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Nanofiltration and nanostructured membranes—Should they be considered nanotechnology or not?

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ABSTRACT

Nanofiltration is frequently associated with nanotechnology – obviously because of its name. However, the term "nano" in nanofiltration refers – according to the definition of the International Union of Pure and Applied Chemistry (IUPAC) – to the size of the particles rejected and not to a nanostructure as defined by the International Organisation of Standardisation (ISO) in the membrane. Evidently, the approach to standardisation of materials differs significantly between membrane technology and nanotechnology which leads to considerable confusion and inconsistent use of the terminology. There are membranes that can be unambiguously attributed to both membrane technology and nanotechnology such as those that are functionalized with nanoparticles, while the classification of hitherto considered to be conventional membranes as nanostructured material is questionable. A driving force behind the efforts to define nanomaterials is not least the urgent need for the regulation of the use of nanomaterials. Since risk estimation is the basis for nanotechnology legislation, the risk associated with nanomaterials should also be reflected in the underlying standards and definitions. This paper discusses the impacts of the recent attempts to define nanomaterials on membrane terminology in the light of risk estimations and the need for regulation.

1. Introduction

Nanofiltration (NF) is a technology that – based on its name – seems to be part of the nanotechnology boom. However, the use of the term "nano" in nanofiltration is misleading. Membranes are defined on the basis of the size of the particles that they reject [1]. Hence, the term "nano" does not refer to structural elements of the membrane such as pores. Most NF membranes do not show physically distinguishable pores, instead the separation depends on solution/diffusion processes in the membrane. In contrast to the well-established definitions and standards for membranes, and despite the flourishing research on nanotechnology, there is still no commonly agreed definition of nanotechnology, nanomaterials or nanoparticles. Hence the development of regulations and legislation on the use of nanomaterials is still ongoing. It is evident that it is not possible to enact laws without clear definition of the subject. For this reason several countries have produced their own set of definitions [2]. Only recently, the International Organization for Standardization (ISO) has released a proposal for definitions and standards of nanomaterials in the ISO/ICE 80004 series [3–5].

In a first attempt to bring together the established membrane terminology and the nanotechnology perspective, the Network of Excellence NanoMemPro (an EU FP6 project) introduced the umbrella term "nanoscale based membrane technologies" [6]. This term covers all membranes associated with nanotechnology. On the

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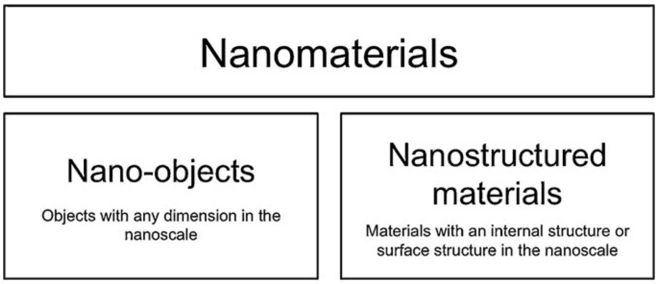

Fig. 1. Categorization of nanomaterials according to ISO (ISO/TS 80004-1 [3]).

other hand, several different terms are currently used for membranes that are functionalized with nanoparticles, while there is no new terminology for conventional membranes with an internal nanostructure. However, a clear and sound terminology is needed for any material that falls under the ISO definition of a nanomaterial to allow legislative authorities for risk estimation and the consequent introduction of coherent and enforceable regulations on the production and use of nanomaterials. The attempt to define nanomaterials will concern also certain materials that were considered to be conventional until now such as NF-membranes and, if regulated accordingly, this totally new perspective on membranes may have an unforeseen and extensive impact on existing industries, because the use of membranes is widespread in drinking water treatment and the food industry.

This article analyses the impact of the new developments in defining nanotechnology on established membrane technologies in view of impending regulation and for risk estimation. It addresses the question: what do the new definitions mean for established membranes technologies, especially nanofiltration?

