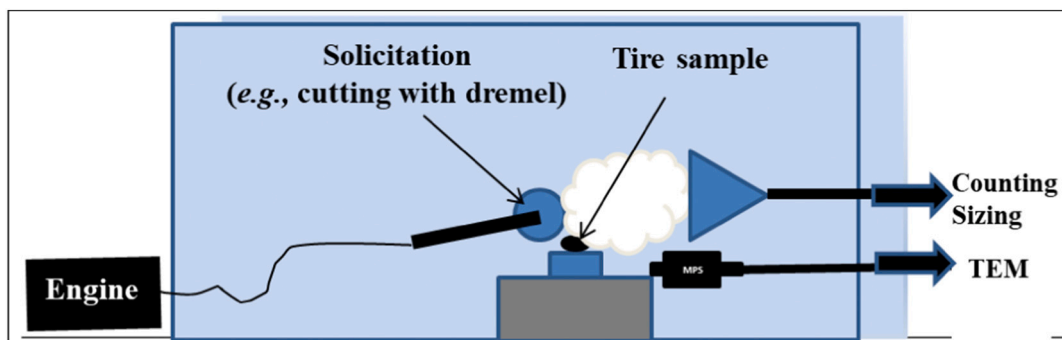


Fig. 1. Experimental setup when solliciting tire samples in a glove box. Counting (3775 CNC from TSI) sizing (APS & SMPS) after diluting aerosol by 100 (VKL-100 Palas company).

used for particle counting with the DMS 500. The use of electronic microscopy can be a real advantage when monitoring particulate matter. For this purpose, particle sampling on TEM grids was assured with a Mini Particle Sampler (MPS supplied by Ecomesure) (R'mili et al., 2013). When the MPS sampling time was too short to cover the entire particle emission, additional MPS samplings were performed. Holey TEM grids are used with the MPS through which the particle flow passes. The grids act like filters through which particles are deposited while the air can still pass through. The latter along with microscopy analysis was used for both the retread/ repair and incineration experiments. It made possible differed particle analyses. The diluter also allowed avoiding overloading the grids. Particle analysis was performed using a JEOL JEM 1400 Plus transmission electron microscope (TEM). The possibilities offered by such instrument include imagery (morphology, size) and elemental analysis of large areas of the grid integrating several particles or of single particles (minimum size of 10 nm) using the energy-dispersive X-ray (EDX) technique. A gas analyzer Horiba PG350 allows monitoring the temporal evolutions of the O_2 , CO_2 and CO concentrations. The combustion phase can therefore be identified monitoring the temporal evolution of these gases.

Dremel Tools (Dremel with Flexshaft (225/400) and a disk (digital SC 545) (28,000 rpm)) when cutting and grinding or regroover (PSO PS15 STANDARD Tyre) were operated with tool engines placed outside of the glove box to avoid contamination by engine emissions. The drill engine was too close to the drill bit to prevent total cross contamination. After different tests, a brushless drill (BOSCH GSB 18 V-EC) / bit drill DEWALT DT4912-Q2 industrial cobalt (brushless), was used through a glove box wall to avoid contaminating the glove box with the drill engine.

2.2. Protocols

2.2.1. Protocols when retreading / repairing

The experimental setup devoted to retread / repair tasks is presented in Fig. 1. Handlings is performed based on videos on the topics. Testing was carried out at laboratory and under nanosafety conditions having a low particle background noise to facilitate particle characterizations when operating test protocol and to prevent nano-exposure of operators. The tests were performed in a glove box with a HEPA filter (ref plaslabs lansing mich 800-866-7527, volume 0.288 m^3) to manage the confinement and the contamination risk; The particle background noise in the glove box is constantly below to $50 \text{ particles/cm}^3$. In addition, the tool engines could be a major source of particle contamination. The total duration of each drilling, grinding and cutting test, is equal to 30 min with 15 min for background noise and 15 min for sollicitations and particle monitoring. The specific nature of the regrooving which adds mechanical and thermic sollicitation, leads to a shorter test duration which includes 11 min of background noise and 2 min of regrooving. No significant impacts, from the particle residence time in the glove box was identified by PLC (Particle Loss Calculator) calculation. Each test was repeated 3 times.

2.2.2. Incineration protocol

The experimental setup and protocols used for these experiments are close to those used in the previous study (Dutouquet et al., 2022) Laboratory scale incineration experiments were carried out using a tubular furnace. The experimental setup is presented in Fig. 7.

The furnace is 150 cm long with an inner diameter of 95 mm. A mass of sample of about 250 mg is introduced in an Inconel-made meshed

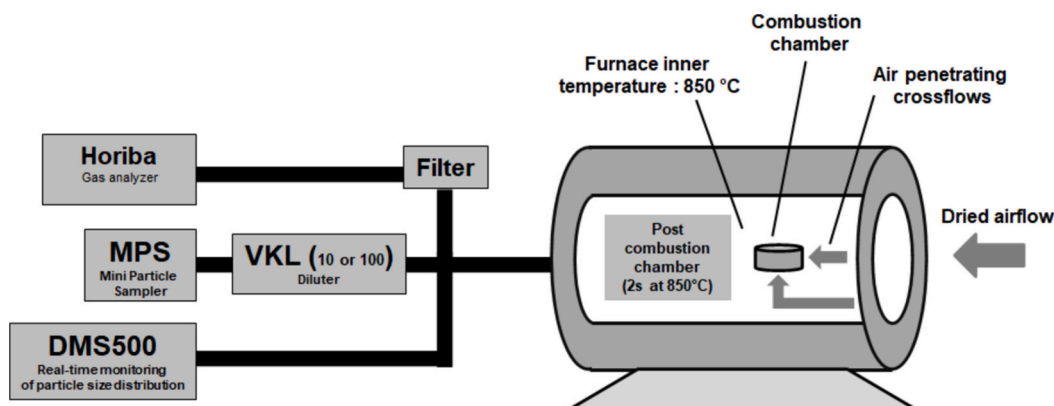


Fig. 2. Incineration experimental setup.

