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Perspectives

Energy consumption during nanoparticle production: How economic is dry synthesis?

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Abstract

The production of oxide nanoparticles by selected wet-chemistry or dry processes is compared in terms of energy requirements. Clear differences arise for production using electricity-intensive plasma processes, organic- or chloride-derived flame synthesis and liquid based precipitation processes. In spite of short process chains and elegant reactor design, many dry methods inherently require vastly bigger energy consumption than the multi-step wet processes. Product composition strongly influences the selection of the preferred method of manufacturing in terms of energy requirement: Metal oxide nanoparticles of light elements with high valency, e.g. titania demand high volumes of organic precursors and traditional processes excel in terms of efficiency. Products with heavier elements, more complex composition and preferably lower valency such as doped ceria, zirconia, and most mixed oxide ceramics may be readily manufactured by recently developed dry processes.

Introduction

Nanosized materials are on the advance into markets as different as semiconductor manufacturing, catalysis and drug delivery systems (Pratsinis, 1998; Roco & Bainbridge, 2002; Stark & Pratsinis, 2002; Bummer, 2004). Patent and scientific literature on their production and usage has attained an exponential growth phase. While just a few years ago, nanoparticles were only available for a few elements and mostly at an exorbitant price, today, inorganic nanoparticles of almost any composition are offered at more and more competitive prices (Rittner, 2003). With increasing amounts of particles produced, lower prices inevitable require more careful analysis of energy requirements in the

possible processes and may early on enable a both economically and ecologically safe choice on the required technology. The dispute on wet or dry processes has not been limited to classical powder technology, the discussion has even been heated up for smaller and smaller particles. We therefore decided to run a more detailed analysis on the inherent resources for the different processes. In order to compare different methods regarding the resulting environmental loads we made a life cycle inventory using the emissions as CO₂ equivalents. (Godal & Fuglestvedt, 2002) and energy balances as indicators. This approach offers a very reliable method and has been well established for full life cycle assessments (Pennington et al., 2004; Rebitzer et al., 2004).

At present, the liquid and gas-based processes to manufacture fine particles on an industrial level cover a estimated world wide market volume of at least 15 billion US dollars per year (ItN Nanovation GmbH Press report, 2004). With the emergence of numerous new products and nanoparticulate materials (Narayanan & Laine, 1997; Laine et al., 1999; Jensen et al., 2003; Eliezer et al., 2004; Kim et al., 2004), it has been repeatedly speculated that dry processes are more economic and environmentally friendlier than their wet counterparts due to fewer process steps (Pratsinis, 1998). We are putting the hypothesis on the test by starting with a comparison of most classical titania manufacturing and extend the analysis to more complex mixed oxides or metals.

Titania (TiO_2) manufacturing

The classical process

The production of titania particles, (4.35 Mio metric tons / year (2004), mainly pigment) is well studied and therefore serves as a reference case in the present work. While pigmentary titania itself is not a nanoparticulate material due to its large size (220–300 nm) it may well serve as a starting point to compare different processes for fine particle manufacturing. Today, the more traditional wet-phase sulfate process accounts for a big part of titania manufacturing while the newer chloride process mainly works in the gas phase and can result in smaller titania particles and different

