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AEROSOLISATION DE PARTICULES DE CARBURE DE SILICIUM SUR DES PERIODES LONGUES

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TITLE

Aerosolization of silicon carbide particles for prolonged durations

RESUME

Certaines applications industrielles utilisent de la poudre de carbure de silicium sur des durées allant de plusieurs heures à plusieurs mois. De telles durées induisent des problématiques d'émission (exposition des opérateurs, rejets à l'environnement) mais aussi de modification de la poudre elle-même. Ces problématiques sont très peu considérées à ce-jour, les tests de pulvérulence étant principalement centrés sur les quantités de matière émises, et ce pour des durées plus courtes.

Les résultats présentés ici portent sur l'étude par agitateur vortex [3, 4] de la pulvérulence en masse et en nombre de deux types de poudre de SiC de diamètre médian (x_{50}) de 66 µm et 38 µm, sur des périodes de 6 heures. Le mécanisme de génération de poussières peut comprendre la libération d'aérosol due à l'attrition des particules due à l'impaction entre particules et à la paroi des particules. Cette étude souligne la nécessité de procéder à des essais à long terme sur la poussière pour les matériaux durs comme le SiC et à la caractérisation des propriétés des matériaux en vrac en raison de la génération et de la libération de poussières. En outre, les résultats peuvent être trouvés dans le matériau en vrac pour des applications à long terme basées sur la poussière.

ABSTRACT

Silicon carbide (SiC) particles used in long-duration industrial applications releases potentially hazardous dust which can also change the bulk material quality. However, most dustiness tests do not study dust released over long durations nor do they measure the effect of dust generation on the bulk powder as they emulate applications lasting for short duration (few seconds to minutes) which has minimal effect on the bulk sample.

In this study, we test the number and mass dustiness of two different samples of SiC powders with median particle sizes (x_{50}) of 66 µm and 38 µm, over six hours using a vortex shaker. The dust generation mechanism might include the release of aerosols due to the attrition of particles owing to inter-particle and particle-wall impaction. This study emphasizes the need for long duration dustiness tests for hard materials like SiC and characterization for change in bulk material properties due to dust generation and release. Furthermore, the results can aid in selecting the bulk material for long-term applications based on dustiness

MOTS-CLÉS : Aérosolisation; mécanisme de génération ; particules de carbure de silicium ; agitateur vortex / **KEYWORDS:** Aerosolization; Generation mechanism; Silicon carbide particles; Vortex shaker

1. INTRODUCTION

Hard particles, such as silicon carbide (SiC) having diameters in the range of 30–100 µm are widely used in high endurance applications such as the production of abrasives, wear-resistant machineries (Harris, 1995) and more recently as heat transfer fluid (HTF) in solar thermal (García-Triñanes et al., 2016). Stresses generated due to continuous conveying and processing of the bulk leads to aerosolization of dust particles from the bulk material. Further, it can also lead to attrition of particles which can potentially influence the physical, mechanical and thermal properties of the material.

This study analyses the effect of time-scale on powder dust generation while considering the effect of dustiness testing on the particle size distribution and particle shape properties of the tested samples compared to their pristine state. Based on the time-evolution of dust generation, we propose stages of dust generation mechanisms which can possibly provide explanations concerning the emission of dust and its subsequent effect on physical properties of the powder sample. Such information can be useful in understanding the risks from the powders being used in long-term industrial applications which can span anywhere between few weeks to months.

2. MATERIALS AND METHODS

2.1. Silicon carbide particles

Two sets of silicon carbide powders (CAS Number: 409-21-2), SiC F220 and SiC F320 (from Mineralex, France) were used "as-received" following the EN standard 15051 (CEN, 2006). The powder test samples were characterized for volumetric and number size distribution by laser diffraction (3D measurement) and image analysis (2D measurement), respectively. Also, the specific surface area and moisture content were measured using the gas adsorption surface area analysis (BET) and the halogen moisture analysis, respectively. The material parameters are shown in Table 1. The size analysis was repeated three times. The values for particle density was provided by the manufacturer.

Table 1. Powder properties of SiC F220 and F320 test samples									
Properties	Units	SiC F220			SiC F320				
Particle density	kg/m³	3,210			3,210				
Size distribution by volume ^b	μm	x ₁₀ 39	x ₅₀ 68	<i>x₉₀</i> 115	x ₁₀ 25	x ₅₀ 38.5	<i>x₉₀</i> 60		
Specific surface area	m²/g	0.029			0.052				
Moisture content	%	< 0.1			< 0.1				

2.2. Dustiness tester: The Vortex shaker method

The vortex shaker (Le Bihan, 2014; Morgeneyer, 2013) was used as the dustiness tester due to its low requirements of sample sizes, ease of operation and the ability to retain the powder sample after the test. The experimental setup used by (Chakravarty et al., 2017; Jensen, 2012) was adopted for the present study, and has been comprehensively described in the recent publication (Chakravarty et al., 2018).



Figure 1: The Vortex shaker experimental setup

The released aerosol was sampled using a respirable cyclone (BGI GK2.69). Three trials were performed for each of the two powders, F220 and F320. Each test used 2 g of powder weighed with an accuracy of ± 0.001 g using an analytical balance (MS1003S, Mettler-Toledo, Inc., Columbus, OH, USA), manually filled in a centrifuge glass tube (diameter 0.025 m, height 0.15 m).

