Application of standardization for the design and construction of carbon nanotube-based product pilot lines in compliance with EU regulation on machinery

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Abstract. The “PLATFORM” manufacturing ecosystem for pilot production of pre-commercial CNT-based nano-enabled products, consists of three pilot lines (PPLs) for the manufacture of buckypapers, doped prepregs and doped veils. The PPLs have been constructed with the ultimate goal to commercialize these products in the European market in 2020/2022. This goal requires having the PPLs in compliance with the applicable product safety regulation by that date (CE marking). The main EU regulation for new machinery (as the PPLs) is the Directive 2006/42/EC on Machinery (MD). This Directive sets out the general mandatory Essential Health and Safety Requirements (EHSRs) related to the design and construction of machinery, while particular technical specifications for fulfilling them are provided in European harmonized standards. Application of harmonized standards is voluntary but confers a presumption of conformity with the EHSRs they cover. The PPLs are unique machines for own use and must comply with the MD before they are put into service, in 2020/2022. But the MD does not provide specific EHSRs for nanosafety and no harmonized standards are available in this field for the safe design of the PPLs. In this context, this paper shows the standardization strategy followed by the project PLATFORM (GA 646307) to design the PPLs in compliance with the EHSR referred to the risks to health resulting from hazardous substances emitted by machinery (MD, Annex I, EHSR 1.5.13). In the absence of nanosafety harmonized standards to satisfy the aforementioned EHSR, the design and design verification of the PPLs were carried out through A & B - type harmonized standards (e.g. EN ISO 12100, EN ISO 14123-1/2), and other European and international standards.

1. Introduction and motivation

Pilot Lines (PLs) are pre-commercial production facilities, aimed at developing, testing, demonstrating and scaling new technologies and products, with the objective of accelerating their
introduction into the market [2]. The additional feature "Open" of some PLs (OPLs), implies their accessibility and possibility of use by third parties, such as companies, and in particular SMEs. OPLs can be grouped into more complex pre-commercial production ecosystems such as the Open Innovation Test-Beds (OITBs) [3] where, in addition to the physical manufacturing facilities, other advanced services are also offered to companies (e.g. technological, business, marketing), with a single-entry point for users. OPLs and OITBs are strategic instruments of the European Commission to bridge the "valley of death", and successfully introduce innovations based on Key Enabling Technologies (KETs) into the market [2,3]. They are the embryo of tomorrow's nano-manufacturing industry in Europe.

Regulation is one of the barriers commonly mentioned for scaling up and marketing innovative products. In the field of nanotechnology and nanomaterials, the ambiguity and lack of uniformity of the current European regulatory framework and the parallel uncertainty generated about the responsibility associated with new innovations, represent relevant barriers for the introduction of new nanotechnological products in the market [4].

Standardization can contribute significantly to managing ambiguity in a context of regulatory uncertainty, providing technical criteria for new products. In contrast, its absence can delay the introduction of innovations in the market and increase opportunity costs (certifications and additional tests, costs to adapt the products to the different standards of the Member States) [4].

In this context of pilot production, the H2020 PLATFORM project (https://www.nanocomposites-hub.com) is an initiative in the field of nanotechnology and advanced materials, aimed at building up a platform for the open pilot production of pre-commercial carbon nanotube-based nano-enabled intermediate products. It consists of three pilot lines (PPLs) for the manufacture of buckypapers (PPL1), doped prepregs (PPL2) and doped veils (PPL3), for their use in the manufacture of composite-based components for aeronautics and automotive (Tables 2,3). The project ultimate goal is to commercialize all these products in the European market in 2020/2022.

The PPLs are new and unique manufacturing machinery for own use, which arises directly from the innovation processes conducted by the PLATFORM project, and by other previous research projects. The manufacture of new machinery is regulated in Europe by the Machinery Directive 2006/42/EC [MD] [1,5] and, in practice, by the national regulations that transpose this Directive in the Member States. According to the MD, the new machinery must meet demanding product safety requirements for commercialization, in order to facilitate the free movement of machinery in the Single Market and ensure the protection of people and the environment.

