

# Chapter 3

## The Life Cycle of Engineered Nanoparticles

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**Abstract** The first years in the twenty-first century have meant the inclusion of nanotechnology in most industrial sectors, from very specific sensors to construction materials. The increasing use of nanomaterials in consumer products has raised concerns about their potential risks for workers, consumers and the environment. In a comprehensive risk assessment or life cycle assessment, a life cycle schema is the starting point necessary to build up the exposure scenarios and study the processes and mechanisms driving to safety concerns. This book chapter describes the processes that usually occur at all the stages of the life cycle of the nano-enabled product, from the nanomaterial synthesis to the end-of-life of the products. Furthermore, release studies reported in literature related to these processes are briefly discussed.

**Keywords** Life Cycle • Nanomaterials • Nanocomposites • Release • Risk

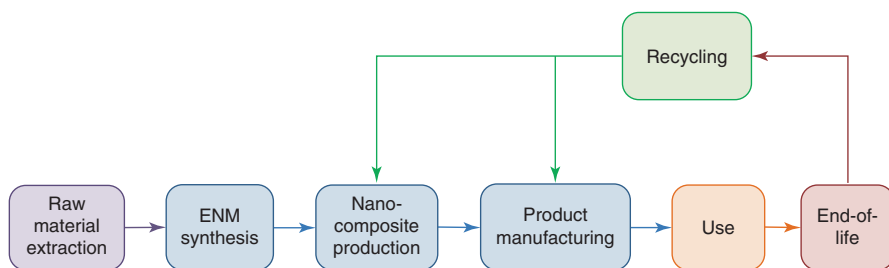
### 3.1 Introduction

The increasing use of nanomaterials in industrial and consumer products results in a potential risk for workers, consumers and the environment.

A starting point for any comprehensive risk assessment or life cycle assessment is the identification of all relevant life cycle steps, so that all scenarios with a potential risk can be evaluated. The life cycle is totally product-dependent, as each product has its own manufacturing processes, uses and waste treatment and, so, its own hotspots for nanomaterial release and associated risks. This chapter presents a brief overview of the most common processes that take place at different steps of a product life-cycle (Fig. 3.1) and highlights the potential contribution of each step to the release of nanomaterials and associated risk.

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**Fig. 3.1** Life-cycle of nano-enabled products

It is also important to clearly identify all the processes implied in each life cycle stage to know which mechanisms drive to release. Apart from release quantification, the form of release (isolated particles, aggregates, embedded in a matrix, surface modified by hydration or oxidation, etcetera) may be determinant for the hazard evaluation.

### ***3.1.1 Production Levels of Engineered Nanomaterials***

Some nanomaterials, such as carbon black and silica, have been industrially used for decades. However, during the last decade, new materials and modifications have allowed a dramatic expansion of nanotechnology. Despite the multiple materials that are being investigated at a research scale, at this moment it is estimated that nanomaterials produced at an industrial scale belong to only around 20 chemical classes [1]. At the moment, any attempt to determine nanomaterials production has to be based on estimations as they do not have to be reported. Only France and Denmark have recently regulated nanomaterials in products, so these nano-additivated products have to be registered and labelled in order to inform the consumers [2, 3].

Attempts to estimate the production levels and applications of nanomaterials have been based on information provided by industry through surveys. Sometimes the data collected relates to production capacities and sometimes to actual production amounts and the geographical area under scope also differs [4–6]. One of the most thorough recent surveys is that of Piccino et al., who send a survey to industrial representatives from companies producing or using nanomaterials to estimate the worldwide or Europe-wide production of such materials [7]. A considerable large variability among answers by different industrial representatives reflects the general uncertainties related to the actual worldwide production volumes. However, there was general agreement that silica, titanium dioxide, zinc oxide, carbon nanotubes, iron oxides, aluminium oxides, and cerium oxides are the nanomaterial types with highest production volumes. The median production quantities for each of these nanomaterials ranged between 55 and 5500 tonnes per year worldwide [7], depending on the material. These values are also consistent

with estimated annual production volumes in China in 2012, which ranged from 200 to around 1300 tonnes for titanium dioxide, zinc oxide, aluminium oxide, zirconium oxide, and silver [8].

According to the French registry [9], the quantities produced and imported in 2014 are around 274,000 and 122,000 tones, respectively. Carbon black and silicon dioxide are the category of substances with largest produced or imported quantities, both above 100,000 t/year. These are followed by calcium carbonate and titanium dioxide, with volumes ranging 10,000–100,000 t/year. Other materials reported to be produced or imported in amounts above 1000 t/year are aluminium oxide, boehmite ( $\gamma$ -Al(OH)O), calcium 4-[(5-chloro-4-methyl-2-sulphonatophenyl)azo]-3-hydroxy-2-naphthoate, reaction mixture of cerium dioxide and zirconium dioxide, polyvinyl chloride, and magnesium silicate.

## 3.2 Engineered Nanomaterials Synthesis

The synthesis of ENMs is the step of a nano-enabled product life cycle that has received the highest attention in the literature in relation to the potential risks for human health [10–13]. By contrast, the potential release of ENMs to the environment during this step has received little attention and it is commonly assumed to be low, though this completely depends on the procedures used during the production, cleaning and maintenance [14–17].

