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# Thermal disposal of waste containing nanomaterials: first investigations on a methodology for risk management

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**Abstract.** Considering the wide use and production of NMs since last two decades, these trendy nanomaterials (NMs) are expected to end up in thermal disposal and waste incineration plants (WIP). It seems relevant to assess the risks related to the thermal disposal and incineration of waste containing NMs (WCNMs). The objective of this work is to present a first approach to develop a preliminary methodology for risk management in order (1) to give insights on nanosafety of exposed operators and on potential environmental risks related to the incineration and thermal disposal of WCNMs, and (2) to eventually support decision-makers and incineration plant managers. Therefore, the main challenge is to find (a) key parameter(s) which would govern the decision related to risk management of NMs thermal disposal. On the one hand, we focused on the relevant literature studies about experimental works on incineration of NMs. On the other hand, we conducted an introductory discussion with a group of experts. The review of this literature highlights that the nano-object's nanostructure destruction appears as a relevant indicator of the risks related to the NMs incineration. As a consequence, we defined a "temperature of nanostructure destruction" (TND) which would be the temperature from which the nanostructure will be destroyed. This parameter has been assumed to be a consistent indicator to develop a preliminary methodology. If the combustion chamber temperature is higher than the TND of the NM (or if they are close to each other), then the nanostructure will be destroyed and no risks related to NMs remain. If the TND of the NMs is higher than the combustion chamber temperature, then the nanostructure will not be destroyed and risks related to NMs have to be considered. As a result, five groups of NMs have been identified. WCNMs including carbonic NMs appear to be in good position to be destroyed safely in WIP. On the other hand, based on this criterion, there would be no available thermal disposal plants to safely manage WCNMs including CeO<sub>2</sub> and ZrO<sub>2</sub>. Finally, a decision tree has been designed. TND is used as criteria to assess if a waste can be managed safely or not by a specific thermal disposal and which safety measures have to be taken.



## 1. Introduction

Since the last few years, the disposal via incineration of waste containing nanomaterials (WCNMs) has increasingly drawn attention of the authorities, the scientists and the waste incineration plant (WIP) managers.

Toxic effects of different types of NMs have been demonstrated (Kühnel & Nickel 2014; Wang et al. 2014). Nevertheless, the data are incomplete and more detailed studies are required. Their potential releases in the environment during their life cycle and their subsequent impacts on health have been an increasing concern for many years: firstly, the workers exposure to NMs during production (Oberdorster et al. 2010, Wohlleben et al. 2014, Bressot et al. 2015), then the emissions related to the wear and tear of products containing NMs (Shandilya et al. 2015), their end of life (storage or recycling) and finally their elimination via thermal disposal like incineration (which is the most widespread thermal disposal treatment technology in Europe). Indeed, the behavior and the fate of NMs in incineration treatment facilities have been investigated, lately, by some studies at lab-scale (Ounoughene et al. 2015; Massari et al. 2014; Derrough et al. 2013; Vejerano et al. 2014, Le Bihan et al. 2014, Vejerano et al., 2013a, 2013b;) and at real scale (Walser et al. 2012). Moreover, Roes et al. (2012) have evaluated the risks related to waste incineration of polymer nanocomposites without experimental works.

Regarding the risks related to the incineration of WCNMs, it seems necessary and relevant to develop strategies and tools to manage them. A risk is defined as function of exposure and hazard. However, in this present work, only the exposure is considered for assessing the risk because, taking account of the precautionary principle, it is assumed that the hazard is maximum as toxicity data of a lot of NMs is not yet available, so it is prudent here to assume the NMs may be toxic. In the future, when sufficient toxicological data will be available, it will be relevant to take into account the specific toxicity of each NM.

The objective of this work is to present a first approach to develop a preliminary methodology for risk management in order (1) to give insights on nanosafety of exposed workers and on potential environmental risks related to the incineration and thermal disposal of WCNMs and (2) to eventually support decision-makers and incineration plant managers. Therefore, the main challenge is to find a key parameter which would govern the decision related to risk management of NMs thermal disposal.