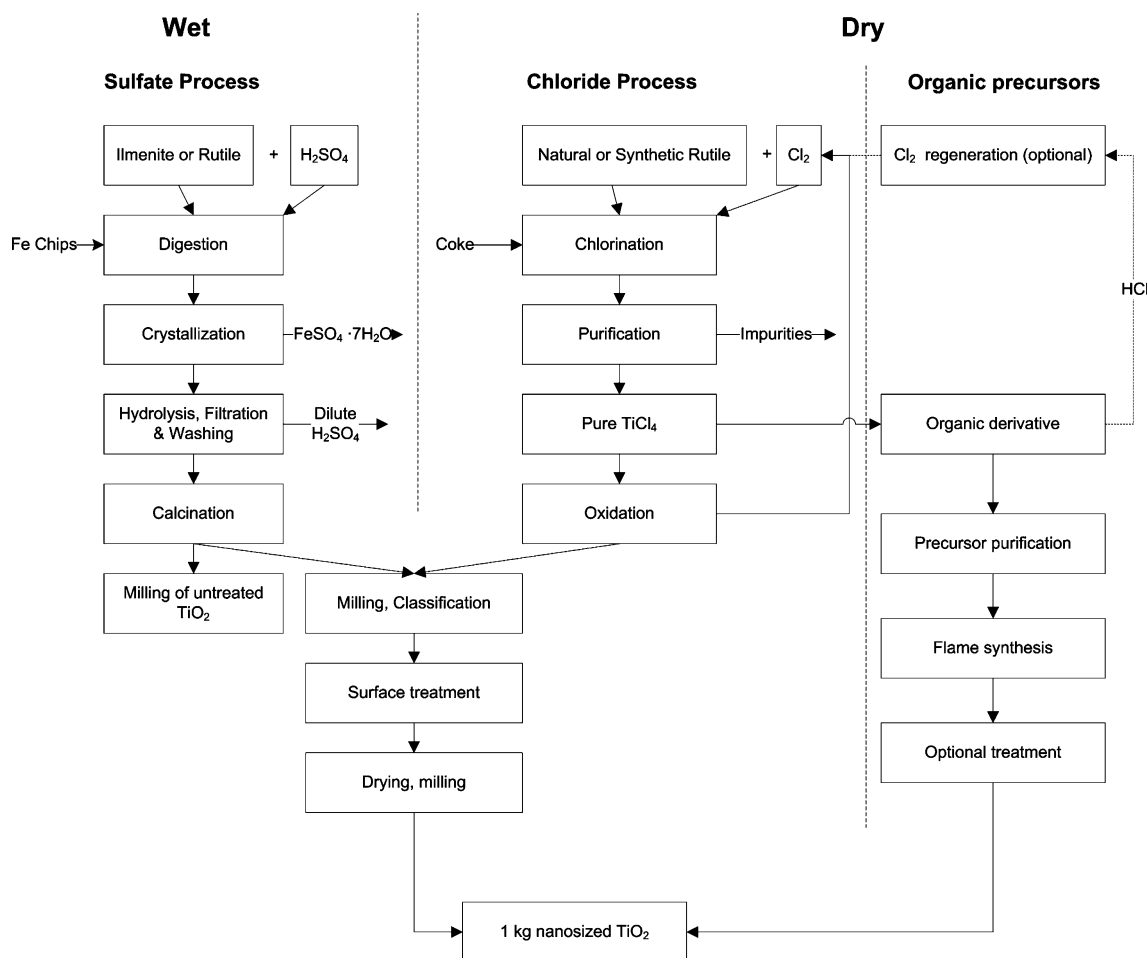
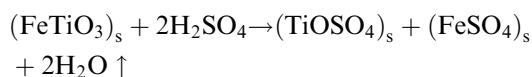


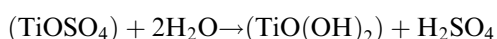
Figure 1. Schematic mass flux for different production processes manufacturing nanosized titania. While the sulfate process is a low-temperature liquid based method, the chloride process uses mainly gases and proceeds at much higher temperature. The use of organic metal derivatives (right) may have advantages in terms of remaining acidity and after-treatment.

particle morphology. In both processes, particles are refined after preparation by milling and surface treatments depending on their final application. Titania manufacturing (Reck & Richards, 1999) starts from ores containing ilmenite (45–60 wt% TiO₂) or rutile (up to 95 wt% TiO₂). Major impurities such as iron, silicon and other transition metals have to be removed to ensure good product quality. Figure 1 compares the liquid (sulfate) and the gas-phase (chloride) process.