In recent years, a strong emphasis has been placed on the safe design of nanomaterials (ENMs) based on a Safe-by-Design (SbD) approach [15,21]. However, less attention has been given to the safe design of the manufacturing processes of these ENMs and nano-enabled products (NEPs); although such processes, can be a primary source of emissions, wastewaters and wastes containing ENMs, and therefore, of risks for the safety and health of the workers and the environment, derived from exposure to ENMs.

SbD is the best strategy to eliminate hazards and minimize risks, from the initial design stage of machinery and processes [1]. The MD integrates a robust SbD approach for the design of new machinery, strongly supported by standardization, which has been used by the PLATFORM project to align the design and construction of the PPLs, and ensure compliance with the MD when putting them into service (expected in 2020/2022) [22]. However, the MD does not provide specific requirements for nano-manufacturing processes, and there are no harmonized standards available in the field of nanosafety for the design of the PPLs.

In this context, the challenge is to design, construct and put into service in 2020/2022, three PPLs for the manufacture of CNT-based nano-enabled products, in compliance with the MD, and based on a SbD approach. The regulatory uncertainties cited above, will impact on their design, construction and commercialization, and specifically on the responsibility of the manufacturers of this machinery.
2. The regulatory and standardization context for the design and construction of PPLs

The MD represents the European regulatory framework for the manufacture of new machinery [1,5]. As a product regulation, the primary responsibility regarding the conformity of the manufactured machinery, always falls on the manufacturer side (natural or legal person).

The MD applies to machinery when it is placed on the market and/or put into service. In the case of machinery manufactured by a person for their own use (such as PPLs), the MD applies when the machine is first put into service. In other words, such machinery must meet the requirements of the MD before being used for the first time in the EU, for the intended purpose (2020/2022 in the case of PPLs). Machinery specially designed and constructed for research purposes, for temporary use in laboratories, is initially excluded from the application of the MD [1].

The MD offers three conformity assessment procedures, depending on the type of machinery and the availability of harmonized standards: 1) Self-assessment, 2) EC-type examination and 3) Full quality assurance system. The first procedure is the simplest and the one used by 80% of the manufacturers. It will also be the procedure to be used by the PPLs [1].

Annex I of the MD defines the mandatory Essential Health and Safety Requirements (EHSRs) for the different types of machinery (general principles), and their translation into technical specifications is done through European harmonized standards. There are three types of harmonized standards available regarding the MD [11]: 1) Type-A standards (basic safety standards), giving basic concepts, principles for design and general aspects that can be applied to machinery; 2) Type-B (generic safety standards), dealing with one safety aspect (B1) or one type of safeguard (B2) that can be used across a wide range of machinery and, 3) Type-C (machine safety standards), dealing with detailed safety requirements for a particular machine or group of machines. The use of harmonized standards is voluntary, and the manufacturer is not required to use such standards to ensure the conformity of his machine with the EHSRs. However, machinery manufactured in accordance with a harmonized standard, is presumed to comply with the EHSRs of the MD covered by that standard. In practice, half of the manufacturers use a combination of harmonized and non-harmonized standards to satisfy the EHSRs of the MD [5].

In particular, the EHSR 1.5.13 of the MD refers to the health hazards derived from the "emissions of hazardous materials and substances" produced by machinery, with the view to protect the user. Although this requirement does not explicitly refer to emissions containing ENMs, its general principles can be applied to the PPLs. The requirement refers to the principle of SbD to avoid hazards or reduce the risks of inhalation, ingestion, contact with the skin, eyes and mucous membranes and penetration through the skin of hazardous substances; as well as the implementation of risk reduction measures, installed as close as possible to the emission source to ensure the maximum effect [1]. This EHSR also covers emissions of conventional pollutants that the PPLs can produce, such as e.g. VOCs or dust.