Due to the novelty of the field and the continuous research in the development of new nanomaterials, multitude of synthetic methodologies can be found in the literature. Most of these processes are adequate for laboratory scale and even pilot scale synthesis, but completely unworkable at industrial scales. Synthetic methods are often divided in top-down and bottom-up methods [18].

- **Top-down methods.** The successive cutting or slicing of bulk materials into nanomaterials play an important role in industrial synthesis of nanostructures that need specific shapes/sizes such as nanotransistors. Lithography, milling and attrition are the most common top-down processes used at the moment. The principal disadvantages of top-down approaches are the internal stress and the imperfection introduced in the surface structure due to the use of so energetic techniques. Such imperfections may have dramatic effect over surface chemistry and physical properties of such prepared nanomaterials.
- **Bottom-up methods.** Atom by atom chaotic building of nanomaterials comprises most of synthetic methods as these are also the most common procedures in materials science. The main disadvantage of these methods is that usually a distribution of sizes is obtained, but compositions are more homogeneous than in top-down approaches.

Synthetic methods are usually divided in dry or wet synthesis; open or enclosed reactions; and gas-, solid- or liquid-phase reactions. Moreover, depending on the synthetic method and the material, different purification steps may be necessary

and, in the case of coated particles, one or more modification steps with extra purification are needed. To facilitate comprehension, this section is organized first by nanomaterial group, and then by synthetic methods.

### 3.2.1 Carbon Based Nanomaterials Production

Sizable quantities of carbon based nanomaterials can be produced using various methods; among them, plasma based, thermal and hydrothermal syntheses processes are the most used techniques [19–22].

The two most common plasma methods in the literature are Arc Discharge and Laser Ablation. Arc Discharge Method consists in passing current between two graphite electrodes under helium, hydrogen or methane at low pressure in presence of transition metal based catalysts [23–28]. This causes vaporization of graphite that condenses over the cathode (and walls of the reactor). Carbon nanotubes (CNT) can also be produced by Laser Ablation, which is similar to Arc Discharge but the energy is provided by a laser. This laser vaporizes graphite and catalyst, so that nanocatalysts are formed and the carbon nanomaterials grow over them [29–35].

Thermal synthesis methods are also very abundant in the literature for the production of CNT. Carbon Vapour Deposition (CVD) consists in the decomposition of a carbon source (usually a hydrocarbon over a transition metal catalyst). Both type of carbon source and catalyst affect in the CNT growth. Carbon based nanomaterials can also be produced by sono- or hydrothermal methods, which consist on the heating of a hydrocarbon/water mixture under pressure in the presence of a catalyst (usually Ni) [36–38].

These methods usually produce low quantities of carbon based nanomaterials (fullerenes, SWCNT, MWCNT, etcetera) mixed with other allotropic forms of carbon. The conditions used during the synthesis favour one form over the others, but purification steps are always necessary.

Graphene, graphene oxide and derivatives are synthesized very differently [39–43]. The bottom-up approaches used for the rest of carbon based nanomaterials are modified to get 2D carbon layers over a support, which has to avoid 3D growth [44]. Graphene can be also produced by top-down approaches. The purest and most perfect graphene is produced by exfoliation of graphite [45–47], graphite oxide (followed by reduction) [48–50] and carbon nanotubes [51].

Carbon based nanomaterials surface can be modified to improve their dispersibility, their compatibility with a matrix or to functionalize them to add chemical groups that can later react or bond to any other entity, such as antibodies [52, 53], quantum dots [54] or gold nanoparticles [55]. These modifications are usually based on the following approach. In a first step, the nanomaterials are oxidized by a hydrothermal process (i.e.: sonication in presence of diluted nitric acid) that causes defects on the surface. Then, the hydroxyls and carboxylic acids formed are used for subsequent functionalization by traditional chemical reactions. Other strategies include direct arylation, carbene or nitrene addition or Friedel-Craft acylation.

Carbon nanotubes and nanofibers have centred most of the attention of hygienists and toxicologists due to their fast increase in production volume and observed dustiness [16, 56–64]. Most published studies on occupational exposure to CNT show that, as the synthesis processes take place in closed reactors, most of the exposure occurs during material recovery and during cleaning and maintenance operations [57, 65–67].

### 3.2.2 *Metallic Nanomaterials Production*

Metallic nanomaterials (MNM) are traditionally synthesized by the reduction of a precursor under controlled conditions and in the presence of a stabilizer [68]. The solvent, conditions and stabilizers used depend on the element of the NP and on the purpose. Wet syntheses have been traditionally considered of lower risk in terms of occupational exposure due to lower aerosol formation compared to the work with powders.

Noble and semimetallic nanoparticles (Ag [69], Au [70], Pt [71], Pd [72, 73], Ru [74], Rh [75], Ir [76]) are usually industrially synthesized by reduction of a precursor salt in water (such as  $\text{HAuCl}_4$  or  $\text{RuCl}_3$ ). Once the colloids are synthesized, they can be directly functionalized *in situ* or phase-transferred to an organic solvent for further surface functionalization when necessary [77]. The conditions and precursors used completely determine the results in terms of size and morphology [78]. Although less common, all these materials can be produced by other methods, such as electrochemical deposition, physical synthesis or sol-gel method.