More precisely, the sulfate process (Federal Environmental Agency, 2001; Figure 1) starts from ground ore which is reacted with concentrated sulfuric acid, water and steam to form titanium-oxy-sulfate:

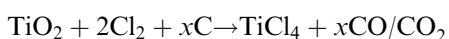


After dissolving TiOSO₄ and FeSO₄ in water, FeSO₄ is precipitated and TiOSO₄ is hydrolyzed to hydrous titanium dioxide according to:

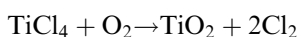


After dehydration by calcination, a relatively coarse titania (TiO₂) is generated with varying contents of rutile.

The chloride process starts with the preparation of titanium tetrachloride from natural or synthetic rutile, petrol coke and chlorine and attains conversions of titania to titanium tetrachloride of 90–100% and a chlorine usage of 98–100% (Federal Environmental Agency, 2001).



Impurities (transition metals and silica) are removed as chlorides by fractional distillation. The pure titanium tetrachloride is burnt to titania in an oxygen flame where it forms the final fine product particles:



Washing is required to remove remaining chlorine. The chlorine and low amounts of hydrogen chloride are recycled back to the chlorination step. In both processes, a shuttle compound (Cl₂ or H₂SO₄) enables to keep by-products at a low level and provides an elegant way to manufacture titania powders. The present comparison on more novel nanoparticles production processes is first made for titania, in a

second step for an example of a heavier oxide (zirconia).

Newer processes

More recently, several new gas-phase processes have been proposed for nanoparticles synthesis: Plasma and laser assisted particle synthesis involve evaporation and optional reoxidation of the metal or metal mixture and gives access to a broad variety of materials (Besling et al., 1998; Kruis et al., 1998; Buchner et al., 1999; Buchner et al., 2001; Stark & Pratsinis, 2002; Jensen et al., 2003; Chen & Shen, 2004; Eliezer et al., 2004). Both academic and industrial research have resulted in a series of organic precursors for flame based production methods (Zhu & Pratsinis, 1996; Laine et al., 1999; Madler et al., 2002a; Jensen et al., 2003). These flame spray processes resemble the industrially used flame aerosol process and account for increasing volumes of most different oxide particles. Beyond alkoxides (Kammler & Pratsinis, 2001), glycolates (Narayanan & Laine, 1997), carboxylates (Loher et al., 2004) even metal organic compounds (Madler et al., 2002a) have been successfully used for the preparation of different oxides. Figure 1 includes a process diagram for the use of such organic precursors and shows how the latter may be related to the production of titanium chloride for titania manufacturing. Flame based nanoparticle production (Wenger & Pratsinis, 2000; Kammler et al., 2001; Gutsch et al., 2002; Madler et al., 2002a; Althaus et al., 2003) using organic precursors may use purified titanium tetrachloride from the first part of the chloride process. It is further reacted with the corresponding organic compounds and the hydrochloric acid is optionally recycled by regeneration to chlorine. While titania itself is well accessible by the previously described chloride or sulfate process, most other oxides can only be prepared using organic precursors since most metal chlorides are not volatile enough. Combustion of the organic derivative in a flame today results in complex oxides or even salt nanoparticles (Loher et al., 2004). (Figure 2)

Energy assessment for traditional titania processes

The corresponding system boundaries are set from cradle-to-gate, starting with the raw materials

