



## Review

# Permanent agglomerates in powdered nanoparticles: Formation and future prospects



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## ABSTRACT

In pace with the advancement in nanotechnology, different types of nanoparticles, have been largely produced and are now commercially available. Considering both transport and storage efficiencies, these nanoparticles are normally sold in powder form. Unfortunately, one of the major issues associated with nanopowders is that they tend to form hard agglomerates. These hard agglomerates are hardly segregated even external driving force (such as sonication) has been provided. Such kind of agglomerates is undesired as it reduces the surface area-to-volume ratio of the nanoparticles and thus limits their application performance. Formation of hard agglomerates has to do with the drying step, which is also the main step to convert nanosuspensions into dry powders. Thus, the present work aims to provide a comprehensive review of the occurrence and factors that lead to the hard agglomerates formation in nanopowders. In particular, the creation of interparticle necks upon sintering is reviewed. Approaches to avoid hard agglomerate formation, as well as dissolution of the interparticle necks are also considered here. Lastly, prospects of these nanopowders (if formation of hard agglomerates is unavoidable) in the future nano-research are discussed.

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## 1. Introduction

Nanoparticles, which are the fundamental building block of nanotechnology, have been industrially employed in various applications [1] as well as being vigorously investigated for more potential usages in multidisciplinary fields. All these advancements are mainly due to the specific physicochemical properties of nanomaterials, which could not be attained by the bulk counterparts [2]. In this regard, many manufacturers and distributors of nanoparticles are ready in the market to fulfil the needs of nanoparticles supply. It is interesting to find that commercial nanoparticles are normally offered [3] and shipped in dry

powders form [4]. In some cases, the dry powders are further processed into capsule or tablet form. The advantage of selling dry powder instead of particle suspension is the subsequent reduction in both transport and storage costs [5].

Production of nanopowders can be rather straightforward, begin with the synthesis of nanoparticle suspension from their precursors, then, the formed nanoparticles are being dried into powder form. To date, various drying methods have been implemented for conversion of nanoparticle suspension into nanopowders, these include thermal drying, spray drying, supercritical drying, spray drying, alcohol dehydration, and etc. [6–8]. Meanwhile, nanopowders also can be produced by broken down their bulk coarse solids through mechanical ways. Ball milling [9–11] and cryogenic grinding [12] are the developed

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mechanical ways to achieve the top-down synthesis. In fact, the top-down approach that involves dry/wet milling for the production of large amounts of nanopowders offers advantages such as scalable and simple operation [13].

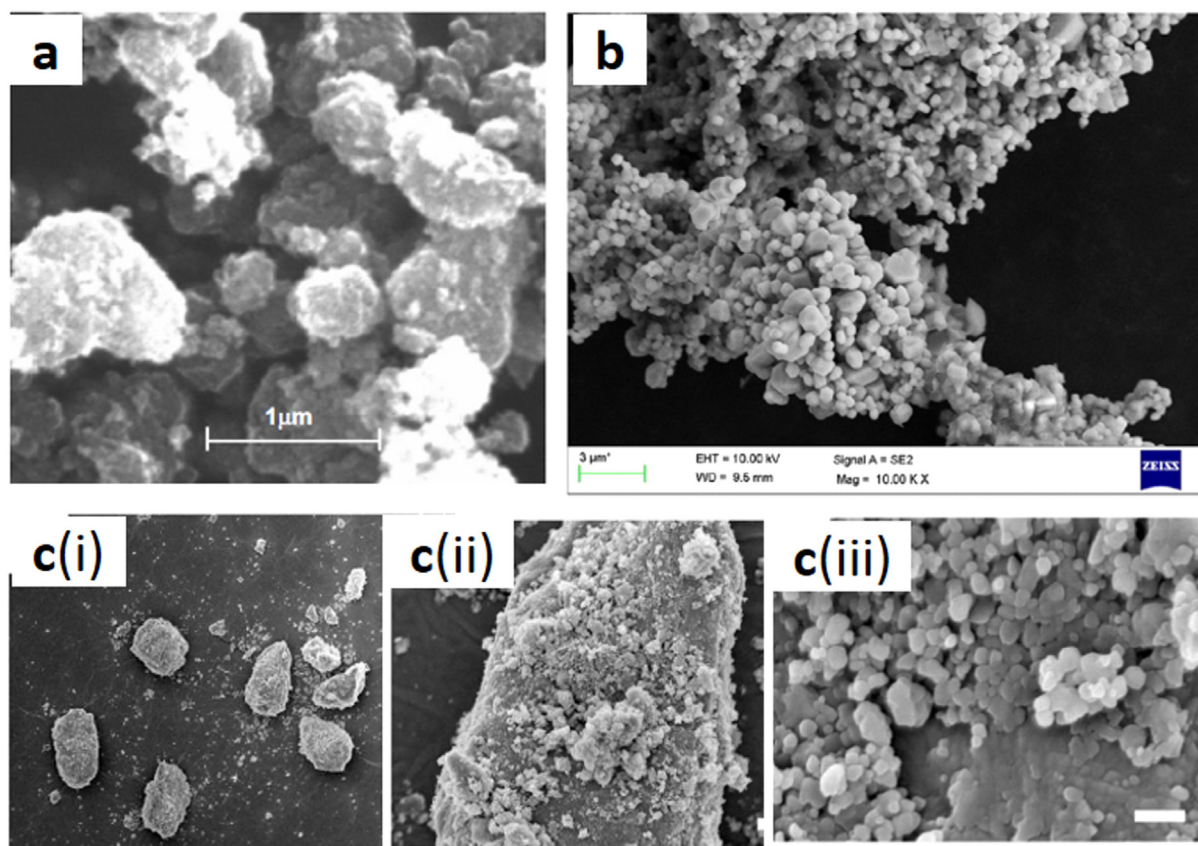
However, despite the established profile of nanopowders as mentioned above, challenge arose when the end user attempts to re-disperse these nanopowders into suspension in which the dispersed particles tend to be present in agglomerates form instead of singly dispersed particles. This circumstance is unwanted as it reduces the surface area-to-volume ratio and thus limits its applicability. For instance, in the usage of nanoparticles for in situ underground remediation, formation of agglomerates reduce the reachability of nanoparticles to the contaminated sites and subsequently suppress the effectiveness of the whole in situ remediation process [14]. Meanwhile, in the effort to incorporate multi-walled carbon nanotubes (CNTs) into polymer matrix to synthesis mixed matrix membrane, Ahmad et al. [15] found that the CNTs powder, despite being sonicated before added into the polymer solution, has formed groups of clusters on the membrane surface. Such structure was found to retard functionality of the mixed matrix membrane for CO<sub>2</sub> separation. Similarly, particle agglomeration is undesired for in vivo biomedical uses [16]. In fact, the problem associated with the nanopowders turn into agglomerates after dispersed into liquid medium is commonly experienced by researchers throughout the world. This has been a hot discussion topic in online research sharing platform (e.g. *ResearchGate* [17]).