More reactive metallic nanoparticles are synthesized by similar approaches (which can be also used with noble metals), but under more controlled conditions (air-free atmosphere, organic solvents, ionic liquids, etcetera). The most common strategy consists on direct reduction or decomposition of organometallic compounds or metallic carbonyls, such as  $\text{Fe}(\text{CO})_5$ ,  $\text{Co}_2(\text{CO})_8$  or  $\text{Ru}(\text{cod})(\text{cot})$ . Initially these syntheses used to produce polydisperse NP, but their optimization has improved the control of size and nowadays they are used for the synthesis of zerovalent nanoparticles of several metals: Fe, Co, Ru, Ir, Au, Ni or Rh, and different types of mixtures (core-shell, alloys...) [79, 80]. Surface modification of these materials is usually done *in situ*, and the organic modifier is used as stabilizer.

Though most metallic nanoparticles are produced by wet processes, occupational exposure cannot be neglected [81–86]. Release of metallic nanomaterials to the environment has received some attention, particularly Ag-NPs release, but most studies use assumptions to estimate the release during the MNP synthesis rather than actual measurements [87].

### 3.2.3 *Oxide Based Nanomaterials Production*

Several physical and chemical routes for the synthesis of nanometal oxides (NMOx) have been reported. Solution routes are the most widely used at laboratory scale, as they need more easily accessible set-ups and allow having a better control size and

shape. In contrast, at the industrial scale, gas phase methods are the most commonly used as they are usually cleaner and better conversions are obtained. Moreover, they can be used for the deposition of thin films or nanostructures over a particular substrate. Both solution and gas-phase methods are widely used to produce different NMOx such as ZnO [88], TiO<sub>2</sub> [89], Fe<sub>x</sub>O<sub>y</sub> [90], CeO<sub>2</sub> [91, 92], ZrO<sub>2</sub> [93], Cu<sub>x</sub>O [94], Al<sub>2</sub>O<sub>3</sub> [95], SiO<sub>2</sub> [96–98], Co<sub>x</sub>O<sub>y</sub> [99], etcetera.

Solution routes are based on the decomposition of a salt or alkoxide precursor in solution, normally by means of a source of energy, to form the nanomaterial. Thereafter, this nanomaterial is separated from the solution by centrifugation, nano-filtration or other nano-appropriate techniques. Some examples of solution routes include solvo-/hydrothermal method, precipitation method, electrochemical synthesis, sonochemical method, sol-gel method and microemulsion.

In gas phase methods, metal vapour is produced by thermal, laser ablation, electron beam, ion beam, molecular beam or by vaporizing and dissociating any metal precursor. This metal vapour reacts with oxygen to produce the metal oxide that is deposited on the bottom and the internal walls of the reactor.

Different surface modification strategies exist. The most commonly reported are: (i) chemical functionalization of the surface by bonding of a silane derivate, which renders a very stable modification; (ii) addition of compounds that have affinity for NMOx surfaces, such as carboxylates or phosphates; and (iii) polymer grafting [100].

Occupational exposure to nanometal oxides in production facilities has not received much attention in the literature. In general, the reported studies show that the exposure is due to specific operations such as reactor opening, material recovery or cleaning and maintenance [86, 101–104].

### 3.2.4 *Quantum Dots Production*

Quantum dots (QD) can be produced by several methods, both top-down and bottom-up approaches [105–107]. The advantage of top-down processes is that very well-defined QD are produced. This is necessary, for instance, when producing nano-transistors for computing. However, these high energy methods usually result on physical and chemical damage to the particle surface. On the other hand, bottom-up approaches produce smaller and purer particles, as necessary in sensing applications.

Lithography, reactive-ion etching and wet chemical etching are the most commonly used top-down processes. The bottom-up processes are very similar to the ones explained for nanometal oxides synthesis.

The surface of most quantum dots is very easy to modify, as several functional groups have affinity for them (thiols, amines, carboxylic acids, etcetera). Thus, a multitude of papers report QD functionalization with biomolecules (DNA, RNA, proteins...) [108, 109], polymers [110] or other nanomaterials [111].

Despite of the high concern about quantum dots toxicity [106, 112–114] and their potential impact over workers and the environment, there is a lack of experimental data on the exposure to quantum dots during synthesis steps. Moreover, the only study that was found at the moment focused on a lab-scale synthesis [104].

### 3.2.5 *Polymeric and Ceramic Nanofibres Production*

Nanofibres are usually produced by the electrospinning method [115–119]. This method produces non-woven fabrics, in which the fibres are randomly oriented and connected by physical entanglements or bonds, without any knitting or stitching. In the electrospinning method, polymers are usually dissolved in a proper solvent (or molten) and the nanofiber is produced by high voltage. Recently, this technique has been extended to ceramic nanofibres synthesis. In this case, polymeric nanofibres loaded with ceramic precursors are prepared by electrospinning and, later, combusted to render the ceramic nanofiber.

Nanofibres production is usually done in closed conditions and neither occupational nor environmental exposure have been reported during this life cycle step. All the publications have focused on secondary manufacturers [120].