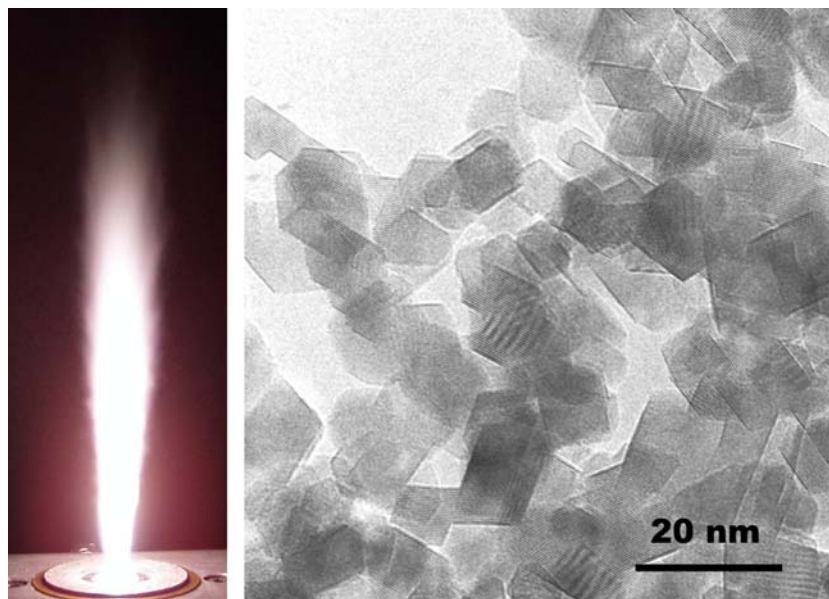


Figure 2. Flame reactor (left) producing ceria nanoparticles and transmission electron microscopy image (TEM) of the resulting products (cerium oxide, right). Product properties of such nanoparticles may be varied in terms of size, degree of agglomeration and crystallinity.

Table 1. Energy balance for the production of one ton titania (Federal Environmental Agency, 2001)

Process	Energy [GJ/t]	TiO ₂ manufacturing	Follow up treatment	Acid concentration and filter salt decomposition	Total
Sulfate ^a	Electric Energy	1.5–2.3	0.6–1.4	0.1–1.3	2.2–5
	Steam	3.7–7.7	6.7–10.4	0–5	10.4–23.1
	Gas	7.3–11.8	2.3–4.2	0–0.1	9.6–16.1
	Coal	–	–	5–8	5–8
	Total Energy	12–20	9–14	5–15	32–40
Chloride ^b	Electric Energy	1.5	0.8	–	2.3
	Steam	1.7	7.6	–	9.3
	Gas	2.8	4.2	–	7
	Coal	–	–	–	–
	Total Energy	6	12.6	–	~19

^aThe range represents data from several different plants with varying performance.

^bData from one plant in Europe.

(ore) and considering processes to the finished product (nanoparticles; ISO 14040, 1997). This procedure excludes applications and allows a comparison of different production processes. The energy data and green house gases for the chloride and sulfate process have been previously calculated for TiO₂ production in Europe and can be obtained by the database ecoinvent (Althaus et al., 2003). The overall energy balances are displayed in Table 1. The sulfate process can be divided into three main steps: titania manufacturing, follow-up

treatment (milling and drying) and sulfuric acid recycling (Federal Environmental Agency, 2001). Since large amounts of liquid are transferred and heated, the main energy consumer is the titania manufacturing step. The energy consumption for the recycling of the acid again reflects the large amounts of liquids in this process. The chloride process can be divided into two main process steps: titania manufacturing and follow up treatment (Federal Environmental Agency, 2001). The energy consumption for the follow up

treatments is similar to the liquid phase process while the gas-phase process needs considerably less energy for the titania manufacturing.

Energy assessment for newer titania processes

For organic precursors, the main factor determining energy requirements is the maximum metal concentration in the solvent as evidenced by the data in Table 3. It can be expressed as the ratio of solvent to metal oxide mass (solvent/MeO_x) and attains up to 7.7 kg precursor/ kg metal oxide in the carboxy process (Loher et al., 2004) or 3.5 kg precursor/ kg metal oxide for the corresponding propoxide (Kammler & Pratsinis, 2001; Tsantilis et al., 2002). For shorter carboxylic acids, much higher metal concentrations are possible (see Table 3). The resulting green house gases contain both manufacturing of the organics and combustion. They are significantly higher than in the chloride or sulfate process (Table 2). Since the production of titanium tetrachloride accounts for around 60% of the CO₂ emissions in the chloride process, 2 kg CO₂ equivalents were taken out of the energy balance and added to the case of organic precursors to consider the titanium tetrachloride manufacturing. For the production of the carboxylic acids, the CO₂ equivalent of similar fatty acids was taken as a representative (Althaus et al., 2003).