2. Definitions in nanotechnology

At the end of last century, the term nanotechnology was introduced to describe the production and manipulation of nanoscale materials. But only recently have definitions in nanotechnology been started to ensure a common language in science and industry and to allow regulation and legislation. While many countries have established their own definitions [2], the International Organization for Standardization is elaborating a universal set of standards on nanotechnology [3,4]. The first published ISO standard in 2008 focused on nano-objects only [7]. In 2010 the ISO/TS 80004 series on standards in nanotechnology started with ISO/TS 80004-1 which encompasses core terms such as “nanotechnology”, “nanoscale” and “nanomaterial” [3].

The whole set of standards is based on the definition of *nanoscale* as “size range from approximately 1–100 nm”. The lack of a practical alternative has overridden the concerns that merely a physical dimension would not justify a new technology term. Consequently, *nanomaterial* is defined as “material with any external dimension in the nanoscale or having internal structure or surface structure in the nanoscale”. This very broad term would include also natural or unintentionally produced nanomaterials and thus requires further clarification, while *engineered nanomaterials* are intentionally “designed for a specific purpose or function”. As shown in Fig. 1, nanomaterials are divided into *nano-objects* (“object with any dimension in the nanoscale”) and *nanostructured materials* (“material having internal nanostructure or surface nanostructure” where *nanostructure* is the “composition of inter-related constituent parts, in which one or more of those parts is a nanoscale region”). Table 1 gives an overview of the different terms as defined in ISO/TS 80004-1 [3]. The ISO standard on nanostructured material is still under preparation (ISO/TS 80004-4).

Table 1
Overview over standards and terms in ISO/TS 80004-1 [3] relevant in membrane technology.

Nanoscale	Size range from approximately 1–100 nm
Nanotechnology	Application of scientific knowledge to manipulate and control matter in the nanoscale in order to make use of size- and structure-dependent properties and phenomena, as distinct from those associated with individual atoms or molecules or with bulk materials
Nanomaterial	Material with any external dimension in the nanoscale or having internal structure or surface structure in the nanoscale
Engineered nanomaterial	Nanomaterial designed for a specific purpose or function
Nanostructure	Nanostructure: composition of inter-related constituent parts, in which one or more of those parts is a nanoscale region [Note: a region is defined by a boundary representing a discontinuity in properties.]
Nanostructured material	Material having internal nanostructure or surface nanostructure
Nano-objects	Objects with any dimension in the nanoscale

3. Nanoporous membranes

Pressure driven membrane based separating processes have been state of the technology for 50–80 years [8]. During this time, technology developments made smaller and smaller pores possible, leading to a classification into four types of pressure driven filtration processes: microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO). According to the definition of the International Union of Pure and Applied Chemistry [1] the four filtration methods are characterized as follows:

Reverse osmosis (RO): Liquid-phase pressure-driven separation process in which applied transmembrane pressure causes selective movement of solvent against its osmotic pressure difference.
Nanofiltration (NF): Pressure-driven membrane-based separation process in which particles and dissolved macromolecules smaller than 2 nm are rejected.
Ultrafiltration (UF): A separation process whereby a solution containing a solute of molecular size significantly greater than that of the solvent molecule is removed from the solvent by the application of a hydraulic pressure which forces only the solvent to flow through a suitable membrane, usually having a pore size in the range of 1–100 nm.
Microfiltration (MF): Pressure-driven membrane-based separation process in which particles and dissolved macromolecules larger than 100 nm are rejected.

However, membranes categorized as NF membranes are not uniform. Polymeric NF membranes are usually dense with no detectable pores and resemble RO membranes, while ceramic NF membranes are microporous and similar to UF membranes. Fig. 2 shows a scanning electron microscopy image of a UF membrane (cut-off around 2000–3000 Da) in comparison with a tight (dense) NF membrane (cut-off around 200 Da).