The aim of the present study is to provide a first insight into risks (exposure) regarding the incineration of WCNMs through the review of available data from literature concerning exclusively experimental works. This concise overview allows to identify and to define a key parameter which can determine the risks. Thus, a preliminary methodology for managing the risks related to the NMs thermal disposal can be proposed through the design of a decision tree.

## 2. Literature review related to experimental works on WCNMs incineration

The review of the bibliography related to incineration of WCNMs allowed to identify eight relevant studies exclusively related to experimental incineration tests performed on different NMs. The critical results of these studies are presented and their limitations are discussed. The table hereinafter (Table 1) describes succinctly those experimental studies.

Table 1: Literature review: Experimental studies on NMs incineration

References	Type of study	NMs	Results
Walser et al. 2012	Real scale: NMs injection in different WIP spot	CeO <sub>2</sub>	CeO <sub>2</sub> found in residues and it has been efficiently filtered by electro-filters
Vejerano et al. 2013a	Lab-scale: mix of waste tested in a non-modified tubular furnace – 850 °C – Study of NMs influence on	Paper, plastics and NMs: Ag, NiO, TiO <sub>2</sub> , CeO <sub>2</sub> , C <sub>60</sub> , Fe <sub>2</sub> O <sub>3</sub> , QD	Some NMs induce PAH concentration increase

	PAH production		
<b>Vejerano et al. 2013b</b>	Lab-scale: mix of waste tested in a non-modified tubular furnace – 850 °C – Study of NMs fate	Paper, plastics and NMs: Ag, NiO, TiO <sub>2</sub> , CeO <sub>2</sub> , C <sub>60</sub> , Fe <sub>2</sub> O <sub>3</sub> , QD	Persistence of some NMs in fumes and slags
<b>Derrough et al. 2013</b>	Lab-scale: NMs tested in a non-modified tubular furnace – 850°C and 1100 °C – Study of NMs fate	Sn, Ni, Ag	At 850 °C and 1100 °C, the NMs have reached their melting point
<b>Le Bihan et al. 2014</b>	Lab-scale: NMs tested in a modified tubular furnace – 850°C – Study of NMs fate	Black carbon, Silicon/Silica, Paint withTiO <sub>2</sub>	Persistence of NMs in fumes and slags
<b>Massari et al. 2014</b>	Lab-scale: paint tested in a non-modified electrical furnace – 950°C – Study of NMs fate	Paint withTiO <sub>2</sub>	The TiO <sub>2</sub> has been found in residues without any change
<b>Ounoughene et al. 2015</b>	Lab-scale: nanocomposite tested in a modified tubular furnace – 850°C – Study of NMs fate and behavior	Nanocomposite (polymer/nanoclay) Halloysite nanotubes (HNTs) incorporated in a polyamide matrix	HNTs have been found in fumes and slags ; they induce a two step mechanism in the thermal decomposition of the nanocomposite
<b>Vejerano et al. 2015</b>	Lab-scale: mix of waste tested in a non-modified tubular furnace – 850 °C –Toxicity study of NMs and particulate matter	Paper, plastics and NMs: Ag, NiO, TiO <sub>2</sub> , CeO <sub>2</sub> , C <sub>60</sub> , Fe <sub>2</sub> O <sub>3</sub> , QD	Some incinerated NMs have oxidative potential

Among those studies, the only real scale study has been performed by Walser et al. (2012). The authors have shown that nano-CeO<sub>2</sub> is found in residues and fumes and it has been efficiently filtered by electro-filters.

The other studies have been carried out at lab-scale. Ounoughene et al. (2015) and Le Bihan et al. (2014) have highlighted that, in order to perform lab combustion tests representative of the industrial incinerator furnace, it is important to control the combustion conditions in the lab-scale furnace. Indeed, in a real scale incineration process, combustion key parameters are controlled: temperature (850 °C in the combustion and post combustion-zone), residence time (at least two seconds in the post combustion zone at 850 °C), air-excess (never below 11 % of oxygen) and turbulence (a good mix between combustible and oxygen). Apart from Ounoughene et al. (2015) and Le Bihan et al. (2014), the lab scale studies have been performed using a furnace which doesn't strictly apply incineration combustion conditions but approaches them as much as possible. However, despite of this lack concerning representativity of combustion conditions, the experimental studies give interesting insights on the incineration of WCNMs, as described below.