Table 2. CO₂ emission for titania production- classical processes

Process type	Precursor	kg precursor/kg TiO ₂	kg CO ₂ /kg TiO ₂
Sulfate Process	TiO ₂ /H ₂ SO ₄	2	5 ^a
Chloride Process	TiCl ₄	2.4	4 ^a

^aAs obtained according to (Althaus et al., 2003).

Table 3. CO₂ emissions for titania production using different organic precursors

Precursor	kg precursor/kg TiO ₂	Production ^a kg CO ₂ eq/kg TiO ₂	kg CO ₂ ^b /kg TiO ₂	kg CO ₂ ^c /kg TiO ₂	Total CO ₂ emission/kg
Ti-octanoate	7.8	10	18	2	30
Ti-pentanoate	5.7	7	11	2	20
Ti-isopropoxide	3.6	6	7	2	15

^aCO₂ equivalents for the production of *x* kg organic precursor (Althaus et al., 2003).

^bCO₂ emission by combustion of the precursor.

^cCO₂ emissions for the production of TiCl₄.

This unfavorable comparison in the case of titania can mainly be attributed to the low atomic mass of 48 g/mol for Ti and a high valency of 4. Chemistry therefore dictates a high content of organic residues and resulting high carbon dioxide production. Clearly, elements with higher atomic mass and low valency will perform better. Therefore, we compared the different flame processes for zirconia manufacturing to alternatives such as liquid phase or plasma synthesis.

Zirconia (ZrO₂) – manufacturing

As an example of a heavier oxide, zirconia was chosen for its high industrial relevance and abundant data available on the zirconia production by different processes.

The classical zirconia process

Zirconia can be produced in a similar manner as titania by the chloride process (Figure 3; Burgess, 1922; Gmelin, 1958). Petrol carbon and ZrO₂ ore (Baddeleyite) are mixed and reduced in an electric arc furnace to a mixture of carbide and carbonitrid at 900–1200°C (Gmelin, 1958). The reduced product is crushed and heated in an atmosphere containing chlorine, resulting in the formation of zirconium tetrachloride. After distillation, zirconium tetrachloride is hydrolyzed with water to zirconia and hydrochloric acid. The crude zirconia product is calcined to remove water and milled to the desired product size. Dopants such as yttria or calcia for partially or fully stabilized zirconia are added during the precipitation step. High temperature calcination is required for homogeneous distribution of the stabilizer within the zirconia.