## 3.3 **Nanocomposite Production**

The incorporation of the nanomaterial in or on a matrix is a key step in nano-enabled products life cycle (except on those cases where nanomaterials are a final product by themselves, such as nanocatalysts). Nanotechnology has greatly progressed during the last two decades and, nowadays, we can find applications for almost any type of nanomaterial in any type of matrix.

A nanocomposite is a multiphase solid material that has at least one of the phases in the nanoscale. The main difference between nanocomposites and traditional composites is the high surface of contact between the phases in the first case. The addition of nanomaterials to solid matrices produces materials with enhanced, or even completely new, attributes, such as conductive polymeric matrices, electroluminescent metals, semiconductor ceramics or photo-luminescent textiles. The properties and quality of the resulting nanocomposite depend on the constituents of the composite, but also on the degree of dispersion and homogeneity of the different phases, which depend on the compatibilization between the phases and the mixing/addition methods.

In addition, nanomaterials can be also added to the surface of a material to obtain new or improved surface properties. This surface addition can be done by in situ nanocomposite formation, such as surface treatment of ceramic tiles with a solution of nanosized titania and a resin that is later dried [121]; by physical or chemical

attachment of the nanomaterial to the surface, such as textiles with silver nanoparticles bonded to the fibres [122]; or by direct deposition of a thin layer over the surface, such as solar cells of nanosized TiO<sub>2</sub> prepared by direct CVD over the cell surface [123].

Other types of nano-additivated formulations include nanomaterial dispersion in emulsions, such as paints or cosmetics, or mixtures of non-consolidated solids, such as catalytic mixtures for gas emission treatment.

The literature on this field is very broad (8,280; 88,000 and 75,000 results in Google Books, Google Scholar and Web of Science, respectively, when looking for *all in title: nanocomposites*).<sup>1</sup> Due to the scope of this book, this chapter will only provide an overview on the production processes for the main types of nanocomposites: polymeric, ceramic, metallic and textiles. If needed, the reader can expand this information in some of the existing reviews [124–126].

### 3.3.1 Polymeric-Matrix Nanocomposites

Research on polymeric nanocomposites has exponentially grown in the last decades and this has been reflected in an increase in the number of products launched to the market based on such materials, from conductive polymeric materials to artificial tissues.

Polymeric matrix nanocomposites can be synthesized by different techniques that can be divided in three major groups: solution casting, melt blending and *in situ* polymerization [127–130]. Solution casting consists in the dissolution of the polymer and dispersion of the nanomaterial in a solvent (usually using ultrasonication). Then, the nanocomposite is obtained by removing the solvent. In the melt blending method, the polymer and the nanomaterial are intensively mixed in an extruder or a mixer at a temperature that allows polymer mobility. *In situ* polymerization consists on the mixture of the nanomaterial and monomers (in solution or not) under conditions that favour the polymerization. Polymerization can be catalyzed by the nanomaterial itself (i.e. silicate layers promote intercalated monomer polymerization) or by the addition of polymerization catalysts. Moreover, the nanomaterial may be coated with vinyl moieties where polymerization can start.

The choice of the synthesis method and its conditions completely depend on the polymer and on the type of nanomaterial. A good compatibility of the polymer and the nanofiller is critical for a homogeneous physical-chemical behaviour of the composite and to reduce nanomaterial release in following life cycle phases [131]. The most common strategies used to improve such compatibility are the use of additives that act as a surfactant between the nanomaterial and the polymer [132–135], and the surface modification of the nanomaterial to make it more compatible with the polymer [135–138].

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<sup>1</sup> Search done on the 21st January 2015.



### 3.3.2 *Ceramic-Matrix Nanocomposites*

Nano-additivation of ceramic materials has resulted in the development of new materials with enhanced properties. The most important disadvantage of ceramic materials is their fragility, and the addition of nanomaterials is mainly used for the reinforcement of the ceramics that allows their use in new applications (i.e. armours, surgery materials or artificial bones). In addition, nanomaterials can also confer other properties that make these materials useful in fields such as optical, electronic or sensing [128, 139]. Ceramic matrices were traditionally reinforced with metallic particles [140–142], but nowadays one can find in the literature ceramics reinforced with carbon based nanomaterials [143, 144], nanometal oxides [145, 146] or quantum dots [147].

Three methodologies are basically used in the processing of ceramic matrix nanocomposites: powder process, polymer precursor process and sol-gel process [128]. Powder process consists of the mixing of the different materials that are thoroughly milled together in wet conditions; later the mixture is dried and consolidated, usually by pressure or moulding. This process is simple but results on a heterogeneous material. Polymer precursor process is similar, but the nanomaterial precursor is added to a polymer that is later pyrolyzed. Sol-gel process consists in the hydrolysis and condensation of molecular precursors dissolved in organic media to form a sol-gel, which is later dried and consolidated.