Membranes can be made from inorganic materials (e.g. ceramics or metals) or – most commonly – from polymers. Ceramic membranes have the advantage of being more resistant to mechanical forces, chemicals and temperature, but they are significantly more expensive to manufacture. The production method also determines the form of the pores. Ideally a uniform pore size distribution can be achieved. However, small pores are difficult to characterize. Therefore the effective pore diameter in RO, NF and UF is defined by the molar mass of the smallest globular molecules or particles that are

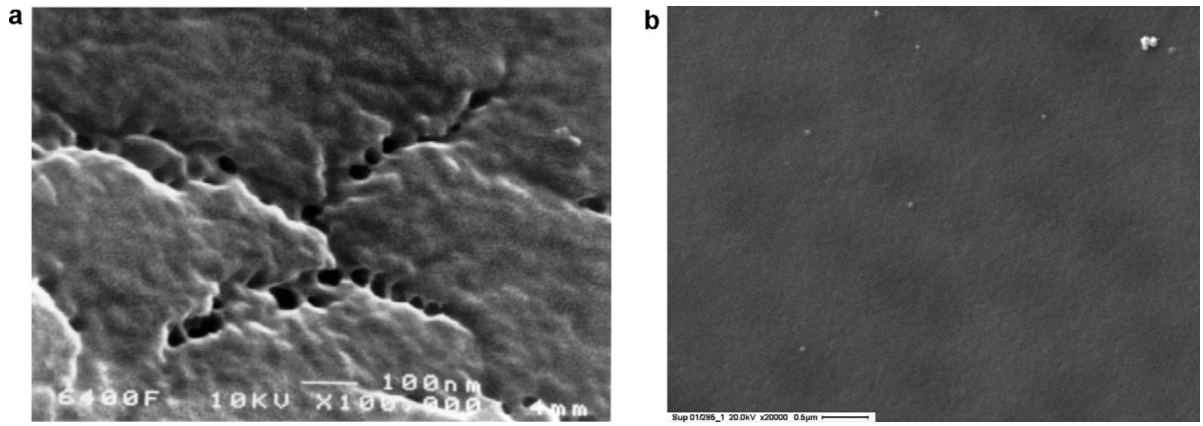

Fig. 2. Scanning electron micrographs of a UF (left) [9] and a tight NF (right) membrane illustrating the difference between porous and non-porous membranes. *Source* (left): Reprinted with permission, copyright 2003, John Wiley and Sons.

unable to pass the membrane (rejected by 90%). The value is given as nominal molecular weight cut-off (NMWCO [Dalton]).

The different filtration methods overlap in their application range (Fig. 3). A general classification scheme is based on the ability of the membrane to reject dissolved species (Table 2): RO and most NF membranes may be considered dense membranes (transport diffusion based only) that are able to reject dissolved species such as ions, while UF and MF membranes can usually only reject suspended particles. It is evident that the choice of the membrane type requires a trade-off between the pore size or membrane density and the contaminant rejection. Generally, for smaller pores (denser material) rejection will be increased but so will the pressure needed and thus the energy demand. Hence, the pressure requirements for NF are lower than for RO, resulting in a significant energy saving. Aside from size exclusion, electrostatic interactions are also a very important parameter with regard to species rejection in RO, NF and tight UF. This is described by the Gibbs–Donnan effect [10] due to which, for example, the rejection of divalent anions with negatively charged membrane materials is higher than the rejection of monovalent anions. If different ions are present in the feed water, the Gibbs–Donnan effect can result in a separation of ionic species which would be difficult to achieve purely by size effects [11].

The use of membranes for the separation of specific substances or a mix of substances from a fluid is often more energy efficient than thermal methods [8]. Since it requires no heating, membrane filtration is the method of choice, especially with thermally instable solutions or suspensions. Main application areas are thus in the food industry, biotechnology, medicine and pharmaceutics but also in the metal industry. Additional increasingly important sectors are the drinking water industry and wastewater treatment.

The four types of filtration have different application areas based on the size of the particles rejected. In practice, the NMWC should be at least 20% smaller than the molecular weight of the substance to be rejected. Microfiltration is used for example in the production of fruit juice, wine, milk and beer for the rejection of bacteria, to separate oil–water emulsions, in water treatment and in biotechnology for the harvesting of cells. Also ultrafiltration is used for a wide range of applications such as cold sterilisation, metal recovery, water disinfection or the separation of proteins (e.g. from milk). Nanofiltration is mainly used for the removal of hardness and natural organic matter in drinking water production and reverse