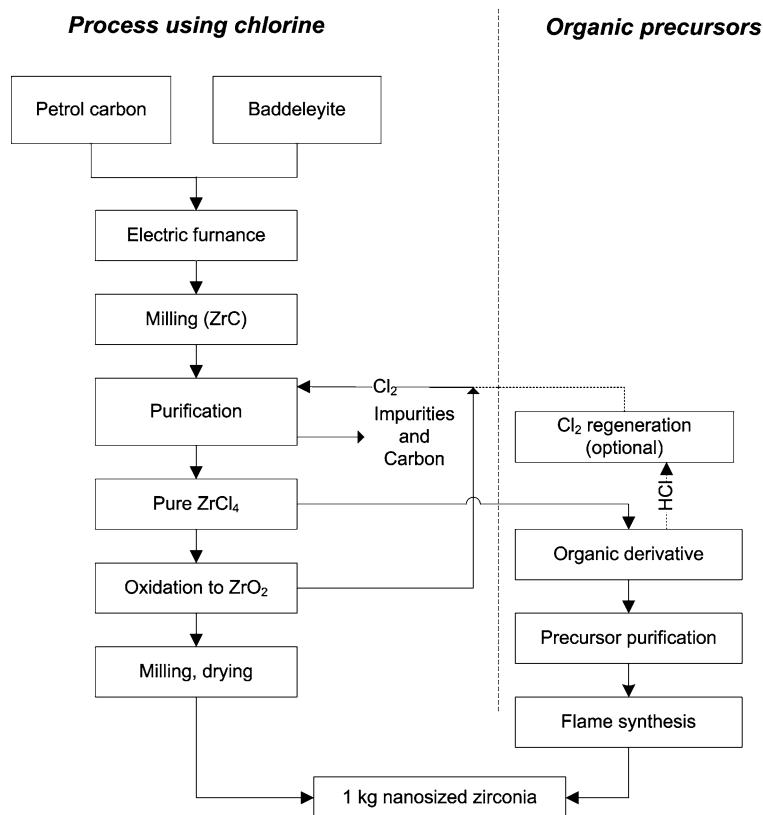


Figure 3. Schematic mass flux of zirconia production processes using chlorine (left) or organic precursors (right). As in the case of titania, the ore is extracted by chlorine and directly oxidized (left) or converted to an organic derivative (right) prior to combustion.

Newer zirconia manufacturing processes

Both flame spray based production method and sol-gel wet phase methods require organic zirconia precursors. They are prepared by reacting zirconium tetrachloride with the corresponding alcohols or carboxylic acids. As in the flame based titania process hydrochloric acid may optionally be recycled by regeneration to chlorine (Figure 3). The zirconia alkoxides or carboxylates may either be hydrolyzed (wet phase preparations) or burnt to the oxide and combustion products.

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Table 4. Relative CO₂ emissions for zirconia production starting from ZrCl₄

Methods	kg precursor/kg ZrO ₂	Production ^a kg CO ₂ eq/kg ZrO ₂	kg CO ₂ ^c /kg ZrO ₂	Total CO ₂ emission ^d /kg CO ₂ /kg ZrO ₂
ZrCl ₄ , hydrolysis	1.9	–	–	5
Zr-octanoate	5.4	7	11	20
Zr-pentanoate	4	5	7	14
Zr-isopropoxide	2.7	3	4	9
Zr, Plasma	0.7	40 ^b	–	~40
ZrO ₂ , nano-milling	1	5	–	~35

^aCO₂ equivalents for the production of x kg organic precursor (Althaus et al., 2003).

^bCO₂ equivalents for electricity and production of Zr metal (DEFRA, 2004).

^cCO₂ emission by combustion of the precursor.

^dIncluding estimated CO₂ emissions for the production of ZrCl₄.

Plasma processes (De la Veaux & Zhang, 2004) offer an alternative way to produce numerous nanoparticles mainly in the gas phase. The plasma process vaporizes solids, powders or wire and condenses or reoxidizes the resulting hot vapor to metal or oxide nanoparticles. A dilution gas separates the constituents during preparation and allows to access very small particles.

Energy requirements for zirconia manufacturing

The energy requirements of the production of zirconium tetrachloride are significantly higher than for titania tetrachloride due to the preparation of zirconium carbide. This step is necessary for all processes making zirconia. If we only compare different processes, we may subtract this first general step which stays the same for all zirconia manufacturing processes. Purification is more energy intensive due to the higher boiling point of zirconium tetrachloride. The product itself, zirconia, is much harder than titania and requires significantly more energy for milling of agglomerates after calcination which may be estimated to result in at least an additional 2 kg of carbon dioxide. Nano-milling of micron-sized zirconia is a much more demanding process (Table 4). The corresponding energy requirements for nanoparticles of a heavy oxide now reveal that significantly less CO₂ equivalents are produced per kg of ZrO₂ if compared to titania production and using organic precursors (Table 4). This can mainly be attributed to the higher molar weight of ZrO₂ with 123 g/mol if compared to TiO₂ with 80 g/mol. Or, since the atomic mass of zirconia is much higher than titania, more product is accommodated in one volume of precursor. Even though no additional high energy consuming milling or drying are necessary in the flame process, the conventional liquid process is still favorable in terms of CO₂ emissions. The differences, however, have become rather small and increased quality may allow to shift to the relatively new flame spray synthesis.