### 3.3.3 *Metal-Matrix Nanocomposites*

Particulate reinforced metal-matrix composites have been used for decades [148], but the reinforcement with nanomaterials has been developed recently and metal-matrix nanocomposites are still in their infancy [149]. The main advantages of metal matrices are their inherent thermal stability, resistance to abrasion, and thermal and electrical conductivities. But their development was strained by their cost and the difficulties of preparation [128, 149, 150]. The nano-additivation of metal matrices confers a combination of ceramic and metal properties to the material. This makes the material ideal for multiple applications, such as structural materials in the aeronautic industry or in light energy conversion.

Several methods for metal-matrix nanocomposites processing are described in the literature, including vapour phase processing, spray pyrolysis, powder metallurgy, solidification, chemical and deformation processes. The most used and cheapest method is solidification, which consists on the melting of the metal and the nano-reinforcement and rapid solidification of the melt by different processes. Liquid infiltration is similar, but in this case only the metal is melt and surrounds the nanomaterial. The homogeneity of the mixture can be improved by ultrasounds. The other methods are similar to the ones used in nanomaterial synthesis (sol-gel synthesis, CVD, spray pyrolysis, etcetera) (see Sect. 3.2).

### ***3.3.4 Nano-additivated Textiles***

Nanomaterials can be integrated in the textiles in different phases of their fabrication, which leads to different types of nano-additivated textiles: (i) Nanotextiles, when the nanomaterial is added once the fabric is produced, most of the products falls in this category; (ii) Nanocomposite textiles, when the material used to make the fibres is a nanocomposite; and (iii) Nanofibrous materials, when they are made from nanofibres (woven or non-woven) [151]. Nanomaterials are added to textiles to provide new or improved properties. The most common ones are antimicrobial activity and UV-filtering, but they are also used as flame retardants, water repellent, static protection, electrical conductivity, enhanced resistance or strength, photo- or electro-luminescent, self-cleaning, etcetera.

Several methods have been used for surface modification of fibre-based materials, such as textiles and membranes [152–155]. Usually they involve small modifications during the fabric processing; the nanomaterial is added as any other additive by methods such as impregnation, roll-to-roll and pad-dry-cure. The main problem of these methods is that the nanomaterials are usually not well fixed to the fabric and majorly released during the washing process [156–161]. In order to minimize the release of nanomaterials, binders and functionalized particles are used to improve particle affinity for the textiles. Ultrasounds, UV irradiation, plasma-treatment and ion-beam-assisted deposition are very effective for the surface modification of textiles, but impractical for large scale manufacturing (several preparatory steps, time-consuming and costly).

Nanocomposite fibres have emerged in the last decade as a very interesting material for nano-enabled textiles processing. In this case, nanocomposite material is produced as any other polymer-matrix nanocomposite (see Sect. 3.3.2). The main challenge is to get nano-reinforced polymers that can be processed as fibres and that, later, do not reduce the mechanical properties of the fabric.

Textiles and other non-woven products (such as filtering membranes) can be made of nanofibres. Nanofibres are produced by electrospinning (see Sect. 3.2.5) and can contain pure polymer(s) or nanocomposites. At the moment, nanofibres are not woven at industrial scale and are usually used as additive over other fibres [151]. Non-woven nanofibres are used as layer and barrier materials [115, 117, 118, 162, 163].

### ***3.3.5 Occupational and Environmental Exposure During Nanocomposite Production***

Most of the available studies on occupational exposure to nanomaterials focused on the nano-additivated material preparation. Nanomaterial synthesis is usually done in close reactors and workers are basically exposed during the nanomaterial recovery and during cleaning and maintenance. In contrast, weighting, pouring, and mixing of nanomaterial and bulk materials (common steps during nano-additivated of

materials) are usually done in open conditions, and can involve big amounts of nanomaterials, so that the exposure during this step is potentially high [10, 12, 164–166]. This has been corroborated by exposure monitoring campaigns in workplaces as described in different reviews [167, 168] and other later studies [169–175].

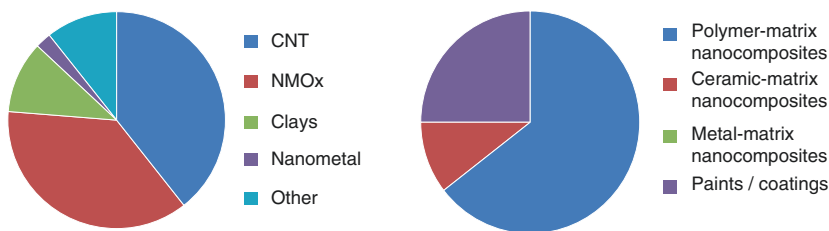
On the other hand, nanomaterial release to the environment during this step is usually considered unlikely. Once the process is finished, the nanomaterials are embedded in a matrix or a mixture, so their recovery is easier and also the waste treatment [16].

### 3.4 Product Manufacturing

Product manufacturing involves a series of processes to convert the nano-additivated material into the final product. Machining is necessary to obtain final products with specified dimensions, surface finishing and tolerances. Most of the machining processes are physically aggressive and can lead to nanomaterial release. Although these processes are carried out by machines, they usually need an operator, sometimes in close and long contact to the material (i.e. sewing). Some examples of machining processes include soldering, welding, cutting, sewing, grinding, shredding, sanding, punching and drilling. Moreover, one has to consider that several of these processes may be necessary to get the final product, which may mean the product manufacturing divided in several phases that can even occur in different companies or locations.