3. The SbD approach in the context of the MD

The MD deploys a robust SbD model for machinery, based on risk assessment (RA). General principles are specified in Annex 1 of the MD (principles of safety integration), and technical specifications are provided by the harmonized standard EN ISO 12100: 2010 [1,11]. This standard describes the three-step method for the implementation of a risk assessment and risk reduction process, hierarchical and iterative, aimed at achieving the greatest practicable risk reduction, to ensure a safe design of the machine. Figure 1 shows that the measures implemented by the designer of the machine, can be supplemented by the end user in the workplace to cover the residual risks.

Specifically, for hazardous substances emitted by machinery, the harmonized standard EN ISO 14123-1:2015 customizes the three-step method in the following way:

1) Eliminate or prevent risks resulting from exposure to hazardous substances (“inherently safe design”); 2) Reduce risks that cannot be eliminated through safeguarding and complementary protective measures, in the following order of priority: 2.1) Reduction of emission, 2.2) Reduction by ventilation or other engineering means and 2.3) Reduction of exposure by machinery operation or segregation; and finally, 3) Provide information about the residual risks to the user, and advice on protective measures.
to be implemented by the user to reduce exposure. EN ISO 14123-1 [13] defines “hazardous substance” as any chemical or biological agent that is hazardous to health. Example: substances or preparations classified as very toxic, toxic, harmful, corrosive, irritant, sensitizing, carcinogenic, mutagenic, teratogenic, pathogenic or asphyxiant. For EU countries, see also Regulation (EC) No 1272/2008.

In this context, the three pillars supporting the design of the PPLs are:

1. Anticipatory design, aligning the designs of the PPLs and the regulatory requirements of the MD [1], from the first stages of the design. The objective is to facilitate the progressive implementation of the protection measures, required for putting the PPLs into service in 2020/2022, avoiding future opportunity costs and, ultimately, facilitating the CE marking of the PPLs (TRL9).

2. Safe-by-Design, driven by the methodological approach of the MD [1], to design out hazards or minimize risks from the design stage of the PPLs.

3. Design supported on standardization, which uses standards as a reliable resource, to provide confidence, and presumption of conformity in the case of harmonized standards.

In addition, the design of the PPLs is conditioned by two relevant restrictions:

- No specific EHRSs are available for nanosafety risks, although the general principles of the EHSR 1.5.13 on hazardous substances emitted the machinery, apply to the PPLs.

- No harmonized standards are available for nano-manufacturing processes or nanosafety risks, although some existing harmonized standards can be used to conduct the design.

### Figure 1. Three-step method for risk assessment and risk reduction, in the context of Safe-by-Design of machinery (EN ISO 12100:2010) [11]

4. The standardization strategy and its practical implementation

Table 1 summarizes the main harmonized and non-harmonized standards used by the PLATFORM project to meet the design requirement 1.5.13 related to hazardous substances emitted by machinery, particularly with regard to potential emissions of CNTs produced by the PPLs. In the absence of type-C standards, the standardization strategy followed by the project was to use type A and B harmonized standards to build up the design skeleton, and complete existing gaps with other non-harmonized standards and relevant nanosafety documents.

#### 4.1. Safe-by-Design and risk assessment

The methodological framework for the SbD of the PPLs was provided by EN ISO 12100: 2010 [11]. The project PLATFORM developed a simple Microsoft Excel tool (SbD tool), to systematize the risk assessment (RA) of the PPLs. The tool combined several methodologies proposed by ISO 14121-2:2015 [18] to identify hazards, estimate and evaluate risks and propose prevention and protection measures to reduce the risk. The RA covered the exposure of workers to ENMs, in all the operation modes and stages of the life cycle of the PPLs.
Traditional methods of estimating risk designed for accidental situations are not directly applicable for long-term effects, such as exposure to ENMs [18]. Therefore, the SbD tool combined the previous methods with the "Control Banding" approach of ISO 12901-2: 2014 [20]. This approach allowed to identify hot spots in the PPLs, where potential occupational exposures to CNTs could take place.