Through three experimental studies, Vejerano et al. (2013a, 2013b, 2015) have investigated at lab-scale the effect of NMs on PAH concentration, their fate and their toxicity. They used a tubular furnace which has not been modified (and thus which doesn't strictly respect the real incineration conditions). Furthermore, they mixed their NMs samples with paper and plastics samples (Paper, plastics and NMs: Ag, NiO, TiO<sub>2</sub>, CeO<sub>2</sub>, C<sub>60</sub>, Fe<sub>2</sub>O<sub>3</sub>, QD). They conclude that some NMs induce PAH concentration increase; some incinerated NMs have oxidative potential, and the persistence of some NMs in fumes and slags.

Massari et al., 2014 have investigated the fate of nano-TiO<sub>2</sub> from paint at lab-scale, using a non-modified electrical furnace at 950 °C. The nano-TiO<sub>2</sub> has been found in residues without any change. Derrough et al., 2013 have performed lab-scale tests on Sn, Ni and Ag using a non-modified tubular furnace at 850 °C and 1100°C. At 850 °C and 1100 °C, the NMs have reached their melting point. The results of Ounoughene et al. (2015) and Le Bihan et al. (2014) show that the nanostructure of the NMs may or may not evolve or may get destroyed. In fact, the black carbon particles have been destroyed, the nanoclays (HNTs) have aggregated but have kept their nanostructure; the nano-silica can undergo sintering effects or remain the same like the nano-CeO<sub>2</sub> and the nano-TiO<sub>2</sub>. Furthermore, the polymer matrices emit aggregated/agglomerated nanoparticles during the incineration i.e.: an

organic matrix emits nanometric soot and a hybrid organic/inorganic matrix (silicone type) emits nanometric silicon oxycarbides. The presence of NMs doesn't seem to increase the soot production during the matrix thermal decomposition.

In order to clarify the definition of the nanostructure, the reader should consider that the nanostructure is related to the nanoscopic character (size) of a nanomaterial and more precisely to its specific surface area. According to the European Commission (Commission recommendation on the definition of nanomaterial, 2011/696/EU), a material should be considered as falling under the definition of a nanomaterial when its specific surface area by volume (SSV) is greater than  $60 \text{ m}^2/\text{cm}^3$ . When the nanomaterial's SSV is lower than  $60 \text{ m}^2/\text{cm}^3$ , then regarding that definition the material "loses" its nanostructure. Besides, "waste containing nanomaterials" has not been defined from a quantitative point of view.

These results lead to the conclusion that both flue gas and slag treatments will be affected by concerns about NMs risks and hence, potential precautions will need to be taken in their subsequent treatment steps. Furthermore, those studies didn't quantify the nanostructure of the NMs found in the fumes and in the residues (in terms of mass concentration in the residues or size distribution, for example); they use only SEM and TEM (scanning and transmission electronic microscopes) analysis in order to evaluate qualitatively the presence NMs nanostructure.

### **3. Design of a preliminary method**

On the basis of the literature review focused on experimental works related to NMs incineration, and of an introductory discussion with a group of French experts (experts on nanosafety and on waste management), a key parameter which governs the risks related to WCNMs incineration has been identified and a preliminary risk management methodology concerning incineration of WCNMs could be proposed.

#### *3.1. The risk could be determined by the fate of the nanostructure*

Previously, we have highlighted that the nanostructure can be destroyed, or transformed (i.e only deteriorated, without being destroyed) during the incineration process, or it can remain the same. In this way, the investigation of the fate of the nanostructure seems to be a good way to determine the risk. If the nanostructure is destroyed, then no ("nano"-)risks remain after the incineration. But, if the nanostructure is not destroyed (it remains the same or it is deteriorated), then a risk exists. In order to manage the risk, it is thus important to identify an indicator of the nanostructure destruction.