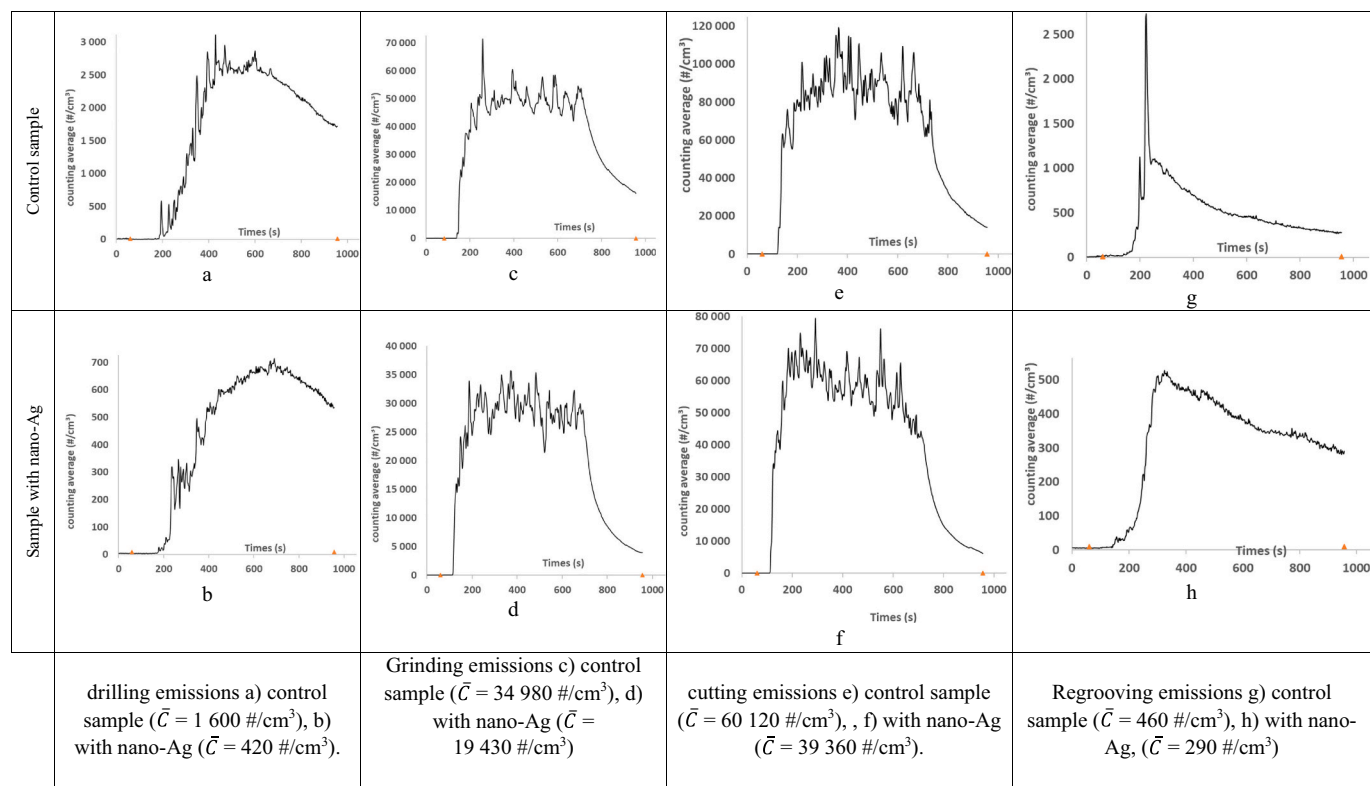


Fig. 3. Counting over time when solliciting two sorts of tire samples (control sample & sample with nano-Ag). \bar{C} : counting average on 3 tests of particle counting due to drilling, grinding, cutting and regrooving over time. Orange triangles indicate the beginning and end of the measurements.

holder (diameter of 30 mm and 25 mm height). The sample holder is meshed to let the air flow in to facilitate the combustion. When incinerating the samples, the sample holder is pushed through the furnace center to assure incineration of its content. As indicated in Fig. 2, dried air is flowed through the furnace with a flowrate of 12 L/min. The flow rate was set to assure combustion in incineration mode. The rules to be followed to assure proper incineration are indicated below:

- The inner furnace temperature was set to 850 °C;
- A residence time of two seconds in the post combustion zone was assured;
- Turbulence conditions were met in the furnace so as to assure a good mix between combustible and oxygen;
- High excess of oxygen concentration.

These four rules are usually applied in Municipal Waste Incinerator Plants. Several instruments were implemented to monitor gas and particulate emissions:

- Monitoring of gases was achieved using an analyzer PG 350 Horiba;
- A particle sizer DMS 500 Combustion was utilized for real-time particle emission monitoring;
- A MPS (Mini Particle Sampler, Ecomesure) allowing particle sampling on TEM (Transmission Electron Microscope) grids was used;
- A Palas VKL 100 diluter is positioned upstream the MPS to lower the aerosol concentration if necessary and to avoid overloading TEM grids.

The TEM analysis of the residue necessitates preparation. The residue is suspended in ultrapure water. A drop of the suspension is then

deposited on the TEM grid and ambient air dried prior to the analysis.

2.3. Samples

Two sorts of samples are tested. The first specimen, named control sample, is a classical formulation. According to Continental company, tread components are constituted with blends of different rubbers (41 %) combined with fillers (30 %), reinforcing materials (15 %), plasticizers (6 %), chemicals for vulcanization (6 %), and anti-aging agents (2 %) (Grigoratos, 2018). The composition of the carbonaceous matrix of the tread is supplemented with fillers containing silica, vulcanizing agents (S, Se, Te, organic peroxides, nitro- compounds), or vulcanizing accelerators (Pb, Mg, Zn, sulphur compounds and calcium oxides) which could slightly enrich elementary chemical composition of emitted particles and correspond approximately to 1 % of the tire rubber mass (Continental, 2013). The control is constituted by the compounds mentioned above. The second specimen consist in the last formulation from control sample but contains 2 % w/w of additional of nano-Ag with a primary particle diameter equal to 20 nm and homogeneously dispersed in the tire matrix.

3. Results

3.1. Retread/repair (cutting, drilling, grinding, regrooving)

3.1.1. Real-time counting

The online measurement includes counting (CPC) and sizing (APS and SMPS using datamerger software to make consistent size distribution curves) of emitted particles from drilling, grinding and cutting over 30 min (11 min for regrooving) and makes possible characterizations in real time leading to the identification of the moment and the duration of a release.