For those protection measures that depend of the control system of the PPLs (e.g. enclosures and LEV-filtration units), the determination of the Performance Level (PL) required by safety functions was carried out with the harmonized standard EN ISO 13849-1: 2015 [12].

4.2. Risk prevention and risk reduction

The selection of prevention, reduction and control measures for health risks resulting from the emissions of the PPLs, was based on EN ISO 14123-1: 2015 [13]. This harmonized standard categorizes the measures into three hierarchical categories: a) Measures for inherently safe design, 2) Measures for risk reduction and 3) Information on residual risks and complementary protection measures. NIOSH references on occupational exposure to CNTs [24], engineering controls [25], and workplace design solutions [28], as well as ISO/TR 12885:2018 [17], were used as relevant references to customize the risk reduction measures to the specific field of CNTs. A summary of the results obtained is presented in table 4.

4.3. Verification of airborne emissions

Given that the main route of exposure to CNTs is inhalation [17], a measurement campaign was developed during the project to verify the emissions and the possible exposures to these nano-contaminants in hot spots of the PPLs. The objective was to provide recommendations for the RA and to improve the design of the PPLs and the workplaces. The measurement campaign was limited by the availability of the PPLs.

The harmonized standard EN ISO 14123-2: 2015 [14] was used as a general reference for the design of verification procedures. The harmonized standard EN 1093-1: 2008 [7] provides the general framework for the evaluation of the emission of dangerous substances in machinery and for the selection of measurement methods. However, given the complexity of measuring the emission of aerosols containing CNTs (cause) according to this standard, following EN ISO 14123-1: 2015 [13], an indirect method for the characterization of emissions through the measurement of occupational exposure (effect) was performed. For this purpose, two parallel approaches, based on the standards EN 689: 2018 [6] and EN 17058: 2018 [10], were combined.

The standard EN 698 deploys the conventional strategy established by the legislation on chemical agents at work for the verification of compliance with regulated Occupational Exposure Limits (OELs), by comparing the measurements in the personal breathing zone of worker (PBZ), with OELs. In the case of ENMs, there are still no regulated OELs. However, there are substance-specific and categorical OELs that can be used as a reference for risk management. According to ISO/TR 18367: 2016 [19], the OELs for MWCNTs range from 1 to 50 μg/m³.

On the other hand, the standard EN 17058: 2018 [10] provides the general framework to assess workplace exposure by inhalation of nano-objects and their aggregates and agglomerates (NOAA), as well as particles released from nanocomposites and nano-enabled products. In this case, the evaluation criteria are based on the results of the offline analysis (PBZ and background) and the online monitoring through Direct Reading Instrumentation (DRI).

In the measurement campaign, a combination of DRIs (particle/cm³ in the window 10nm - 10μm) and filter sampling for offsite analysis [EC-Elemental carbon (μg/m³) and CNT-structures (structure/cm³)], was used for aerosols measurement in source/area. For the measurement in the PBZ, the worker carried two personal sampling lines, for the collection of samples for analysis of EC (respirable fraction) and CNT-structures [6,9,10,26,27]. The main references used for the offline analysis were technical specifications NIOSH 5040 (EC) [26], NIOSH 7402 modified (CNTs by TEM) [27] and ISO 14966: 2002 (fibrous particles by SEM) [16].

Finally, the standard EN 1093-11: 2008 [8] provides the methodology for field evaluation of pollution control systems installed in machines (e.g. capturing devices and LEV units). Since these
systems had not yet implemented in the PPLs at the end of the project, verification of their effectiveness was postponed to a future stage of the PPLs.