Nanomaterial release from nanocomposites during the machining processes has received special attention in the literature in comparison to other processes during manufacturing and use stages. Indeed, almost half of the papers identified in a recent review on nanomaterial release from nanocomposites focused on machining processes [176].

Most of these studies are focused on CNT- and NMOx-based nanocomposites, with almost no attention to nanometal-based nanocomposites. Regarding the matrix, most of the studies focus on polymeric nanocomposites, probably because they are the ones with highest production volumes (Fig. 3.2) [176, 177].



**Fig. 3.2** Summary of reviewed papers by nano-reinforcement, *left*, and by base material, *right*, submitted to machining processes (Based on Froggett et al. 2014)

Nanomaterial release from nanocomposites during the machining processes can be studied simulating real operations or using standardized protocols. Non-standard studies of cutting/sawing [120, 178, 179], grinding [120, 180], shredding [181], sanding [65, 120, 182–186], and drilling [187–190] under different conditions (wet/dry, hot/cold, etcetera) are found in the literature. Studies based on standard protocols usually focus on abrasion, using a Taber abraser [131, 185, 191–198]. Regardless on the type of simulation, most studies analyze the released material and usually conclude that part of the matrix released contains nanomaterials embedded. Only four publications report significant release of isolated nanomaterial [120, 131, 186, 192]. It is important to notice that most of these sanding/abrasion studies do not clearly distinguish between abrasions due to aging or industrial processes.

From the publications mentioned in this section, it can be concluded that the matrix play a more important role than the nano-reinforcement on the overall degradation caused by the machining processes. Moreover, good dispersion of the nanofillers in the matrix could reduce the release of isolated nanomaterials [131].

### 3.5 Use Phase

At the moment, the major usage of nanomaterials is considered to be at the industrial level. For example, they are used as catalysts, membranes, and as additives or technical components of materials in various application fields. In addition, some nano-enabled products are addressed to professionals and consumers. There is no doubt that the diversity of applications of ENMs in commercial products has grown extensively over the past decade, and continues to grow rapidly [199]. However, the actual distribution of nanomaterials over different product categories is largely unknown. According to the (US) Nanotechnology Consumer Products Inventory [200], which has been updated very recently, the number of consumer products that are claimed to contain nanomaterials has increased from 54 products in 2005 to 1628 products in 2013. Although these numbers are likely to reflect real trends, their accuracy is questionable because tracking products that contain nanomaterials is rather challenging. With a few exceptions, current labeling regulations do not require that the nanomaterial be listed specifically as an ingredient. On the other hand, some products on the market with the claim of “nano” may neither contain nanomaterials nor be produced with nanotechnology. Depending on the area of application, interest in reporting the use of nanomaterials can differ, which could result on biased estimations on the main area of application if based on reported use.

Such lack of information regarding the real use of nanomaterials in consumer products may change in the coming years. First, some regulations, such as those affecting cosmetics and food ingredients in the EU are currently already requesting producers to label nanomaterials in their products [201, 202]. And second, some countries established compulsory registries of nano-enabled products.

**Table 3.1** Distribution of sectors of use among the total declared in 2014

Code	Descriptor	Occurrence	Percentage
SU1	Agriculture, forestry, fishery	6417	58.28
SU10	Formulation [mixing] of preparations and/or re-packaging (excluding alloys)	2131	19.36
SU0	Other	877	7.97
SU17	General manufacturing, e.g. machinery, equipment, vehicles, other transport equipment	330	3.00
SU4	Manufacture of food products	233	2.12
SU24	Scientific research and development	227	2.06
SU11	Manufacture of rubber products	161	1.46
SU12	Manufacture of plastics products, including compounding and conversion	161	1.46
SU9	Manufacture of fine chemicals	119	1.08

Adapted from Ministère de l'Écologie du Développement durable et de l'Énergie (2014)

France was the first European country to require the identification of ‘substances with nanoparticle status’ that are produced, imported, distributed, or formulated from the 1st of January 2013 (Article 185 of the French Environmental Code [3]). Since June 2014, the Danish EPA also requests the reporting into the nano-product register of mixtures and articles that are intended for sale to the general public and which contain nanomaterials. They did, however, limit the type of products that should be reported on the basis of their potential to represent a risk to the user or the environment. Therefore, reporting is only requested for products where the nanomaterial itself is released under normal or reasonably foreseeable use or where the nanomaterial itself is not released but substances in soluble form that are classified as carcinogen/mutagen/reprotoxic (CMRs) or environmentally dangerous substances are released from the nanomaterial. In addition, some type of products (mostly those covered by specific product risk assessments, such as medicines or cosmetics) are also exempt [2].

A recent report outlines the results of the two first declaration periods in France (up to 1st June 2014) [9, 203]. Table 3.1 includes the sectors of use with more than 100 declarations in 2014. The sectors with the highest number of declarations were agriculture, forestry and fishery, and formulation [mixing] of preparations and/or re-packaging (excluding alloys) with 58 and 19 of the declarations, respectively. Regarding chemical product categories, the most commonly reported are: (1) coatings and paints, thinners, paint removers, (2) cosmetics and personal care products, and (3) plant protection products, altogether accounting for almost 70 % of the chemical product categories registered (Table 3.2). Finally, among the registered articles, the most frequently reported categories were rubber articles (AC10), machinery, mechanical appliances, and electrical/electronic articles (AC2), plastic articles (AC13), vehicles (AC1), and other articles with intended release of substances (AC30).