Aerosol is generated through the turbulent agitation of a powder-filled glass test-tube mounted on a digital vortex shaker (VWR Signature Digital Vortex Mixer). Prior to starting the vortex shaker, the APS and CPC sampling were turned on along with the inlet flow (4.2 L/min or 7e-05 m³/s) and dilution flow (7.4 L/min or 1.2e-04 m³/s) for 2 minutes. The VS operated at 1500 rpm, was run for six hours to test the powder samples, with a short break of 5 minutes after every 1-hour interval to avoid the overheating of the electric motor.

Since the air flow is not interrupted, the peaks in the dustiness variables are entirely due to the mechanical action of the vortex shaker. Using a low-pressure pump (0.6 L/min or 1e-05 m3/s, Gilian LFS-113DC) attached to the sampler (MPS®), dust particles were collected on Quantifoil copper-carbon grids (Oxford Instruments, UK) (R'mili et al., 2013). The dust particles confined in these grids were further analyzed for their morphology using a Transmission electron microscope (TEM, JEOL JEM-2100F, operated at 100 kV).

3. RESULTS AND DISCUSSION

Both SiC powder samples release respirable fraction of dust particles but differ in dust generation behaviour. The dust generation mechanism might include the release of aerosols due to the attrition of particles owing to inter-particle and particle-wall impaction.

During the six-hour test, the aerosol mode particle size by mass (Fig. 2 top) for F220 shows a greater deviation towards smaller particle sizes compared to the F320 sample. Aerosol generated from F220 and F320 can be classified into four stages based on the evolution of the total respirable aerosol counts (Fig. 2).



Figure 2: Evolution of aerosol mode particle size (top) and total respirable aerosol particle counts (bottom) measured for F220 and F320 over stage I, II, III and IV. The countsare summed over 20-minute intervals. The error bars show the standard deviations calculated from three repeated trials.

Dust sampling time	25th min	to 30 th min	350 th min to 360 th min		
SiC F220	а 1 µл	200 nm	c l µn	200 nm	
SiC F320	e 1 pr	200 m	g 1 pm	200 nm	

Figure 3: TEM micrographs for SiC F220 (top; a,b,c,d) and SiC F320 (bottom; e,f,g,h) aerosol particles captured at 25th- 30th minute interval and from 350th minute – 360thminute interval.

F220 and F320 undergo mechanical stresses due to inter-particle collisions and particle-wall impacts in the VS. Although hard materials like SiC particles are resistant to breakage or fragmentation, they can undergo attrition due to abrasion or combination of fragmentation and abrasion, depending on the stresses they are subjected to (Ness and Zibbell, 1996; Quercia et al., 2001). Generally, the abrasion of particles leads to the rounding of the primary mother particles by reducing surface asperities resulting in the generation of fine-

scale particles, thus creating a bi-modal number size distribution without any significant changes in the PSD by volume (Yang, 2003). The detailed analysis of the particle shape characterization is shown in our recent publication (Chakravarty et. al., 2018)

Based on the present results of the dustiness tests of SiC particles, the initial dust generation strongly depends on the population size of the aerosolizable particles present in the bulk material. The abrasion of larger particles generates fine aerosolizable particles and is a crucial part of the overall dust generation mechanism. The dust generation mechanism can be broadly divided into two stages as mentioned by (Chakavarty et al., 2018), firstly direct release of aerosolizable primary particles, followed by release of aerosolizable fines generated through the attrition of larger primary particles.

4. CONCLUSION AND PERSPECTIVES

Particles used for applications extending over a long period of time, such as HTFs in CSP solar thermal plants require results from sufficiently long dustiness tests to support the selection of material and quantify the risk associated with the handling of new and used particles. In this case study, we investigate dust release over six hours for two potential silicon carbide HTFs (F220 with by volume = 68 μ m and F320 with by volume = 38 μ m) using the VS method.

Test results show the release of the respirable fraction of dust particles from both samples, but F220 is found to be more prone to generate dust than F320. For F320, an initial rise in the aerosol release is followed by a gradual decrease with time, following a power law distribution. Unlike F320, aerosol generation and release from F220 is more complex and the dust released over time shows an increasing trend. F220 and F320 not only differ in dustiness but also in the mechanism of dust generation and release. Two dust generation mechanisms are proposed which can potentially explain the dustiness behaviour of F220 and F320 over a 6-hour duration. Results from the dustiness measurement, TEM micrographs of the aerosol particles and characterization of pristine and tested powder samples by their size and shape suggest that the dust generation from F220 and F320 is related to the presence of aerosolizable fine-scale particles already present in the bulk as well as the particles generated from powder attrition.

The tested F220 powders show changes in particle size distribution and shape properties compared to their pristine form, indicating abrasion as the dominant source of attrition. On the contrary, the F320 powders show barely any changes in particle size distribution or shape factors with vortex testing.

Understanding the difference of aerosol generation behavior based on particle shape requires further work and the effect should be more observable for materials softer and more fragile than SiC F220 and F320 bulk samples could be further characterized by their particle size distribution and shape properties for every hour to analyze the evolution of particle properties with dust generation. The handling of F220 (SiC 220) may generate fine-scale particles which may affect the safe and efficient operation of SiC HTFs in CSP plants.

Our study underlines the importance of characterizing both before and after the dustiness test, as changes in its properties are crucial to understand the underlying dust generation mechanisms.

In the industrial world, powders which have already undergone an ageing process for several weeks or months are employed in the CSP plants. Studying such aged powders with respect to their dust generation behavior appears worthwhile. Further studies are necessary to investigate its potential greater suitability for long-term uses.

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