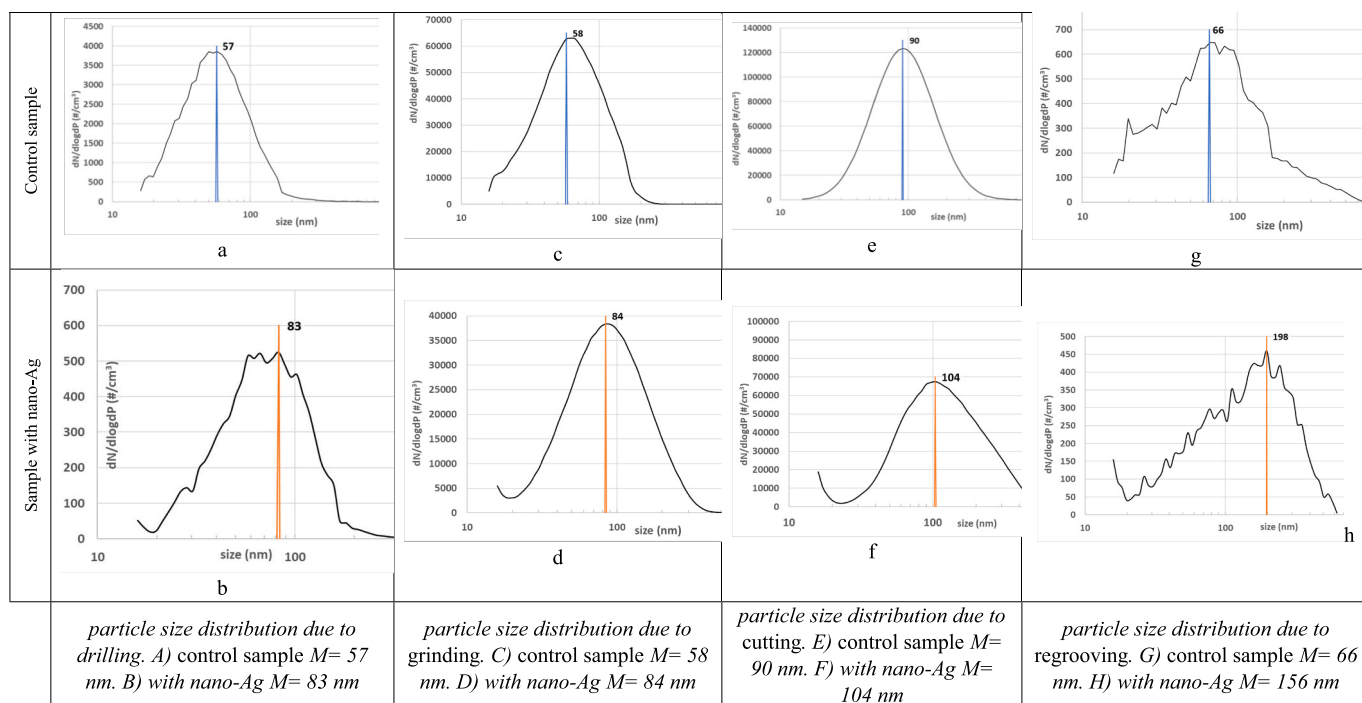


Fig. 4. Sizing over time when soliciting two sorts of tire samples (control sample & sample with nano-Ag). The size distribution cumulates over 3 SMPS and 3 APS results by merging using Datamerger software. M = Main mode on 3 tests of particle counting due to drilling, grinding, cutting and regrooving over time.

• Curve shape of counting

Drilling, grinding and cutting emission curves are similar in shape. After a short exponential growth phase, the curve reaches a plateau remaining for the rest of the test until the operator stops the stress. Then, a decrease phase starts. This last period is long for the drilling and short for the grinding and cutting solicitation. The regrooving emissions are relatively different. The short exponential growth is directly followed by a long decline in emissions.

• Intensity of emissivity of the stress.

The emission levels are estimated with the average counting when soliciting. These values are maximum when cutting or grinding (although slightly lower in the latter case) lower when drilling and the lowest when regrooving. Waquier *et al* disclosed that emissions from their abrasion tests on tires are distributed between 1700 and 50,000 #/cm³ (Waquier *et al.*, 2020). The grinding tests performed in our study showcase emissions between 19,430 and 34,980 #/cm³ (see Fig. 3) and therefore within the range of emissions mentioned by (Waquier *et al.*, 2020).

• The nano-Ag effects on counting.

As the stresses are repeated in the same manner whether the sample contains nano-Ag or not, the results are comparable. When comparing stress on control sample vs with nano-Ag, the results show that the tracer introduced in the samples lead consistently to a lower emission except when grinding.

3.1.2. Real-time sizing

• Analyzes of sizing distributions.

The aerosols produced by stressed tires are submicronic with a main

nanosized part of the particles. The main modes are below 100 nm except when cutting and regrooving nano-Ag samples. To our knowledge, no similar work is available on the size distribution of tire particle emissions according to the types of stress described in this study. Consequently, no comparison is currently possible with the values obtained in our tests.

• The nano-Ag effects on sizing.

The emissions showcase a main mode that shifts to bigger sizes when testing nano-Ag vs without nano-Ag charge. This shift is maximum when regrooving *i.e.*, a size difference equal to 38 nm, intermediate at 27 nm when drilling or grinding and minimum at 14 nm when cutting (Fig. 4).

3.1.3. TEM analysis

• Drilling.

TEM analysis observations made when drilling control samples leads to the identification of two particle categories. First, objects with sizes distributed between a few nm to approximately 1 μm and possessing an irregular shape (see Fig. 5a) can be observed. Second, objects of micronic, submicronic, or nanometric sizes with regular morphologies comparable to dried droplets can be seen. The main chemical elements identified are carbon, oxygen, sulphur, silicon in both cases showing identical concentrations between both particle categories *i.e.* irregular-shaped particles and dried droplets *i.e.* μplastics particles.

The collected objects when drilling the sample with nano-Ag are exclusively irregular-shaped solid particles with sizes distributed between few nm to approximately 1 μm (see Fig. 5b). No traces of dried droplets have been detected. The three main chemical elements detected in the particles are carbon, oxygen and silicon.

• Grinding.

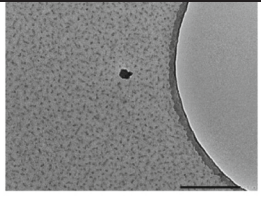
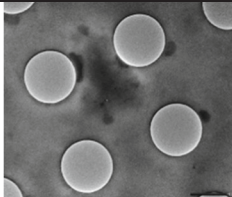
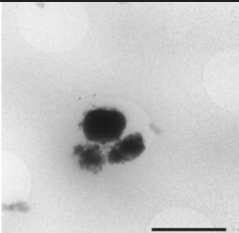
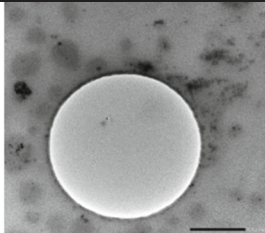
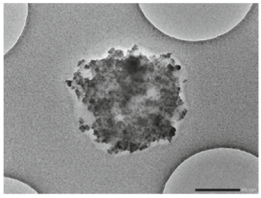
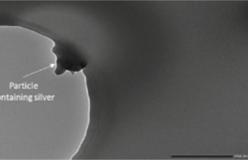
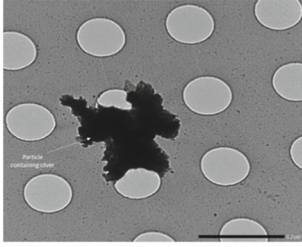
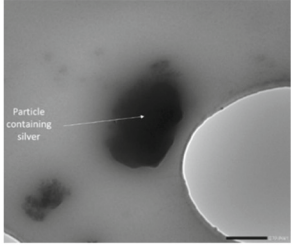
Control sample	 <p>a</p>	 <p>c</p>	 <p>e</p>	 <p>g</p>
Sample with nano-Ag	 <p>b</p>	 <p>d</p>	 <p>f</p>	 <p>h</p>
	<p>a) nanometric irregular-shaped particles on grid after drilling test/ control sample. Scale: 200 nm.</p> <p>b) : micronic irregular-shaped particles after drilling test / sample with nano-Ag. Scale: 500 nm</p>	<p>c) dried droplets from control sample when grinding. Scale: 1 μm.</p> <p>d) submicronic irregular-shaped particles from grinding / sample with nano-Ag. Particle containing 3 % of silver (carbon 86 %, oxygen 8 %, silicon 3 %). Scale: 200 nm.</p>	<p>e) blurred images with irregular-shaped submicronic particles covered by a film when cutting the control sample. Scale: 1 μm.</p> <p>f) micronic irregular-shaped particle containing 3 % (w/w) of silver after cutting the sample with nano-Ag. Other compounds: carbon 83 %, silicon 8 %, calcium 6 %. Scale: 2 μm</p>	<p>g) submicronic/nanometric irregular-shaped particles in presence of droplets when regrooving control sample. Scale: 500 nm. h) submicronic particle after regrooving sample with nano-Ag. particle containing 8 % (w/w) of silver. Other compounds: carbon: 59 %, silicone: 17 %, oxygen: 11 %, aluminium 2 %, iron 3 %. Scale 200 nm.</p>

Fig. 5. Transmission Electronic Microscopy images from solicitation sampling.

The two particle categories *i.e.* dried droplets and irregular-shaped particles described previously are visible when grinding control samples (Fig. 5c). A thin layer partly covering the TEM grid is sometimes detected.