Future updates of this registry and other registries will provide more realistic estimates of the global production of nanomaterials and their main applications.

**Table 3.2** Distribution of chemical product categories among the total declared in 2014

Code	Descriptor	Occurrence	Percentage
PC9a	Coatings and paints, thinners, paint removers	631	24.0
PC39	Cosmetics, personal care products	605	23.0
PC27	Plant protection products	575	21.9
PC13	Fuels	216	8.2
PC32	Polymer preparations and compounds	160	6.1

Adapted from Ministère de l'Écologie du Développement durable et de l'Énergie (2014)

### 3.5.1 *Common Nanomaterial Applications and Potential for Release of Nanomaterials*

Some applications involve the intended release of NM, either to result on an intended human exposure (e.g., application of nano-enabled sunscreen onto the skin), or to application on other surfaces (e.g., generation of a nanocoating by spraying into a glass surface). In these cases, the estimation of the direct release of NMs is rather straightforward. However, understanding which fraction of it reaches its target application point, and which is the fate of such fraction after application is still largely unknown.

In other many applications, NM are part of the product matrix and are not intended to become released during use. Nevertheless, some of the normal use processes for some products may result on such unintended release. These can be mechanical processes, such as washing, wearing, tearing, breaking, and drilling, or physical-chemical degradation processes, such as weathering and chemical abrasion. The amount of the NM released from the matrix during the use stage will depend on several factors: the amount of NM in the product, the product lifetime, the way the NM are incorporated in the material (surface applications or in matrix), the surface contact area of the product that is affected by the process inducing release, the transfer factor of the NM within the matrix, the thickness of the product, and the frequency and duration of use.

During the last years, an increasing interest has resulted on research on the release of NM during the use phase of nano-enabled products. Indeed, it is assumed that unintended emissions from diffuse sources are one of the most important sources of NM releases to the environment [14]. Nevertheless, the number of studies evaluating release of NM from solid nanocomposites is still very low (Table 3.3) [176].

In general, weathering studies with polymeric nanocomposites have shown the degradation of the polymeric matrices due to photo- and chemical degradation. As a consequence, the nanoparticles tend to accumulate in the degraded zone, at the surface of the nanocomposite. However, free released NM are barely detected and rarely freed from the matrix in which they were included, even when weathering experiments have been combined with secondary mechanical forces [204, 205].

The release of nanomaterials (embedded in organic binder, as aggregates, or as single particles) from conventional paints during run-off events has been reported [195, 206–209]. However, the amounts released greatly differ among studies.

**Table 3.3** Summary of the current literature on release of nanomaterials from solid nanocomposites

	Weathering	Washing	Contact
Textiles/Fabrics	0	7	2
Thermoset	8	0	0
Termoplastic	6	0	2
Paints/Coatings	9	0	1
Cement	2	0	0
Dental glass	0	0	1
Ceramic	0	2	0
Total	25	9	6

Adapted from Froggett et al. [176]

A considerable number of studies have also focused on the release of nanomaterials (mostly silver) from textiles during washing processes [157, 160, 161, 210–212]. High releases have often been reported during the first washing event [211]. All these studies suggest that the silver particles in the textile dissolve to silver ions in the water and form secondary particles. A similar process seems to occur when textiles containing silver nanoparticles are immersed in artificial sweat [213–215].

The available research still provides a rather partial view of the potential release of nanomaterials or dissolved ions from consumer products. And further research is needed to understand and model which factors and how determine release under different processes.

## 3.6 End of Life

Products containing nanomaterials will eventually reach the end of their useful lives and, unless recycled, be discarded. In addition, waste materials containing nanomaterials are being generated during the manufacture of nanotechnology products. These waste streams generated during the life cycle of products containing nanomaterials are potential sources of nanomaterials into the environment. The handling, treatment and disposal of such wastes will determine the resulting environmental releases of nanomaterials. Therefore, the development of appropriate end-of-life management strategies for waste streams containing nanomaterials is critical.

This section provides an overview of the most common recycling and end-of-life processes for products and waste streams containing nanomaterials.

### 3.6.1 Recycling

Two categories of waste can be considered in terms of recycling processes. First, waste streams that are treated as broad waste categories, such as plastics or paper. These are typically highly heterogeneous mixtures of different products that could include multitude of different nanomaterials. And second, narrower categories, such as PET bottles,

tyres, and Li-ion batteries, that are comparatively much more homogeneous. Regardless of the category, current recycling processes will handle products with and without nanomaterials in unknown proportions. Research is needed to estimate the type and quantity of nanomaterials in different material flows entering recycling systems, and on how the presence of nanomaterials alters the quality of the recycled material. Indeed some research has been published on the performance of recycled composites containing nanomaterials, and results show that the presence of nanomaterials may negatively affect the quality of the recycled composites [216, 217]. Such information could result on changes in the optimal applications for the recycled materials or on changes in the recycling processes per se. In addition, information is also needed on the potential release of nanomaterials during these processes and on technical measures that could be used for minimizing them [218]. The generation of such information is necessary to evaluate potential negative impacts on workers or the environment [219].