In view of this, the present article aims to provide a review on the issues pertinent to the occurrence of agglomeration in nanopowders, followed by discussion of the factors that leading to hard agglomerate formation in nanopowders. In addition, available mathematical model used to predict the proneness of nanopowders to form agglomerates

is provided. Then, available strategies to avoid formation of hard agglomerates in nanopowders are reviewed. Lastly, the potential prospects of these nanopowders (if formation of hard agglomerates is unavoidable) in future works are discussed.

## 2. Issues pertinent to the occurrence of agglomeration in nanopowders

Bowen et al. [18] stated that nanopowders may consist of clusters with size about 10–20 times larger than the primary nanoparticles. As shown in Fig. 1, nanoparticles in dry powder form normally present as groups of large clusters. When these nanopowders are dispersed in a liquid medium, they tend to form polydisperse agglomerates instead of singly dispersed particles. This phenomenon is commonly found even sonication has been provided to break the agglomerates during the suspension preparation process (as listed in Table 1). For instance, Choi et al. [19] prepared Fe<sub>2</sub>O<sub>3</sub> nanoparticle dispersion from a commercially available powdered source via sonication; they reported that the particles present as agglomerates of diameter > 600 nm even though the primary particle size was < 50 nm. Tso et al. [4] dispersed SiO<sub>2</sub>, TiO<sub>2</sub>, and ZnO nanopowders in aqueous solution using ultrasonic disruptor; they found that the particles appeared as agglomerates of size 146 nm, 225 nm, and 244 nm, respectively. Similarly, Zhang et al. tested dispersibility of various commercialized nanopowders (e.g. TiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, ZnO, NiO, Silica) and found that none of them can be dispersed into their primary sizes [3]. Based on the data summarised in Table 1, it is obvious that the formation of agglomerates of particles (instead of individual particles) is a commonly encountered issue after dispersing nanopowders into liquid suspension form.



**Fig. 1.** Scanning electron micrograph image of (a) commercialized dry powder form of TiO<sub>2</sub> nanoparticles (Reprinted with permission from [3], Copyright 2007, Elsevier Ltd.), (b) ZnO nanoparticles (Reprinted with permission from [20], under Creative Commons Attribution Licence (CC BY)), and (c) C<sub>60</sub> nanoparticles formed by rubbing bulk solid of C<sub>60</sub> between glass slides in which agglomerates of nano-sized particles were found associated on the surface of large particles (i. scale bar = 10 μm; ii. scale bar = 2 μm; iii. Scale bar = 200 nm) (Reprinted with permission from [21], Copyright 2013, Rights Managed by Nature Publishing Group).

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