5. The current ecosystem for pilot production and the roadmap to market

5.1 The PLATFORM ecosystem for pilot production

Tables 2 and 3 summarize the main characteristics of the three PPLs and the NEPs manufactured by each of them. The starting point for the design and construction of the PPLs was different in each case. PPL1 is a completely new wet manufacturing plant, that uses vacuum filtration technology to manufacture buckypapers, from an aqueous solution of MWCNTs prepared from a commercial customized waterborne dispersion. PPL2 and PPL3 combine new manufacturing modules with pre-existing modules, coming from scale-ups in previous projects. PPL2 uses hot pressing compaction technology to dope commercial prepregs with a commercial tailored macro-particulate CNT-thermoplastic formulation. PPL3 uses melt blown technology to manufacture doped veils, from a tailored CNT-thermoplastic masterbatch. PPL3 can also be used for the manufacture of tailored CNT-masterbatches, replacing the shaping module by a granulation/cooling module.

The ENM used in the three input formulations is the MWCNT - NANOXYL® NC7000™. These MWCNTs are thin and short tangled, with a carbon purity of 90%, and exhibit an average diameter of 9.5 nm, an average length of 1.5 μm and a surface area of 250/300 m²/g. MWCNTs tend to cluster into bundles or agglomerates [23].

Hots spots identified by the RA in the PPLs were: 1) the drying module and the filtration module in PPL1; 2) the scattering module in PPL2, and 3) the shaping module in PPL3. With the most accurate data that can be provided, the emissions to carbonaceous aerosols in the identified hot spots ranged from < 2 μg/m³ to 665 μg/m³ of elemental carbon (EC). PBZ concentrations measured were always < 2 μg/m³ (respirable fraction, PM4). No free CNTs were detected in any of the samples.

The analysis of these data, based on the standards EN 689:2018 [6] and EN 17058: 2018 [10], and taking the existing restrictions into account, suggests that the occupational exposure induced by the nanomaterial-activity is unlikely in the three PPLs, but further measurements are required for confirmation. The limited availability of the PPLs for production conditioned the measurement campaign (short measurement times, limited sample volumes and limited number of samples), introducing uncertainty in the determination of EC concentrations (LOD 1 μg/m³). Therefore, the results must be interpreted in this context, and are subject to new measurements when the protection measures are implemented in the PPLs (see table 4).

Currently, the TRL reached by each PPL is different: TRL 6 for PPL1 and PPL2, and TRL 5 for PPL3. Table 4 summarizes the proposed recommendations - based on the RA - for the progressive implementation of protective measures for the reduction of emission/exposure to CNTs in the PPLs, with the objective to have machines in compliance with the MD when putting into service (2020/2022). Measures have been categorized according to EN ISO 14123-1: 2018 [13]. Some recommended measures are provisional, until more effective ones are implemented.

First measures implemented in the PPLs for risk prevention and risk reduction were: 1) the substitution of hazardous substances by less hazardous alternatives [e.g. using formulations that contain short, thin, flexible, entangled MWCNTs (NC7000) [23], instead of long, thick and rigid ones], 2) the use of dust reduced forms (e.g. aqueous solutions in PPL1 and pellets in PPL2/PPL3, instead of MWCNT powders), 3) the prevention of spills and leaks (e.g. by limiting the volume of the wet stages, and the installation of retention bunds in PPL1) and 4) the use of adequate Personal Protective Equipment, according to the Safety Data Sheets of input formulations and products manufactured [23].

5.2 The roadmap to market

The three stages of the PPLs roadmap for the market, are the following (figure 2):

a) Before putting the PPLs into service (Before 2020/2022). The PPLs are excluded from compliance with the MD, until their putting into service for the commercial manufacture of NEPs (intended use), and for its commercial use by third parties (e.g. SMEs). Until then (expected between 2020 and 2022),
Table 1. Some relevant harmonized and non-harmonized standards used to conduct the SbD of the PPLs

(EHSR: essential health and safety requirement covered; T: type of harmonized standard; H: harmonized/non-harmonized; NEI: not explicitly identified in the standard; NA: not applicable; Y/N: Yes/No; OELV: Occupational Exposure Limit Value; OEB: occupational exposure band).