### **3.6.2 Incineration**

Incineration is a thermal treatment, through which waste is combusted in an oxidizing ambient at temperatures in the range of 850–1200 °C [220]. There are different types of plants, which mostly differ in the off-gas treatment section. Materials (including nanomaterials) that enter an incineration plant can be totally or partially combusted or remain unaltered, depending on the local conditions in the combustion chamber, the melting point and reactivity of the materials, and additional matrix materials in which they are present. Unaltered or partly combusted materials can end up in the slag/bottom ash, retained in the particle control filters and becoming part of the fly ash, or go through such filters and be released to the environment.

Nanomaterials in the waste streams entering an incinerator may exist as free particles (i.e. a powder) or dispersed in a liquid or solid material. Based on theoretical thermodynamic considerations and on some experimental data, it is generally assumed that most nanomaterials in waste would end up in bottom ash. This would be the case for particle aggregates or particles that do not totally combust. A smaller fraction, mainly free particles and some partly combusted materials, would reach the air filtration systems, where a proportion of those would be retained [17, 220–225]. Some experimental data suggest that state-of-the-art flue gas cleaning systems (such as electrostatic precipitators and wet scrubbers) would effectively retain nanomaterials, but the efficiency of current filter techniques is still controversial [221, 222]. Further experiments are needed to fully substantiate these assumptions on the fate of nanomaterials in an incineration plant, and quantify efficiency of filter techniques for different type of nanomaterials. In addition, it remains unclear in what form the NM are present in the bottom ash. Treatment of the bottom ash depends on regional legislation, but it is usually disposed in landfills, unless originated from special waste streams that justify its further confinement. It is assumed that most of the waste streams containing nanomaterials will be considered domestic wastes, resulting in less strict regulations on the fate of resulting bottom ashes. Therefore, understanding in which form the nanomaterials



are usually present in the bottom ash (i.e. whether or not enclosed in vitrified fragments) is important to understand their possible later mobility [225]. This information could be used to evaluate if current treatments are appropriate for the resulting ashes.

### **3.6.3 Landfilling**

Landfill is a system of waste disposal that is based on burial of municipal solid waste (MSW) in specifically designed sites. Although landfill is one of the most exploited treatments for MSW end of life, it is not yet clear how NM behave during disposal. If NM are able to be transported through waste, then the potential for release from landfills to the surrounding environment increases. Existing studies show some degree of mobility for different NM, which depends on the NM and the composition of the leachate (organic composition, ionic strength, pH) [226, 227]. Another concern about the presence of NM in landfills is related to their capacity to influence biological activity. Very few data is available on this issue, and so far this indicates no effects on the overall biological activity [228, 229], although bacterial community structure has been shown to be sensitive to some nanomaterials [229].

### **3.6.4 Waste Water Treatment**

Domestic (and some industrial) waste water containing nanomaterials will end up in sewage treatment plants and industrial waste water treatment plants.

Concerns are related to the impact of nanomaterials on the biological systems within such treatments, and on their fate. Several studies have investigated such processes (see recent review by Neale et al. [230]), but available information is still rather partial. Part of the sewage sludge, when metal concentrations are below established maximum limits, is applied on land as supplemental fertilizer of landfill cover. Current regulations establish metal content limits without consideration of particle size. Yang et al. estimated the proportion of nano-TiO<sub>2</sub> present in a landfill and concluded that it represented around 0.1–0.2 % of the total Ti [231]. However, these values could vary regionally and with changing trends in the production of nano-TiO<sub>2</sub>. Further knowledge on the mechanisms of metal transport in soils and effects of environmental conditions and particle size are needed to evaluate the potential impact of applying sewage sludge containing nanomaterials on soils.

### **3.6.5 Current Practise and Regulations**

Altogether, there is very limited information on the possible risks associated to the presence of nanomaterials in wastes. In the lack of specific evidence for concern, no specific processes are required for wastes containing nanomaterials in neither

Europe nor the United States [232, 233]. In Europe, wastes are classified as hazardous or non-hazardous based on Regulation No. 1272/2008 on Classification, Labelling and Packaging of Substances and Mixtures [234]. This regulation does not include specific requirements for nanomaterials. Therefore, it is likely that nanomaterials will be classified in the same categories as their bulk form, and nano-specific hazards may be overlooked. The classification of waste as hazardous or non-hazardous is a key step as it leads to different requirements under the Waste Framework Directive. For example, mixing restrictions, labelling, and record keeping do not apply to wastes containing nanomaterials, unless they have been classified as hazardous [235].

Even when nanomaterials would be classified as hazardous, they may still be appropriate for use in some consumer products. In those cases, it is unlikely that their classification would result on specific end-of-life treatments for consumer products containing them. However, this is an issue that also applies to other type of hazardous substances.

More details on how current regulations affect wastes containing nanomaterials (and associated gaps) can be found in previous review reports [232, 233, 236].

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