#### *3.2. The temperature as an indicator of the nanostructure destruction*

Actually, we could define a "Temperature of Nanostructure Destruction" (TND) which would be the temperature from which the nanostructure will be destroyed. The nanostructure destruction would be reached when the nanomaterial loses all its nanoscopic dimensions which could happen after a decomposition (via combustion of organic materials), or sintering or a fusion for instance. This could be measured *via* the specific surface area by volume or by mass. As the literature lacks of studies dealing with the concept of the nanostructure destruction, this topic is worth studying. According to the NMs nature (carbonic/organic compound, metal, metal oxide, oxide...), this TND can be related to the temperature of the thermal decomposition for organic or carbonic nanomaterials; or can be related to the melting point for the inorganic nanomaterials and nano-oxides.

#### *3.3. In a first approach, for operational reasons: the TND is considered equal to the melting point of the bulk material*

Many studies have investigated the relation between the melting point of NMs (nano-oxides for example) and the melting point of the corresponding bulk materials (non-nanostructured material) (Cao et al. 2004, Qi et al. 2004, Qi et al. 2005, Luo et al. 2011, Attarian et al. 2007). The melting point

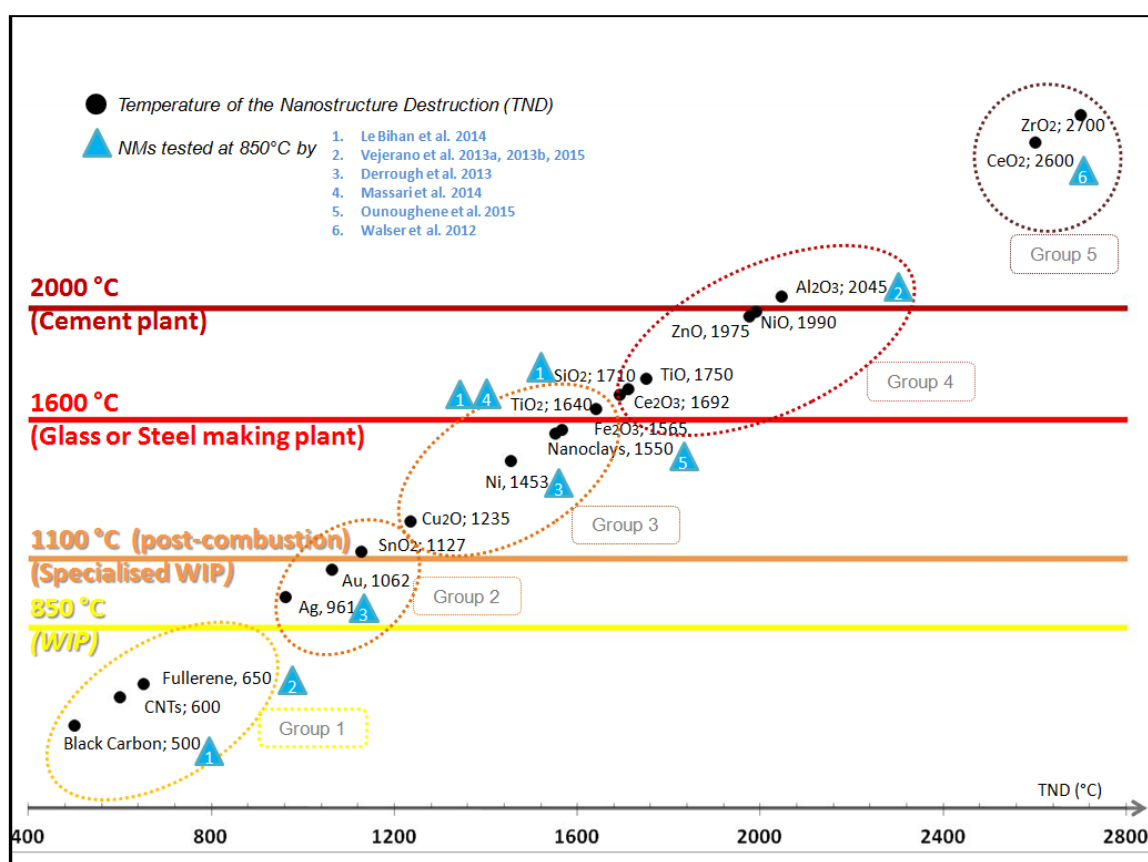
of a NM depends on its size. Because of its high-developed surface, the NM is more reactive than the corresponding bulk material, so the melting point of the NM is lower than the melting point of the corresponding bulk material. Yet the actual values of melting points are not available for most NM because the literature lacks of data.