| Standard             | General scope                                                                                     | Application to PPLs’s design                                                                 | EHSR | H   | T   |
|---------------------|--------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------|------|-----|-----|
| **1. Safe-by-Design and Risk Assessment**                                                                 |                                                                                                 |      |     |     |
| EN ISO 12100:2010   | General principles and methodology for achieving safety in the design of machinery.             | General procedure to conduct the SbD and risk assessment of PPLs                               | NEI  | Y   | A   |
| ISO/TR 14121-2:2012 | Guidance and examples to conduct risk assessment for machinery in accordance with ISO 12100. | Risk assessment in conjunction with EN ISO 12100, ISO/TS 12901-2 and EN ISO 13849-1.           | NA   | N   | NA  |
| ISO/TS 12901-2:2014 | Control banding approach applied to occupational exposures to NOAA by inhalation.             | Risk assessment in conjunction with ISO/TR 14121-2 and EN ISO 13849-1.                         | NA   | N   | NA  |
| EN ISO 13849-1:2015 | Guidance for the design and integration of safety-related parts of control systems              | Risk assessment in conjunction with EN ISO 12100 and ISO/TR 14121-2.                          | 1.2.1| Y   | B1  |
| **2. Risk Reduction**                                                                                     |                                                                                                 |      |     |     |
| EN ISO 12103-1:2015 | Principles for the reduction of risks to health resulting from hazardous substances emitted by machinery | General framework to identify and select risk prevention and risk reduction measures.          | 1.5.13| Y   | B1  |
| ISO/TR 12885:2018   | Health and safety practices in occupational settings relevant to nanotechnologies.              | Safety practices for risk assessment and risk mitigation in workplaces.                          | NA   | N   | NA  |
| **3a. Verification of airborne emissions by measurement**                                                     |                                                                                                 |      |     |     |
| EN ISO 14123-2:2015 | Methodology leading to verification procedures for emissions of hazardous substances in machinery. | Identification of emission sources and verification procedures. In conjunction with EN ISO 14123-1. | NEI  | Y   | B1  |
| EN 1093-1:2008      | Test methods for the evaluation of the emission of airborne hazardous substances in machinery.   | Selection of methods for the measurement of airborne emissions in the PPLs.                    | 1.5.13| Y   | B1  |
| EN 1093-11:2001 (A1:2008) | Decontamination index for the evaluation of the emission of airborne hazardous substances in machinery. | Assessment of the effectiveness of pollution control systems installed in the PPLs (enclosures, LEVs, etc). | 1.5.13| Y   | B1  |
| **3b. Indirect verification of airborne emissions through the measurement of occupational exposure**          |                                                                                                 |      |     |     |
| EN 689:2018         | Measurement of occupational exposure by inhalation to chemical agents, to demonstrate compliance with OELVs | Indirect verification of airborne emissions of PPLs, by measuring occupational exposure (OELVs). | NA   | N   | NA  |
| EN 16966:2018       | Metrics to be used for the measurement of exposure by inhalation of NOAA in workplaces.          | Selection of metrics for exposure measurement of NOAA in workplaces (with EN 17058)           | NA   | N   | NA  |
| EN 17058:2018       | Assessment of exposure by inhalation of NOAA in workplaces.                                      | Indirect verification of airborne emissions of PPLs, by measuring occupational exposure (NOAAs). | NA   | N   | NA  |
| ISO/TR 18637:2016   | Available frameworks for the development of occupational exposure limits (OELs) and bands (OEBs) for NOAAs | Selection of OELs and OEBs for the assessment of occupational exposure and apply control banding. | NA   | N   | NA  |
### Table 2. Main technical characteristics of the PLATFORM ecosystem of pilot lines (PPLs)

| Pilot Line | Owner | Location | Manufacturing technology | Manufacturing modules | Production capacity (m²/day) | TRL | Input formulation and CNT content (wt.%) |
|------------|-------|----------|--------------------------|-----------------------|------------------------------|-----|-----------------------------------------|
| PPL1       | Tecnalia | Spain   | Vacuum filtration        | Dispersion, filtering, washing, drying and final conditioning | 2               | 6   | CNT-waterborne dispersion (< 5%)     |
| PPL2       | Adamat  | Greece   | Hot pressing compaction  | Unrolling, powder scattering, powder compaction, cooling, rolling and packaging | 500             | 6   | CNT-thermoplastic powder (< 20 %)    |
| PPL3       | TMBK    | Poland   | Melt-blown               | Drying, feeding, plasticizing, extrusion, shaping and conditioning | 50              | 5   | CNT-thermoplastic masterbatch (10-20%) |