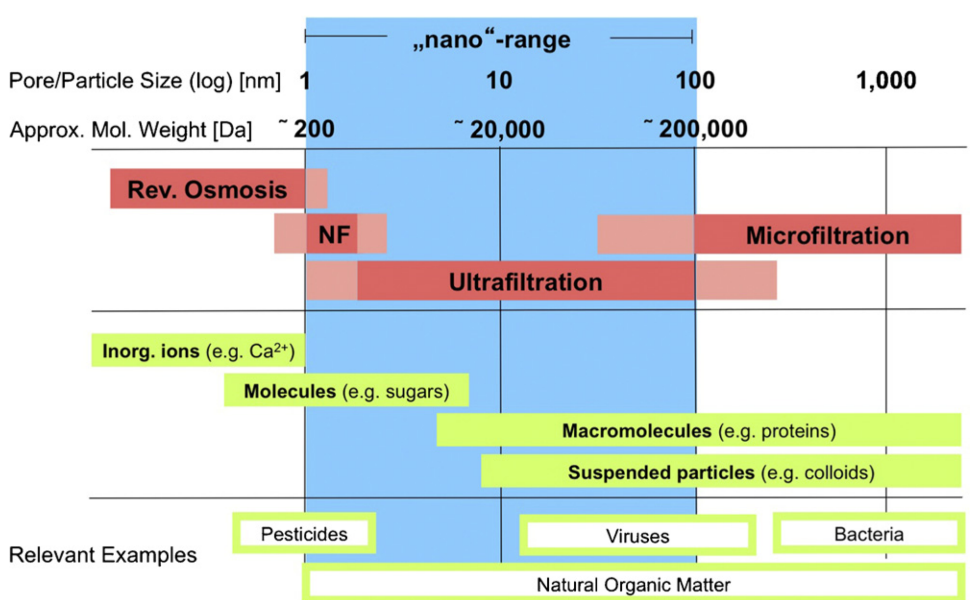

Fig. 3. Comparison of the filtration range of the four filtration methods: reverse osmosis (RO), nanofiltration (NF), ultrafiltration (UF) and microfiltration (MF) with respect to the "nanoscale" as defined by the ISO. Some relevant examples illustrate possible application areas.

Table 2
Comparison of RO, NF, UF and MF (NMWC, nominal molecular weight cut-off).

	RO	NF	UF	MF
Substancespermeatingthemembrane	Solvent only	Specific dissolved species	Dissolved species	Suspended particles smaller than 100 nm
Membrane structure	Non-porous	Non-porous/porous	Porous	Porous
Substances rejected	Dissolved species	Suspended particles, some dissolved species	Suspended particles around 1–100 nm	Larger particles
Pressure [bar] [12]	30–70	10–40	0.5–10	0.5–2
NMWC [Da] [12]	<500	500–2000	2000–500,000	>500,000

osmosis for the production of ultra-pure water and the production of drinking water from sea- and brackish water [9].

4. Membranes functionalized with nanoparticles

The idea of functionalizing membranes with nanoparticles is rather new. Nevertheless quite a few (mainly lab-scale) experiments have been performed so far with different membrane materials and nanoparticles [13]. As an example nano-TiO_2 particles can be integrated into the polyamide top layer of the membrane to decrease membrane fouling and to increase the flux due to the superhydrophilic properties of nano-TiO_2. However, some of the research results are contradictory [13] and the actual application of such membranes is still in its infancy. It is thus not surprising that the terminology for these membranes also is not consistent. Several different terms are currently being used such as nano-activated, nano-enhanced, nanoparticles-enhanced, nanoparticles-based and nano-functionalized membranes. To avoid any ambiguity, the terminology should be standardized. In this paper we will use the term “nano-enhanced membrane” (NEM) since “enhancement” best describes the function of the nanomaterials in the membrane and because “nano” instead of “nanoparticle” also includes carbon nanotubes and other nano-objects.