In order to facilitate the use of the TND in an operational way, in the context of this paper and in the following part: the TND is considered equal to the melting point of the corresponding bulk material. The Figure 1 shows the TND of various NMs. The list of NMs is far to be exhaustive, and should be extended in the future. The NMs have been selected in order to present the concept of TND. In this study, it is considered that the NM is not embedded in a matrix; or if the NM is embedded in a matrix, then the matrix's decomposition temperature is lower than the TND (which is the case for a polymer matrix for example). This is an interesting issue to address later in order to improve the methodology. When NM's incineration has been studied experimentally in literature, a blue triangle positioned next to the NM mentions the study which deals with the fate of the NM (Figure 1). This figure shows that relevant literature data on the incineration of some NMs does exist, however it highlights that further investigations are needed to make it complete. The NMs are classified following the distance of their TND from the combustion (and post-combustion chambers) temperatures of different thermal disposal plants. Five groups have been identified as described hereinafter.

If the TND of the NM is close to the combustion chamber temperature of a WIP (850 °C), then the NM would lose its nanostructure during its stay in the furnace. This is the case of the NMs from the Group 1. Indeed, in the Group 1 which consists of organic NMs like CNTs, fullerene, and black carbon, the NMs would be consumed in the furnace as observed in few studies (Vejerano et al., 2013a; Le Bihan et al., 2014). The study of Schlagenhauf et al. 2014 has highlighted that CNTs are destroyed when the temperature is higher than 600 °C and when the atmosphere is oxidative. Then the nanostructure of a NM from Group 1 is destroyed in a WIP.

With regard to the Group 2, which consists of nanosilver, nano-tin oxide and nano-gold: on the one hand, their nanostructure could change (deterioration or destruction) in a WIP since their melting point is slightly higher than the WIP combustion chamber temperature; on the other hand, their nanostructure could be destroyed in a specialized WIP because their melting point would be close to the temperature of the post-combustion chamber from a specialized WIP (1100 °C). The nanosilver has been tested by Derrough et al., 2013 in a tubular furnace at 850 °C and 1100 °C. It has melted under the two temperatures and thus has lost its nanostructure. This demonstrates that the NMs can have melting temperatures lower than the actual melting point of the bulk material. It also suggests that WCNMs could be destroyed in either standard municipal solid waste incinerators (at 850°C) or specialized industrial waste incinerators at 1100°C. However, further investigations in a furnace which applies the same conditions than an industrial incineration furnace would be necessary (as explained previously).

The melting points of the NMs from the Group 3 (Fe<sub>2</sub>O<sub>3</sub>, Nanoclays, Ni, Cu<sub>2</sub>O, TiO<sub>2</sub>) are much higher than the temperature of the WIP combustion chambers, then, it is expected that the nanostructure of these materials will not change in a WIP (nor in a specialized WIP). This is the case of the nanoclays studied by Ounoughene et al. (2015), and the Ni studied by Derrough et al. (2013), NiO and Fe<sub>2</sub>O<sub>3</sub> studied by Vejerano et al. (2013a, 2013b, 2015). In order to destroy the nanostructure of these NMs from the Group 3, one possibility would be to send them to steelmaking or glassmaking plants which work with furnace reaching 1600 °C. However, it is assumed that the NMs could enter the mentioned industrial thermal processes without affecting their effectiveness. This point needs obviously further investigations.



**Figure 1: NMs classification according to their TND**

The Group 4 consists of many nano-oxides like Al<sub>2</sub>O<sub>3</sub>, NiO, ZnO, SiO<sub>2</sub>, Ce<sub>2</sub>O<sub>3</sub> and TiO. The only way to destroy their nanostructure would be by using a furnace from the cement industry operating at 2000 °C.