The samples with nano-Ag produce, under grinding conditions, first a film which largely covers the grid and, second submicronic irregular-shaped particles. This last category is constituted by objects with sizes ranging between a few nm to about 1 μm . The main compounds detected on the grid by EDX regardless of the type of the analysed objects *i.e.* film or irregular-shaped particles are carbon, oxygen and silicon. Traces of iron, chlorine sulphur are also identified. One particle out of about fifty contains a low concentration of silver included in a carbon matrix (see Fig. 5d).

- Cutting.

The particles analysed after sampling during control sample cutting are covered by a film deposited all over the grid. The film blurs most of the images and is mainly made of carbon (Fig. 5e). The irregular-shaped particles made of carbon oxygen silicon and sulphur are also visible (Fig. 5e), but no solidified droplets are identified. The particle sizes are distributed between few nm to about 1 μm .

The cutting process applied on samples with nano-Ag produces two

categories of depositions. First a carbonaceous film is deposited on the grid surface and blurs the images. A second category of objects is distinguished as irregular-shaped submicronic or nanometric particles commonly covered by the carbonaceous film like described previously. The main elements are carbon, oxygen, sulphur and silicon for the film and the irregular-shaped particles. Traces of iron, chlorine are occasionally detected. Furthermore, one out of about seventy particles on the grid (3 % w/w) is observed to be containing silver on the grid (see Fig. 5f). This particle also contains carbon, silicon and calcium.

- Regrooving.

The prevailed object deposited on the grid when regrooving is a film mostly made of carbon, oxygen, sulphur, silicon and traces of chlorine (see Fig. 5g). Some parts of the porous grid are partially obstructed by this film. The irregular-shaped particles with sizes distributed between few nm to 1–2 μm are visible and made of the same chemical elements as the film. This composite irregular-shaped particle is embedded in a carbonaceous matrix. In addition, some solidified droplets are detectable where no films are deposited on the grid.

When regrooving samples with nano-Ag, the particle depositions are dominated by 1/ a carbonaceous film and 2/ irregular-shaped particles mostly under the form of NOAA. This last category is submicronic and

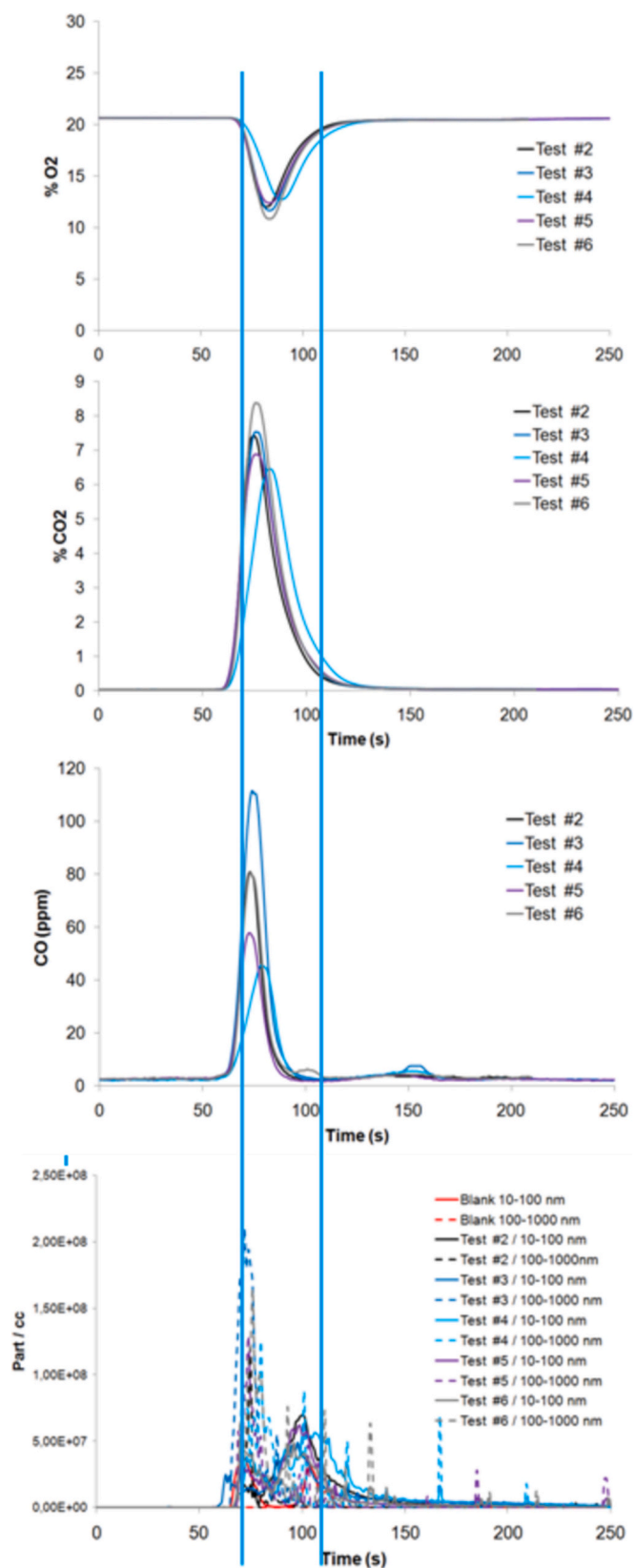


Fig. 6. Temporal evolution of the different gases (O_2 , CO_2 , CO) and of particle emission when incinerating the control sample. Successive tests labelled in the figures were performed. For particle monitoring, the dashed line is about the 100–1000 nm size range whereas the continuous line represents sizes ranging from 10 to 100 nm.