NEMs aim at combining nanotechnology with membrane technology to improve membrane properties and thus increase their performance. There are different directions of research: some scientists are trying to integrate carbon nanotubes as pores in membranes [14,15]. These membranes could be used instead of RO membranes for the production of ultrapure water with the advantage of an increased flux [16]. Other approaches involve the design of membranes from a CNT-mesh [14,17] or the surface functionalization of membranes with different nanoparticle types such as TiO_2, Ag, aluminum oxides, silica, CNT, zirconia or iron oxides nanoparticles [13] to achieve increased flux and/or to reduce fouling, which is still the number one challenge in membrane technology [13,18]. However, a major challenge in applying TiO_2 to polymeric membrane materials is that nano-TiO_2 in the surface layer not only degrades organic substances in the water phase but also the organic membrane material. To avoid this problem Keuter et al. [19] applied nano-TiO_2 and nano-TiO_2/Ag nanocomposites to metallic microsieves. These microsieves have – due to their production process – an almost uniform pore and pore size distribution. Coating by sol–gel or chemical vapour deposition (CVD) processes offers the opportunity to achieve antifouling properties by nano-TiO_2 induced photocatalytic degradation processes. Further efforts are underway to apply these functionalized membranes for direct degradation of organic compounds (e.g. pharmaceutical residues) in combination with UV-LEDs. TiO_2, ZrO_2 and other crystalline nanoparticles may also be used as active layers of ceramic membranes. Such membranes would also – independently of their pore size – be categorized as nano-enhanced membranes.

5. How do membranes fit into the definitions of nanomaterials?

Based on the ISO definition, membranes with pores in the range of 1–100 nm would evidently fall under the term nanostructured materials. In Fig. 1, it is shown that NF and UF reject particles with about 0.2–200 kDa corresponding to a nanoscale diameter of around 1–100 nm. Following the ISO definition, NF and UF membranes should consequently be classified as nanomaterials. However, most NF membranes are considered to be dense with no detectable pores in the separating layer as described in the previous chapter. It is therefore questionable whether tight NF-membranes can be regarded as nanomaterials. If we treat dense NF-membranes as nanostructured materials, any dense material that allows the diffusion of nanosized compounds (e.g. RO membranes, paper, plastics, human skin) would become a nanomaterial. This stance would obviously not make sense and would be in direct contrast to the aim of the ISO-standards to clarify and support legislative processes. It thus becomes evident that the use of the term “nano” in nanofiltration is rather misleading with regard to the terminology for nanotechnology. The introduction of a new term for NF would be an answer but difficult in view of the widespread acceptance of the term nanofiltration since it was introduced in the 1980s. In addition, the term “nano” may also have a promotional effect. Scientists and representatives from industry confirm that in some cases it is favourable to label products and processes with the term “nano” since it may lead to additional funding (e.g. in EU-projects) or attract new customers (“nano” often suggests innovative, new solutions).

In contrast to tight NF membranes, UF membranes and loose NF membranes in fact have nanosized pores and thus clearly can be considered nanomaterials according to the current definition by ISO. It is, however, debatable whether it makes sense to classify UF membranes as nanomaterials from a regulatory perspective based on the risk assessment of nanoporous membranes.

There is a general agreement [2] that there is a need for additional regulation of nanomaterials and that existing regulations such as REACH (European Community Regulation on chemicals and their safe use (EC 1907/2006)) may not sufficiently cover the potential additional risk aspects of nanoscale materials. This debate introduces the risk perspective which is in most cases implicit in any discussion on the definition and regulation of nanomaterials. The regulatory issues surrounding nanotechnology and novel nanomaterials and the increasing production and use of “nano-products” inevitably involve the question about the risk associated to the use of nanomaterials. However, the concerns regarding risks do not focus primarily on nanotechnology itself but rather on engineered nano-objects (ENO) only. It has been shown that ENO are able to cross cell barriers [20] and might in some cases cause adverse effects on humans and the environment [21–24]. Preliminary studies have shown that ENO can be released from different products such as textiles containing nano-Ag [25] or facades treated with paints containing nano-TiO_2 or nano-Ag [26,27] which raises the question of the potential human and environmental exposure