Unfortunately, maintenance of a plasma requires very high temperatures to achieve the necessary gas conductivity and results in significant consumption of expensive electrical energy. For one kilogram zirconia ZrO₂, 0.7 kg (8 mol) of zirconium metal is needed. Assuming 25% of Zr to be ionized, evaporation at 4400°C (Winter, 1986) and requires 6.7 MJ for full dispersion in the gas.

Plasma synthesis requires a dilution gas, typically argon which has to be heated to 8–10,000 K (Fauchais et al., 2001). At a solid loading ϕ of 0.01 g oxide / gram gas, this demands 142 kW h or about 36 kg carbon dioxide equivalents per kg zirconia (DEFRA, 2004; electricity production with oil). At least another 6 kg CO₂ equivalents would have to be added for the Zr metal synthesis (Table 4). While individual plasma synthesis units may be quite different, the overall energy requirements are inherently higher than in wet-phase synthesis. It shall be noted though that for metals, plasma synthesis is currently the only option for large scale synthesis of metal nanoparticles.

Mixed oxide nanoparticles

Today, an increasing number of complex oxides are manufactured mainly using organic precursor based flame synthesis. Amongst others, nanoparticles of ceria, yttrium stabilized zirconia (YSZ), ceria-zirconia (CeZrO₄) or other mixed-oxides (Backman et al., 2004; Marchal et al., 2004; Makela et al., 2004) in general are produced next to optical components or polishing additives (Madler et al., 2002b). Milling has been suggested as a possible route to nanoparticles and has successfully be implemented in YSZ production (Michel et al., 1993). It requires a lot of electrical energy: As an example, milling 1 kg of a mixed-oxide to 100 nm (Schubert et al., 1990) would require a minimum of 100 kW h with an efficiency of 10% (Loeffler & Raasch, 1992) for the ball mill. This energy consumption corresponds to 30 kg of carbon dioxide (DEFRA, 2004). While some mixed oxides are now accessible by wet-phase preparations, a large number of compounds is still manufactured by solid reaction of the constituents at high temperature. The resulting bulk product must be intensively milled in order to obtain a nanoparticulate product. Newer production methods are therefore mainly favorable for the production of complex compositions or where a specific quality (e.g. purity for optics) is required.

Conclusion and outlook

The energy requirements for nanoparticle manufacturing may be rationalized for different product

classes and complexity. For simple oxides such as silica or titania, liquid and gas-phase processes are coexisting now for over two decades. Advanced ceramics, functional inorganic materials and more complicated compositions may require sophisticated wet-phase preparations, often at low concentration. Or, they may not be accessible by conventional synthesis at all. Numerous products are now accessible by flame- or plasma synthesis and similar dry, high-temperature methods. In organic precursor assisted flame synthesis, the inherently required amount of organic residues determines the overall energy requirements and depends on the mean atomic mass of the desired compounds and their mean valency: Classical preparations are favorable in the case of simple composition, low mean atomic mass and high valency. Novel preparation routes such as organic precursor assisted flame synthesis may preferably be used for complex materials containing heavier elements. Abundant novel materials, however, are inaccessible by classical processes and must be manufactured using new processing tools. In the case of metals, plasma synthesis is currently the only way to access metallic nanoparticles in the gas phase. Inherently high energy costs may hinder the development of a real commodity market for such materials. Milling may often accompany any process mainly for deagglomeration and results in significant powder quality enhancement. Rapidly dropping prices and an increasing number of suppliers corroborate the on-going market segmentation in terms of preparation methods and product properties.

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