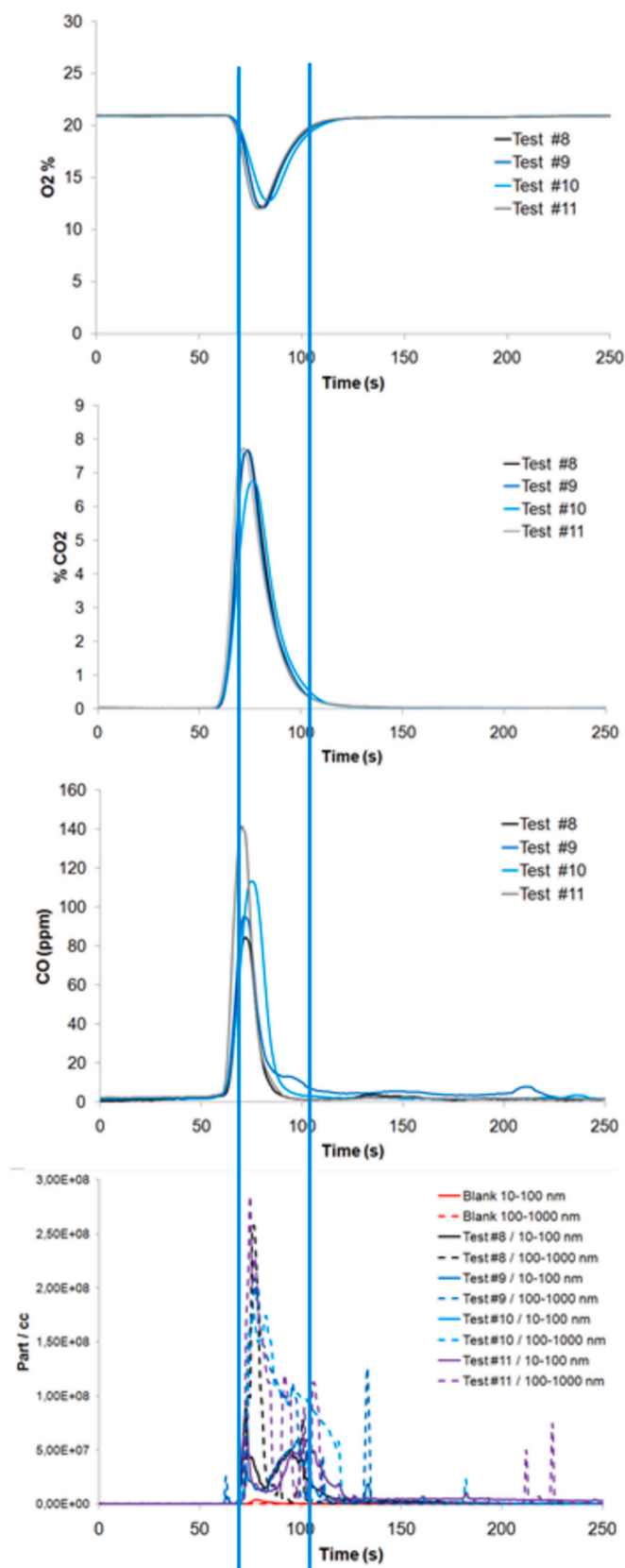


Fig. 7. Temporal evolution of the different gases (O_2 , CO_2 , CO) and of particle emission when incinerating the sample with nano-Ag. Successive tests labelled in the figures were performed. For particle monitoring, the dashed line is about the 100–1000 nm size range whereas the continuous line represents sizes ranging from 10 to 100 nm.

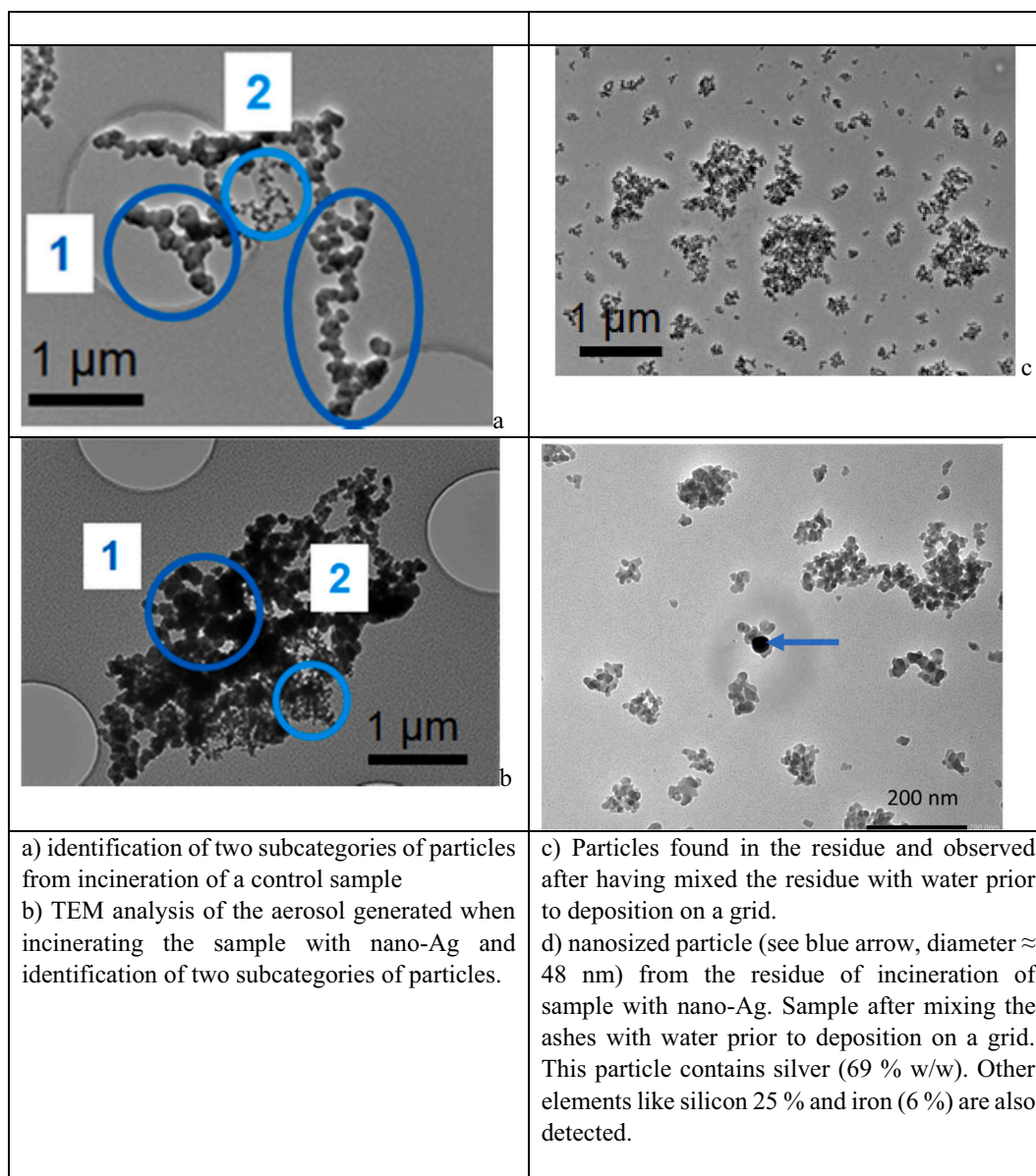


Fig. 8. Observed particles after tire sample incineration.

made of carbon, oxygen, silicon, sulphur. One out of about sixty particles is observed on the grid with 8 % w/w of silver included in a composite object mainly made of a carbon matrix (see Fig. 5h).

3.2. Incineration

3.2.1. Real-time monitoring of gases and particles (real-time counting, sizing and gas analysis)

The examination of the temporal evolution of oxygen, carbon dioxide, carbon monoxide allows verifying that combustion unfolds without a hitch. Particle emissions in size fractions of 10–100 and 100–1000 nm are also monitored. The corresponding graphs are presented in Fig. 6. The two blue lines delimitate a time span when the oxygen consumption rate is higher than 5 %. They represent the beginning and the end of the combustion. The graphs display a good synchronization between the beginning and the end of the combustion and particle emission. It shows that everything went smoothly in the combustion process. In terms of particle emission, blanks were measured prior to the experiments *per se*.

Blank measurements are achieved by pushing through the furnace emptied meshed holder to check particulate level. The curves in red represent the blanks. There are two curves for two size ranges, namely 10–100 nm and 100–1000 nm. For the 10–100 nm size range, the blank intensity is a bit high. Nevertheless, microscopy observations have not shown nanoparticle deposition on TEM grids when recording the blank. The blank corresponding to the 100–1000 nm size distribution is clearly seen to be negligible.

Particulates in the size range of 100–1000 nm are eventually seen to dominate aerosol emission. The graphs make it clear that particle emission stops when the combustion is over, namely when oxygen consumption drops below 5 % (right vertical blue line).