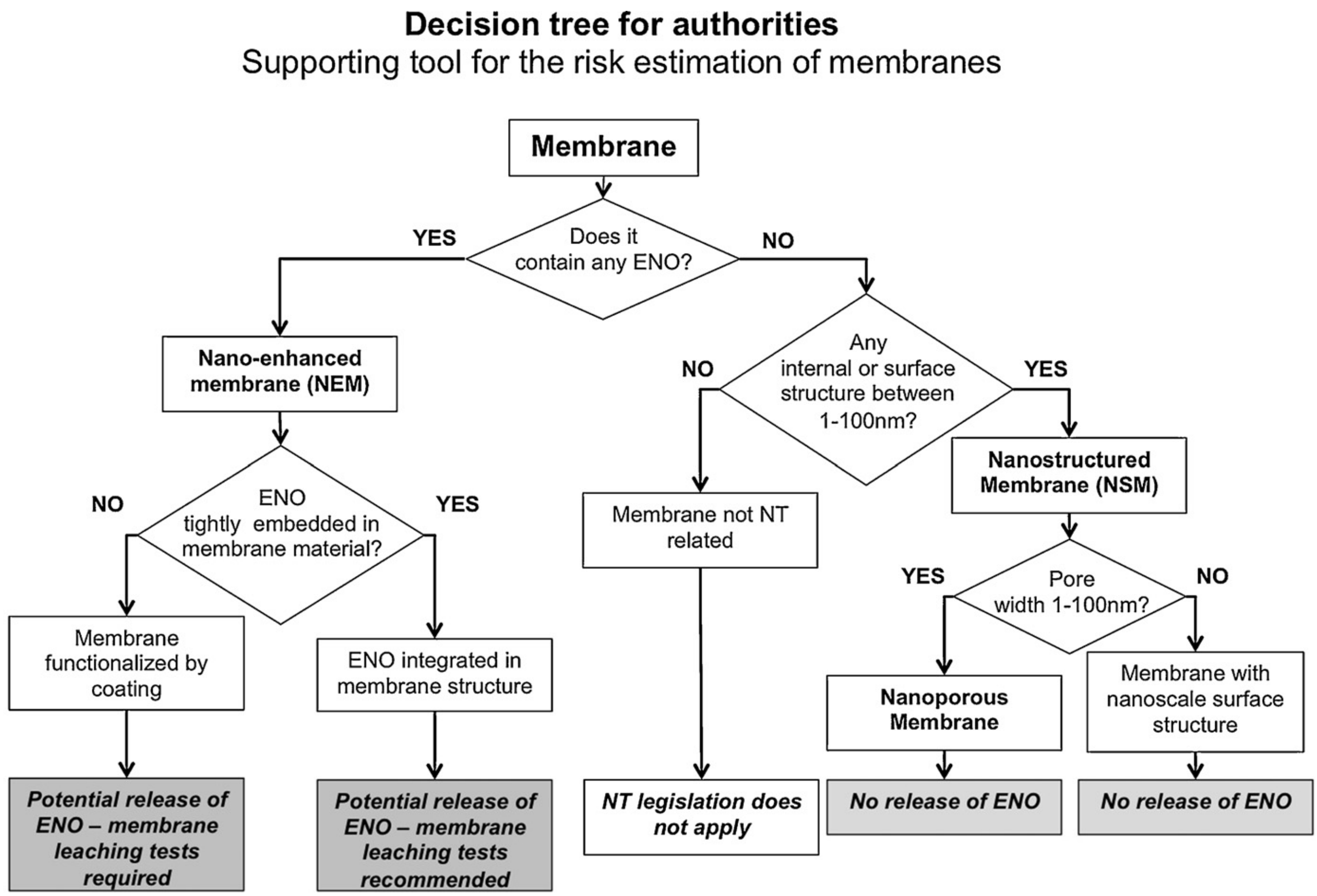

Fig. 4. Decision tree for the risk estimation of nanoparticle release by membranes. ENO, engineered nano-objects; NT, nanotechnology.

[28]. Recent studies have thus modelled the expected concentration of ENO in the environment to identify the potential exposure [29,30]. However, bulk materials with internal nanostructures should not pose any novel risks because of their nanostructure. Risk assessment always includes two aspects: hazard and exposure. Hazard is indicated by the toxicity of a given nanomaterial, while exposure is determined by the likelihood to come in contact with this nanomaterial. For non-functionalized nanoporous membranes there is neither a hazard due to the nanosized pores nor an exposure to nano-objects to be expected.