As for the materials from Group 5 (ZrO<sub>2</sub> and CeO<sub>2</sub>), their high melting point (>2000 °C) makes their nanostructure non-destructible in a WIP combustion chamber of 850 °C as highlighted by Walser et al. 2012 and also non-destructible in any existing furnace from a thermal plant. NMs of this group will be found in the incineration residues and provisions will need to be taken to manage the risk associated with their potential dissemination.

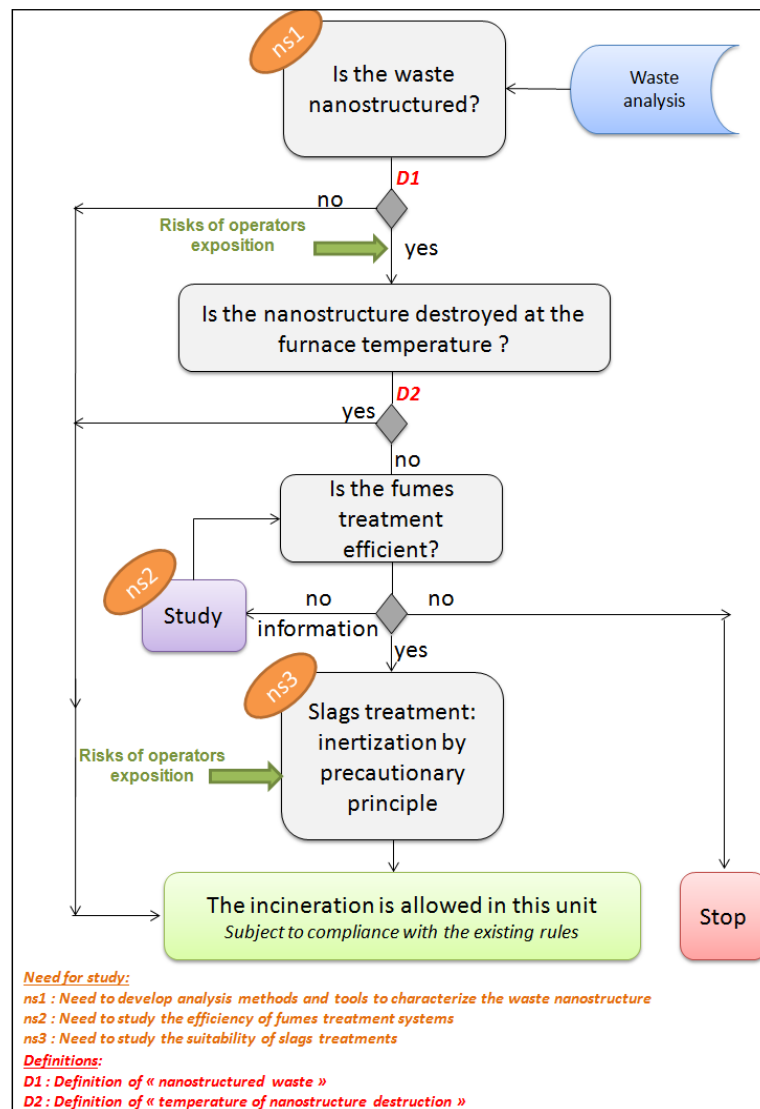
Besides, we have considered that no NMs vaporization occurs in the furnace. In fact, the vaporization temperature of NMs shouldn't be reached in order to avoid any re-crystallization or nucleation process which would lead to the formation of new nanoparticles which could represent a supplementary risk. Here again, further studies are necessary to determine the risks.

At this point, it clearly appears that more investigations are necessary to fully study the thermal disposal of WCNMs and the nanostructure destruction.

### 3.4. Decision tree

It is now possible to gather all this knowledge and use it at an operational level. A decision tree can be designed. It is a first attempt and its final objective is to eventually support waste managers for the selection of suitable thermal disposal treatment for WCNMs (Figure 3).