Fig. 7 presents gas emission over time when incinerating the sample with nano-Ag. The two vertical lines correspond to the moment when the oxygen rate consumption is of about 5 % at the beginning and at the end of the combustion. The left line points out the moment the sample is introduced in the furnace then sealed off. The right line marks the end of the combustion when the oxygen level is equal to its initial

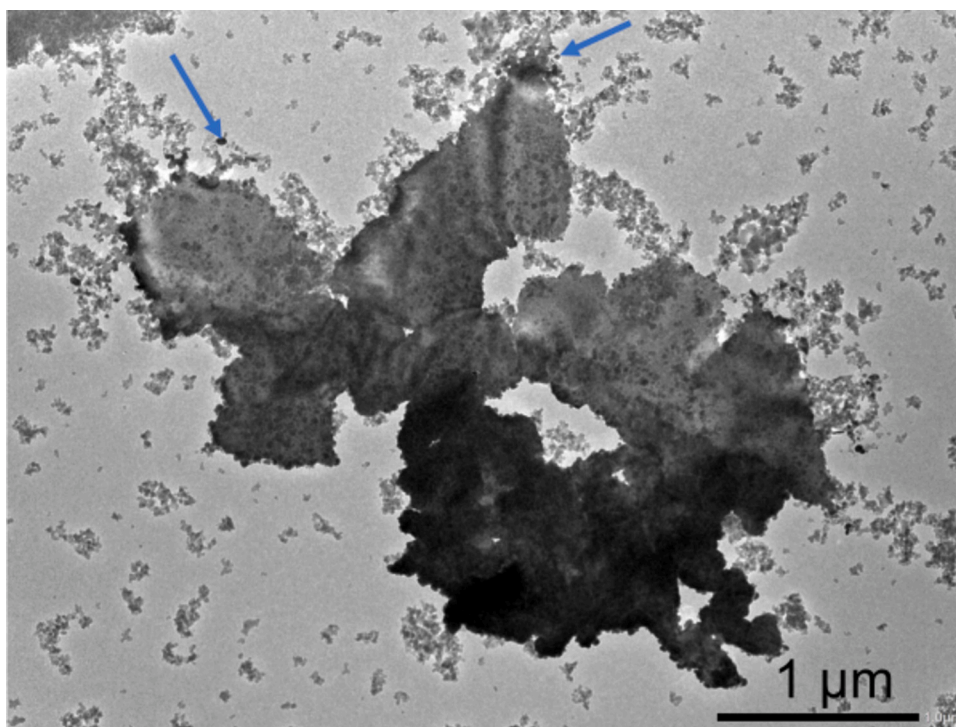


Fig. 9. TEM image of a large particle made of silver with nanosized particles (see blue arrow) could be made exclusively of silver.

concentration. Particle and gas emission evolve in a similar manner and are well synchronized: e.g. particle counting increases when starting oxygen consumption which highlights the beginning of the combustion. Blank emission was recorded along with other recordings. The blanks are assessed when introducing an empty sample holder in the furnace. The red line in Fig. 7 shows the evolution of the blank over time and showcases that blank emissions are negligible compared to the emissions obtained when incinerating the samples. The graphs of Fig. 7 show that 100–1000 nm particle sizes dominate the emission whatever the test under investigation.

3.2.2. TEM analysis

- Analyzes of sampled particles with the incineration of the control sample.

The particles sampled on the TEM grids and corresponding to aerosol emission when incinerating the control samples are presented in Fig. 8a. Though the grid was carefully examined, only one category of particles was found. The grid displays a large number of agglomerates of which many are micrometric in size. EDX analysis makes it clear that all these particles are made of carbon. Two size distributions may be observed for the primary particles of these agglomerates. A first subcategory (1) is made of agglomerates with primary particles ranging from 50 to 150 nm. A second subcategory is made of finer particles with sizes of about 20–50 nm. Particles with elemental composition other than carbon could not be seen on the grids. Given these observations, conclusion is made that these particles are actually soot and that part of these soot are nanosized.

- Analyzes of sampled particles from the incineration of samples with nano-Ag.

All the grids gathered when incinerating the sample with nano-Ag display agglomerates on their surface. Two subcategories are identified and correspond to different size distributions of the primary particles of the agglomerates. The first subcategory (1) is constituted of

primary particles with sizes ranging from 140 to 200 nm (see Fig. 8b). The second category is made of primary particles with smaller sizes, i.e. 20–30 nm (see Fig. 8b). Particles with elemental composition other than carbon are not observed on the grids. The examination of the elemental composition and of the morphology of these agglomerates lead to the conclusion that these are soot and that part of these soot are nanosized.

- Analyzes of the residue after incineration of the control sample.

Particles observed when analyzing the residue from incineration of a control sample are presented in Fig. 8b. Here again, a thorough examination of the grid was conducted and only one category of particle was found. A large number of agglomerates of all sizes is observed, micrometric and submicronic in sizes. The observed agglomerates display primary particles with sizes of about 20–40 nm which labels them nanostructured. Elements such as silicon (dominant compound), carbon and oxygen were found when performing the EDX analysis. All these data substantiate the conclusion that these are silica particles associated with soot (cf Fig. 8b).

- Analyzes of the residue after incineration of the sample with nano-Ag

Images of the particles found in the ash residue are presented in Fig. 8d. There are two categories of particles. Like previously reported, many of the observed particles are agglomerates made of silicon (dominant element), carbon and oxygen. As already indicated, these agglomerates are very likely to be made of silica. As may be observed, a few particles with different shapes are visible on the grid. EDX analysis shows that these particles are made of the zinc and oxygen elements. Silver was also found in the residue in big and small particles (see Fig. 8 d). It should be noted that very few of these particles can be observed on the grids.

- STEM/EDX of particles from residue after incineration of the sample with nano-Ag