Risk research on nanotechnology has become a hot topic not only for scientific reasons but also to support policy makers in their efforts to regulate nanotechnology [31]. However, the proposed standards and definitions of nanomaterials by the ISO [4] do not take these risk based, regulatory requirements into account. Nanostructured materials according to ISO/TR 12802 [4] would include nanodispersions and nanostructured powders that are composed of nano-objects as well as nanoporous materials where no nano-objects are involved. But nanoporous membranes differ significantly from most other nanomaterials, for which a potential release of nanoparticles or other nano-objects cannot be excluded. Concretely, regular UF membranes have nothing in common with nanotechnology, not even during the production phase.

6. Implications for the risk assessment of membranes from a "nano-perspective"

For a sound legislation, a clear differentiation between (a) nano-objects and materials with integrated nano-objects and (b) other nanostructured materials needs to be made based on the significantly different risk associated with these materials. It might even be considered to specifically exclude nanoporous materials from the definition of nanomaterials since their properties and risk aspects are consonant with bulk material rather than nanomaterials. Lövestam et al. [2] state that "*other materials such as nanoporous materials, are in some definitions considered as nanomaterials, as they incorporate nanostructures in order to modify their properties. Nevertheless (...) such bulk nanomaterials should generally not be considered in a regulatory context as it is very unlikely that the nanostructured components would ever be released as 'free' particulate nanomaterials as a result of normal use.*" Nanoparticles are formed from almost any material (e.g. polymers or textiles) during abrasion, grinding or cutting operation even if the material itself is free from nanoparticles [32–35].

A totally different case is represented by NEMs as described in a previous section. NEMs correspond to the definition of nanomaterials since they actually contain (embedded) nano-objects which may be released during production, use and/or disposal. Hence, the risk assessment of NEMs cannot be considered in similar way to that for conventional, nanoporous membranes.

The question of a potential release of nano-objects is the single most relevant question for regulatory purposes and should possibly be reflected in the standards and definitions. Fig. 4 shows a decision tree for the risk estimation of membranes regarding the potential release of nanoparticles. The diagram includes the relevant aspects of (a) whether there are any nano-objects integrated in the material and (b) if yes, how tightly they are bound and thus how easily they might be released [28].

In addition to the regulatory point of view, the membrane industry itself does not consider nanoporous membranes as nanotechnology. UF membranes have been established for more than half a century and it is not obvious, why this – up to now conventional – method should suddenly be attributed to nanotechnology. For several decades, membranes have been applied for water and wastewater treatment in order to provide high water quality and to remove a broad range of contaminants from source waters. Up to the present, membranes in the food and water industry are subject to stringent regulations to ensure compliance with water quality and food safety legislation. It might be necessary to reassess the risks during production and handling of membranes, especially for membranes featuring nanostructures. However, new (nanotechnological) regulations concerning the application of nanoporous membranes should be based on a potential risk and not merely on a new definition and the consequent reclassification of UF and NF to nanotechnology.

7. Conclusions

The use of the term "nanofiltration" as well as the recent definitions of nanomaterials cause confusion rather than provide clarification with regard to hitherto considered conventional membranes. While the term nanofiltration implies that it is a nanotechnological filtration method without necessarily being one, UF is not normally associated with nanotechnology even though UF membranes contain intentionally engineered structures at the nano-scale and thus could in this respect be considered nanomaterials according to the ISO definition. However, UF membranes should also not be regarded as nanotechnology – at least not from a regulatory perspective. Risk aspects are the driving force behind new legislation for nanotechnology and they must thus also be the basis for any definition used for regulatory purposes. The relevant question in this respect is whether there is a potential release of nano-objects which are the subject of investigation in nanosafety research. Since non-functionalized, nanoporous materials do not contain any nano-objects, any risks associated with these can be excluded. Materials where the nanoscale structure does not result from discrete particles and where there is no risk for the release of nano-objects should thus not be considered as nanomaterials from a regulatory point of view. Definitions of nanomaterials must reflect this basic difference to be pertinent for any new nanotechnology legislation.

The same distinction that is made in the definitions and standards accordingly applies to new legislation. It is clearly not logical to generally regulate all nanomaterials as defined in ISO/TR 12802 [4] by the ISO. Regulation of nanomaterials should focus on nano-objects as concluded in the Report "Considerations on a Definition of Nanomaterial for Regulatory Purposes" by the European Commission Joint Research Center [2]. However, it is imperative that this must also include any material that may release nanoparticulate materials.

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