### Table 3. Main technical characteristics of the CNT-based nano-enabled products manufactured by the PPLs:

| Pilot Line | Manufactured CNT-based nano-enabled product | CNT content (wt%) | Format | Length and width (m/mm) | Thickness (μm) | Areal weight (g/m²) |
|------------|---------------------------------------------|-------------------|--------|-------------------------|----------------|---------------------|
| PPL1       | Buckypaper Continuous self-supporting thin sheet/membrane, consisting of 100 % of entangled CNTs | 100               | Rolls, flexible formats | 0.08 -100 75-300 | 30-200          | 30-200              |
| PPL2       | Doped prepreg (FXply™) Commercial carbon fiber prepregs doped with CNTs | ≤2                | Rolls, flexible formats | Same dimensions as the commercial prepreg | Same thickness as the commercial prepreg | 304                 |
| PPL3       | Doped veils Ultralight polymer nonwoven fabric, consisting of thin fibers bonded together thermally | ≤3,5              | Sections 1,1 m² | 1,8 600 | 30-150          | 15-200              |
Table 4. Summary of proposed protection measures to reduce exposure to CNTs, until the PPLs are put into service, categorized according to EN ISO 14123-1 [13]. Measures in italics have already been implemented.

| Pilot Line (PPL) | PPL1 | PPL2 | PPL3 |
|------------------|------|------|------|
| Hot Spot module  | Drying | Filtration | Scattering | Shaping |
| Airborne emission| Drying aerosol | - | Particulate aerosol | Particulate aerosol |
| Non-airborne emissions | Liquid containing CNTs (2% wt.) | - | - | - |
| EC-Source (μg/m³) (1) | < 2 | 665 | < 2 |
| EC-PBZ (μg/m³) (2) (3) | - | - | < 2 | < 2 |
| SEM/TEM (3) | No free CNTs | No free CNTs | No free CNTs |

**RISK PREVENTION AND RISK REDUCTION MEASURES (EN ISO 14123-1)**

1. Elimination or prevention of risks (Inherently safe design)

2. Risk reduction

2.1 Reduction of emission

- Use of CNT dust-reduced forms
  - CNTs aqueous solution instead CNTs in powder form
  - CNTs masterbatch instead CNTs in powder form

2.2 Reduction by ventilation or other engineering means

- LEV from almost complete to partial enclosure
  - Enclosing drying module + LEV + filter
  - Enclosing scattering module + LEV + filter
  - Enclosing die head area + LEV + filter

- LEV without enclosure
  - Mobile LEV Unit with filtration (4)
  - Mobile LEV Unit (4)

- General ventilation
  - Improve the general ventilation system

2.3 Reduction of exposure by machinery operation or segregation

- Separate room
  - Dedicated room + filtration

3. Information and other protective measures regarding residual risks by the user to reduce the exposure

- Engineering measures
  - Portable vacuum unit for cleaning task

- Administrative measures
  - Change the working procedure for cleaning operations

- Personal Protective Equipment (PPEs)
  - PPL1: protecting gloves (nitrile), cloth, respiratory equipment (P3) when the product is dried, safety glasses. PPL2/PPL3: resistant gloves, respiratory equipment (P3) when the product is heated, safety glasses. Ensure proper maintenance of PPEs.