A STEM/EDX mapping on ash residue containing nano-Ag used to

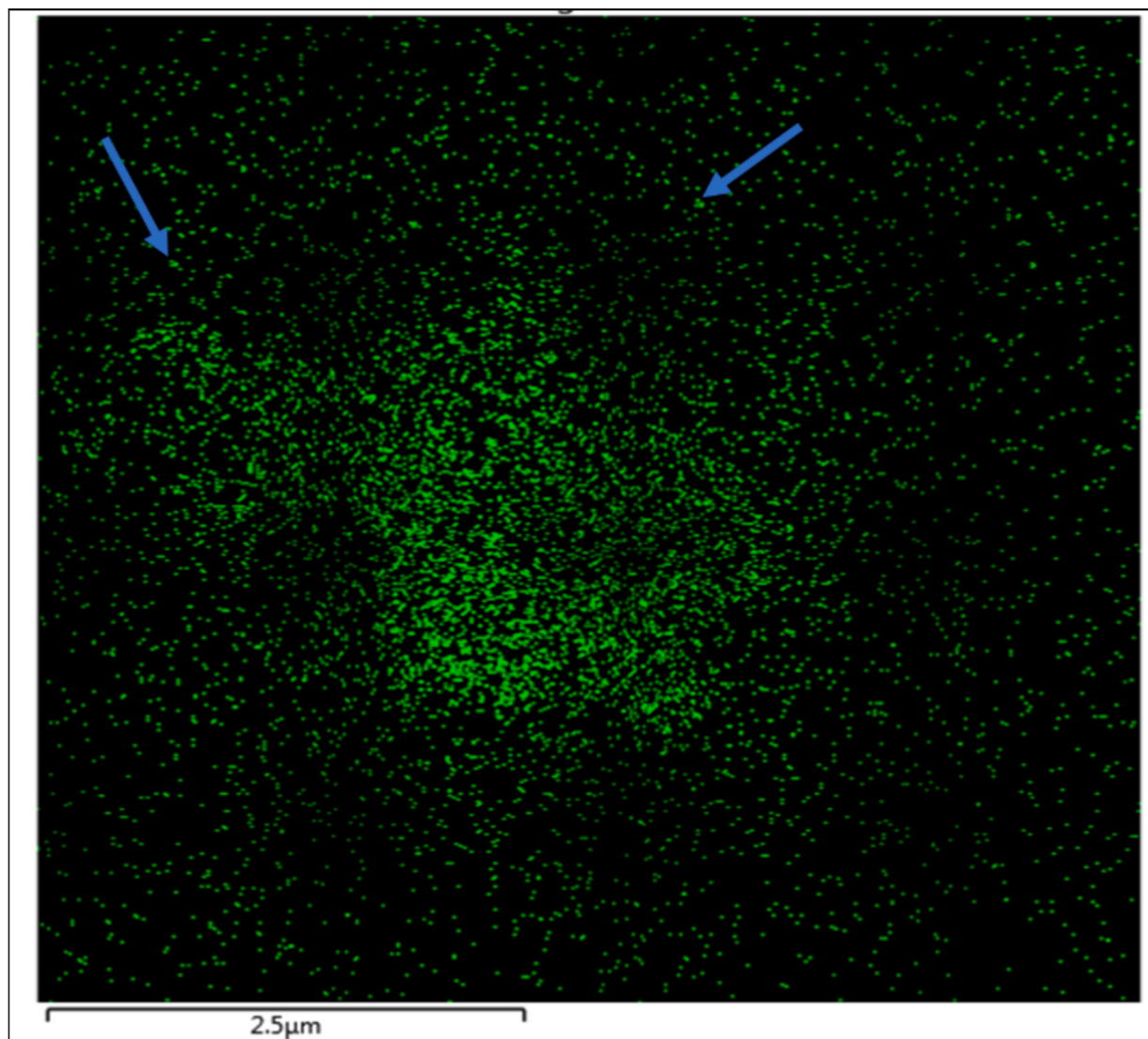


Fig. 10. STEM/EDX mapping of silver (in green) on sample with nano-Ag.

confirm the potential presence of free nanosized silver particles. The interest of STEM/EDX mapping is to locate the presence of chemical element chosen (for example, metallic silver) on a sample. This mapping includes silver and other chemical elements present on the sample such as silicon, oxygen. This approach makes possible a comparison between the position and the particle size of these chemical elements on the sample. A silver particle with no other chemical elements will be considered as a free metallic silver particle. In presence of another chemical element, this particle will be considered as a composite (salt, oxide, mineral, material, ...).

A STEM/EDX mapping on nano-Ag containing sample was performed around a large particle displayed on Fig. 9. Chemical elements present on the sample such as silver, silicon and oxygen were included in the analysis. The following images (see Figs. 10-12) depict the location and the size of the investigated chemical elements. Silver mostly is located in the centre of the image in the large supermicronic particle. However, few nanosized objects (e.g. two arrows mark two nanosized particles isolated from the large particle) made of silver (see Fig. 10) among the supermicronic particles are visible. It shows that very few free nano objects are identified in the residue of samples with nano-Ag. Figs. 11 and 12 highlight an identical spatial distribution of silicon and oxygen. This last point shows the distribution concordance in the sample

between these two chemical elements due to the presence of silica or SiO_2 in the residue.

4. Discussion

The formulation types of tire samples presented in this study are relatively limited and lead to proceed cautiously when comparing with current product in the market.

- Retread/repair

The retread / repair experiments provide the following information: i/ A relatively high and similar level of particle emissions for both samples is observed, ii/ The emitted particles are mainly of irregular shape and to a lesser extent made of dried droplets or films deposited on the grid surface (see Table 2). Particles of irregular shape are systematically observed while dried droplets or films are occasionally absent on the samples (e.g. no dried droplets when drilling/ samples with nano-Ag, no film when drilling/ both samples). Particles of irregular shape are assumed to be originated from pulled out process without passing through a semi-volatile intermediate state. Films or dried droplets come from condensation / coalescence processes. iii/ Based on online results,

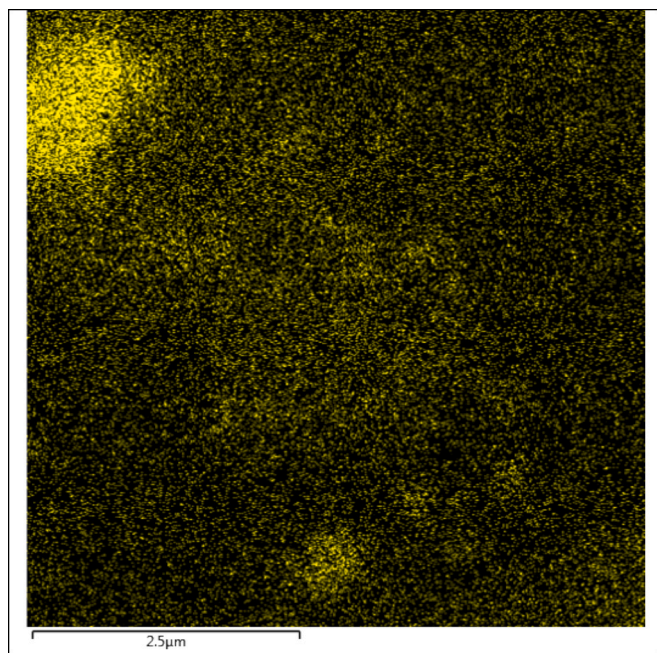


Fig. 11. STEM/EDX mapping of silicon (in yellow) on Sample with nano-Ag.

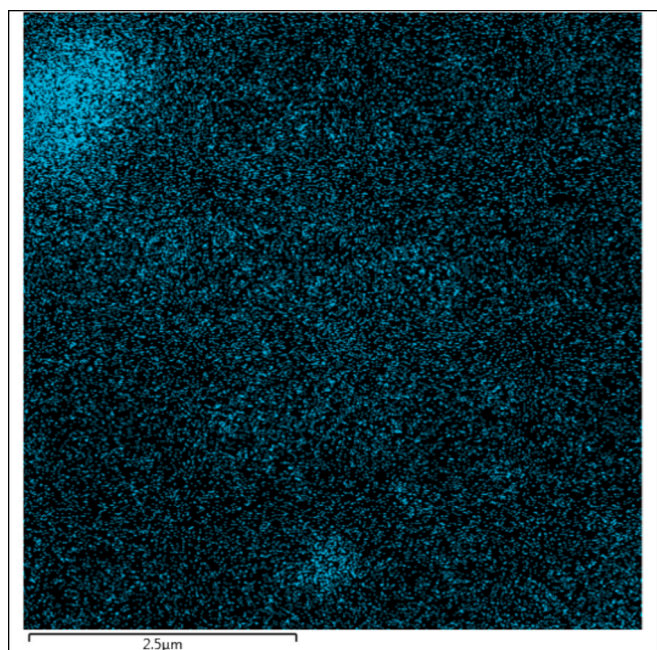


Fig. 12. A STEM/EDX mapping of oxygen (in blue) on Sample with nano-Ag.

and regardless of the nature of the studied samples, the processes are distributed with increasing average counting as follows: regrooving, drilling, grinding and cutting (see Table 2). iv/ Almost the totality of the aerosol is constituted by submicronic particles with a big fraction of nanosized objects and can be considered as μ plastic particles. v/ Carbon and oxygen dominate the elementary chemical composition of emitted objects and is associated regularly with silicon and sulphur while chlorine and iron are occasionally detected.