**Figure 2: Decision tree: first approach for developing a risk management methodology**

The decision tree takes into account the possible release of the NMs during a thermal treatment. It is considered that when WCNMs enter a thermal disposal plant: the operators (or workers) could be exposed to the NMs and the NMs could be released in the environment. Then, in order to prevent any NMs release, a series of questions should be answered.

The first step consists in analyzing the waste by investigating whether the waste is nanostructured related to the first question: “is the waste nanostructured?”. This issue is associated to the first need for study (ns1): “need to develop analysis methods and tools to characterize the waste nanostructure”. Indeed, there is an identified lack in the nanostructure characterization of NMs and new methods should be developed to evaluate the nanostructure of a NM (for example, through the measurement of specific surface area by volume). If the waste is not nanostructured, then there is no risk related to the size of NMs and the incineration is allowed in this unit (subject to compliance with the existing rules regarding other acceptance criteria). If the waste is nanostructured, the next question is: “is the nanostructure destroyed at the combustion chamber temperature?”. This question refers to the temperature of the nanostructure destruction (TND). So, if the TND of the NM is reached in the

furnace, the nanostructure is destroyed and the waste can be safely incinerated in this unit. But if the TND is not reached, then another question should be raised: “is the fumes treatment efficient?”. If the answer is “no”, then the incineration shouldn’t be done in this unit and another solution could be envisioned: for example the NM could be sent to another kind of thermal disposal plant working at a temperature higher than TND of the NMs which could destroy the nanostructure of the NM. As mentioned above, those kinds of thermal disposal units could be: specialized WIP (post-combustion chamber at 1100°C), glass industry and steelmaking plant (around 1600°C), or cement plant (around 2000°C). However, as mentioned previously, further studies need to investigate how the NMs waste stream would affect those industrial thermal processes in order to assess the feasibility. Finally, NMs with TND higher than 2000°C (group 5), should be stored as no available thermal disposal plant works at temperature high enough to destroy their nanostructure.

If the unit has no information about the efficiency of the fumes treatment regarding NMs, then a study has to be launched that refers to “ns2: need to study the efficiency of fumes treatment systems”. If the answer is “yes”, then by precautionary principle, the slags have to undergo inertisation treatments. This action leads to the “ns3: need to study slags treatments suitability”. Moreover, because the waste is nanostructured, operators can be exposed to the NMs and thus they have to be protected and equipped with adequate personal protective equipments.

#### 4. Conclusion

In order to provide a first insight into nanosafety regarding the incineration of WCNMs, the present study proposes an introductory approach for developing a preliminary methodology for managing the risks through the design of a decision tree.

This work presents a review of existing data about experimental works related to incineration of NMs and identifies a key parameter which could determine how to manage the risks related to the incineration of WCNMs. Only a few experimental data is currently available. It has been highlighted that the risk can be determined by the fate of the nanostructure. A temperature of nanostructure destruction (TND) has been defined. By default and for operational reasons, this TND can be equal to the melting point of the corresponding bulk material.

If the WIP combustion chamber temperature is higher than the TND of the NM or if they are close to each other, then the nanostructure will be destroyed and no risks related to the nanostructure of NMs remain (other risks still need to be considered). If the TND of the NMs is higher than the WIP combustion chamber temperature then the nanostructure will not be destroyed and another way to destroy the nanostructure would be to send the NM in a other kind of thermal disposal plant working at a temperature high enough to destroy the nanostructure of the NM, namely equal or higher than the TND (specialized WIP, glass industry, steelmaking plant, or cement plant), assuming that the effectiveness of the thermal industrial processes would not be affected by the NMs waste stream.

Finally, this preliminary study highlights the necessity to develop analytic tools in order to quantify the nanostructure in the waste, in the residues and in the fumes and to further investigate the thermal disposal of WCNMs.

It is important to underline that this method is preliminary: some gaps have to be considered in its future developments, for example, the effect of the matrix (where the NM is embedded) on the TND, and the effect of NM waste stream on the efficacy of the other kinds of thermal processes. In addition, some specific experiments are needed to support the concept of TND and a larger variety of NM should be considered.

However, despite its limits, the present approach could be a useful starting point that highlights issues which are worth studying in order to build a proper methodology for managing the risks related to the incineration of WCNMs.

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