[(1) EC: Elemental Carbon; (2) Respirable fraction (PM4); (3) PBZ: Worker Breathing Zone; (4) Provisionally, while the most effective measures are implemented;]
pilot production activities will be dedicated to testing, adjusting and improving the manufacturing processes; then, the PPLs can be considered machinery for research purposes (Article 1.2/MD). However, during this period, companies where the PPLs are installed should implement additional OHS measures in accordance with current legislation, to ensure the safety and health of the workers using PPLs/work equipment not yet compliant with the MD. OHS regulations refers to Directives 89/391/CEE (OHS framework), 2009/104/EC (Work equipment) and 98/24/EC (Chemical agents), among others. In practice, both for the MD and for the OHS Directives, the legislation that applies are the national regulations that transpose these Directives in the Member States (Spain/PPL1, Greece/PPL2 and Poland/PPL3).

![Figure 2. Roadmap to the market for the PPLs and main regulations involved](image_url)

b) Putting the PPLs into service (2020/2022). The transition from pilot production to commercial production will involve the putting into service of the PPLs and, therefore, compliance with all the applicable requirements of the MD. The putting into service will require the manufacturers of the PPLs to complete six basic requirements: 1) Ensure that the relevant EHSRs that apply to the PPL have been adequately satisfied, not only those corresponding to the requirement 1.5.13 (emissions of hazardous substances), but also the rest of EHSRs applicable to the PPL, such as electrical mechanics, control systems, etc.), 2) Prepare a technical construction file of the PPL that will gather all the information to demonstrate the compliance with the MD; 3) Provide the necessary instructions, 4) Carry out the selected procedure for the evaluation of the conformity of the PPL (Self-assessment), 5) Draw up the EC declaration of conformity, and finally, 6) Affix the CE marking on the PPL.

c) After putting the PPLs into service (after 2020/2022). The employer is now owner of a work equipment in compliance with the MD (Product regulation). To ensure the safety of the PPL made available to workers, he must comply with the use and maintenance requirements established by the Directive 2009/104/EC on work equipment (Use regulation). In addition, from the point of view of the MD, the introduction of substantial modifications in the PPLs (e.g. modifications that result in new hazards/risks), may result in new legal obligations for the user with regard to the MD; since a PPL substantially modified may be considered as a new machine, and require a new procedure for conformity in accordance with the MD [1]. In these cases, the review of the RA can provide clear evidence for making decisions about the degree of modification of the PPL (substantial or not).
6. Conclusions and beyond

"Safe-by-Design" principles established by the MD (Safety Integration), provided a robust framework to conduct the design, construction and improvement of the PPLs.

The PPLs are innovative machines, on the border of the state of the art, without specific design standards (type C). Type A and B harmonized standards provided a first skeleton for the design of the PPLs, and other non-harmonized standards and technical specifications were integrated to complete the design. However, this gap forced designers to make decisions based on their best knowledge, engineering practices and professional judgment, on the application of existing standards and guidelines to the design of the PPLs.

The lack and/or gaps in specific regulation in the field of nanotechnology and nanomaterials, can be a barrier to the scaling and introduction into the market of nano-manufacturing processes, such as the PPLs, generating opportunity costs and additional responsibility for manufacturers and end users.

In this regulatory context, standards are instruments that provide confidence to manufacturers, users and authorities, reducing the level of uncertainty in an ambiguous regulatory context, such as nanotechnology and nanomaterials. In this respect, the PLATFORM project is promoting the development of the standardization document "CEN/WS 89 - Platform - Guidelines and best practices for sustainable production of carbon nanotube-based nano-enabled products", which tries to export the experience gained in the design of this type of manufacturing processes through standardization.

When putting into service, the final design of the PPLs must satisfy, not only the EHSR referred to the emissions of hazardous substances (ENMs and other pollutants such as VOCs, dust), but also the rest of applicable EHSRs, such as mechanical, electrical, etc.

A new H2020 project (OASIS, GA 814581) will build up an Open Innovation Test Bed in the field of light weight nano-enabled multifunctional composite materials and components, integrating the three PPLs into a nano-manufacturing ecosystem of 12 pilot lines. This will allow to progress and consolidate the design of the PPLs, to ensure compliance with the MD when putting them into service, in 2020/2022.

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