In all likelihood, micronic or submicronic particles mostly made of carbon and oxygen originate mainly from rubber constituents and fit into the category of μ plastics. However, emission from tire repair/retread tasks seems a minor source of μ plastics if compared to that of tire/road contacts (Sommer et al., 2018). Waquier et al. mention that amorphous droplets, possibly originate from degraded polymer and are produced when curing rubber at high temperature and pressure *i.e.*, 130–170 °C, > 10 bar (Waquier et al., 2020). The stresses applied on the samples and described in this article appears to reach such conditions on the contact tools/tire enable to produce also semi-volatile components. Films or droplets are suspected to be originated also from semi-volatile emissions, condensed then cooled on the grid.

Carbonaceous films observed in this study are rarely concomitant with presence of droplets except when grinding the control samples. The film deposition is possibly caused by an abundant production of droplets which coalesce and dry as a film over the entire surface of the TEM grid. To our knowledge, it is the first description of these types of objects when retreading or repairing tires. Moreover, no exhaustive studies of this phenomena have been presented until now. In particular, no carbonaceous films have been observed by microscopy from sampling when stressing tire samples. These last observations are additional clues which indicate a possible degradation of the rubber matrix leading to the production of droplets, or films.

The samples with nano-Ag give rise to the detection of low concentrations of silver due to grinding, cutting and regrooving operations but not when drilling. The particle production during drilling is probably too low to make possible a TEM detection in these conditions. Indeed, the tire samples have a low concentration of tracer (2 % nano-Ag w/w). However, the metrological strategy makes possible the characterization of a nanoparticle emission in number, size, shape and elemental composition for the rest of the solicitation *i.e.*, grinding, cutting and regrooving. According to the test conditions described in this article no free nano-silver are detected when sampling emitted particles produced by retread / repair simulations.

- Incineration

For both samples, the emission (in the aerosol) due to incineration is dominated by soot. Many of the soot particles are made of primary particles nanometric in size. The combustion residue was found to be dominated by particles containing silicon and oxygen, silica most likely, for both samples.

Particles containing zinc and oxygen, were found in the residue of the sample with nano-Ag. But these were very few in number.

The examination of the grids did not lead to the detection of other elements except silver in two residue particles out of forty objects from samples with nano-Ag. Silver is detected mainly in the form of large micronic or supermicronic particles. A STEM/EDX mapping analysis also reveals rare nanoscale objects containing silver. These nano-silver objects are away and isolated from large particles made mainly of silver.

5. Conclusions

Apart from the tire use phase, which has already been extensively studied, five tasks are necessary to simulate life cycle operations: drilling, grinding, cutting, regrooving and incineration. A known charge of nano-Ag is introduced in a classical tire formulation for acting as tracer and used in the five task simulations.

The emitted particles from retread/repair and incineration observed in this study were successfully characterized in number, size shape and elemental composition. These emissions were mainly submicronic and the produced amounts depended largely on the used stresses. The nature

Table 2

Comparison between the results in retread/repair simulation. * Films or droplets made of tire compounds are suspected to be originated from semi-volatile emissions, condensed then cooled on the grid. ** irregular shapes particles made of tire compounds indicating the pulled-out process without passing through a semi-volatile intermediate state. (a) presence of silver concentration; ND/ Not Determined.

Process	Drilling		Grinding		Cutting		Regrooving		Incineration (aerosol)		Ash Residue	
	Online measurement	Offline measurement	Online measurement	Offline measurement	Online measurement	Offline measurement	Online measurement	Offline measurement	Online measurement	Offline measurement	Online measurement	Offline measurement
Main data	Counting average/ max/(#cm ³) Sizing (N/PSM/SPM) Mode (nm) Dried droplets (Y/N) or Filaments*(Y/N) Films* (Y/N)	Irregular particles*(Y/N)	Counting average/ max/(#cm ³) Sizing (N/PSM/SPM) Mode (nm) Dried droplets (Y/N) or Filaments*(Y/N) Films* (Y/N)	Irregular particles*(Y/N)	Counting average/ max/(#cm ³) Sizing (N/PSM/SPM) Mode (nm) Dried droplets (Y/N) or Filaments*(Y/N) Films* (Y/N)	Irregular particles*(Y/N)	Counting average/ max/(#cm ³) Sizing (N/PSM/SPM) Mode (nm) Dried droplets (Y/N) or Filaments*(Y/N) Films* (Y/N)	Irregular particles*(Y/N)	Counting average/ max/(#cm ³) Sizing (N/PSM/SPM) Mode (nm) Dried droplets (Y/N) or Filaments*(Y/N) Films* (Y/N)	Irregular particles*(Y/N)	Counting average/ max/(#cm ³) Sizing (N/PSM/SPM) Mode (nm) Dried droplets (Y/N) or Filaments*(Y/N) Films* (Y/N)	Irregular particles*(Y/N) Dried droplets (Y/N) or Filaments*(Y/N)
Control samples	1600/2150 Np/SM	57 Np/SM	53 860 / 26 534 Np/SM	58 Np/SM	601 20 / 119 210 Np/SM	104 Np/SM	460 / 2750 Np/SM	66 Np/SM	ND 2.0 10 ⁶ Np/SM	ND Np/SM	ND Np/SM	ND Np/SM
Samples with nano-Ag	420/710 Np/SM	83 Np/SM	19 430 / 35 220 Np/SM	84 Np/SM	39 560 / 79 540 Np/SM	104 Np/SM	260 / 530 Np/SM	198 Np/SM	ND 2.7 10 ⁶ Np/SM	ND Np/SM	ND Np/SM	ND Np/SM

of the emissions was also studied, and different categories of particulate matter were identified: e.g., solid particles but also solidified liquid particles emitted during repair operations were observed and can be considered as µplastic particles. In contrast, incineration mainly resulted in the production of an aerosol of soot particles.

The composition of the samples with nano-Ag emitted the same amount or sometimes less nanoparticles (i.e. drilling, cutting and regrooving) than the control samples. Whatever the studied samples, particle emissions are distributed with increasing average counting as follows: regrooving, drilling, grinding and cutting. According to the solicitation investigated, some differences are identified: neither dried droplets when drilling samples with nano-Ag, nor films when drilling/control samples or samples with nano-Ag were observed. Wear debris are considered as originating from pulled out processes. Dried droplets / films depositions may arise from condensation/coalescence processes. The main elementary chemical composition detected on the grids are carbon, oxygen, and to a lesser extent silicon and sulphur. Chlorine and iron are occasionally identified. Silver is also identified at low concentration between 3 and 8 % of the particles in irregular-shaped particles originated from samples with nano-Ag.

Incineration of both sorts of samples (one control sample, the other with nano-Ag) were performed in a laboratory scale furnace. The emitted aerosols and the bottom ash were studied. In both cases, soot is the main produced type of particles. The residue resulting from combustion is constituted mostly by particles made of silica. Few particles of ZnO and of silver are additionally distinguished in the residue of the sample with nano-Ag. Silver is identified mostly in the form of large micronic or supermicronic particles. A STEM/EDX mapping analysis exhibits rare nanoscale objects containing free nanosized silver particles separated from the large silver object.

CRedit authorship contribution statement

Christophe Bressot: Writing – original draft, Supervision, Conceptualization. **Laurent Meunier:** Resources, Methodology, Formal analysis. **Christophe Dutouquet:** Writing – original draft, Validation, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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