



next-CSP

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WP1 – Assessment of particle suspension as heat transfer fluid and storage material

Deliverable D1.1. Report on particle selection for solar heat capture and storage

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1. Objectives of D 1.1.

The objective of this deliverable is to summarize the selection of a particulate material for a UBFB or dense up-flow powder circulation system in solar energy capture and storage systems. The main criteria for selecting the material are identified to define a suitable powder for solid/gas suspensions that can satisfactorily be applied as heat transfer fluid (HTF) or heat carrier and thermal energy storage (TES) material.

The assessment is based on thermophysical and thermomechanical properties, towards attrition behaviour, and considering health and environmental hazards of the candidate powders. Finally, a cost comparison of the different candidate powders is presented.

2. Selection of Potential Particulate Materials

The temperature in the solar receiver could reach 1000 °C, making inorganic non-metallic particles the material of choice. A distinction can be made between natural minerals and heat-treated minerals or ceramics. In the following silica sand, quartz sand, cristobalite, alumina, silicon carbide, sintered bauxite, and olivine are evaluated. Softer minerals such as calcium carbonate or barium carbonate were not included because of their thermal instability at higher temperatures (≥ 900 °C) and their high degree of particle fracture and attrition.

The selection of candidate particulate materials is a function of the solid particle receiver employed. The particle size is limited by the employed particle circulation system. The use of a UBFB or dense up-flow receiver narrows the range down to Geldart group A or near-B particles. Coarser particles (group B and D) require a high fluidization velocity. Since the fluidization air will exhaust the UBFB at the bed temperature, sensible heat loss increase significantly with increasing air velocity. Falling particle receivers commonly use large particle sizes, in the range of 300 to 700 μm , however with a reduced heat transfer coefficient as a consequence [1] : the size of the particle indeed influences the thermal bulk conductivity and determines the heat transfer coefficient. Moreover, one should bear in mind that not all particle sizes are commercially available.

Suitable particles for UBFB receivers and associated storage should have: (i) attractive thermophysical properties determined by density, thermal conductivity and specific heat capacity; (ii) a high thermal resistance; (iii) high fracture resistance, and (iv) attractive rheological properties. Furthermore, (v) the health and safety hazards should be taken into account and (vi) the environmental impact should be reduced to an acceptable level. A suitable particle should moreover be (vii) inexpensive and available in large quantities.

Focus in this Deliverable lies on a particulate material selection for the UBFB or dense up-flow receiver. The different gas/solid contacting modes in solar particle receivers are reviewed in detail by Zhang et al.[2]. The considered materials, with their respective material properties are given in Table 1. Possible particle size ranges depend upon the pre-treatment of the minerals, but are all commercially available with required 30 – 100 μm range.

Table 1: Particulate material properties of the particle candidates

Material	Composition (a)	Density [kg/m ³]	Sintering temperature [°C] (b)
Silica sand	SiO ₂	2610	1760
Cristobalite	SiO ₂	2330	1714 (β)
Quartz	SiO ₂	2610	1670 (β tridymite) 1714 (β cristobalite)
Alumina	Al ₂ O ₃	3960	2072
Sintered bauxite	Al ₂ O ₃ , aluminosilicates	± 3900	~1550
Silicon carbide	SiC	3210	> 1700
Sintered bauxite (proppants)	Al ₂ O ₃ , SiO ₂ , (TiO ₂ , Fe ₂ O ₃)	± 3600	~1550
Olivine	NiCa-MgSiO ₄	± 3400	≥ 1400

(a) idealized representation of composition, without impurities present

(b) sintering temperature of pure substances

3. Thermophysical Properties

To assess the particle thermal qualities, the density ρ , the specific heat capacity c_p and the thermal bulk conductivity λ_B are dominant properties.

3.1 Thermal conductivity

The thermal conductivity of the bulk determines the charging and discharging heat transfer characteristics of the HTF. The bulk conductivities of silica, alumina, bauxite and silicon carbide were estimated via the basis Zehner, Bauer and Schlünder (ZBS) model [3,4] and plotted against temperature in Figure 1. The validity of the model for particulate material in a similar size range is confirmed by Baumann and Zunft [5]. The basis ZBS model requires the following parameters for evaluation: the molecular conductivities of the participating phases – air and the candidate particle, and the bulk porosity – set at a value of 0.35.

The bulk conductivity values, as shown in Figure 1 are in good agreement with Baumann and Zunft [5].

SiC and alumina show the highest effective conductivities. Baumann and Zunft reported that the bulk thermal conductivity is only moderately dependent upon the molecular conductivity and increases with decreasing bulk porosities, which can be derived from the ZBS model [5]. Moreover, the effective conductivity increases with coarser particle sizes, where the bulk porosity is lower.

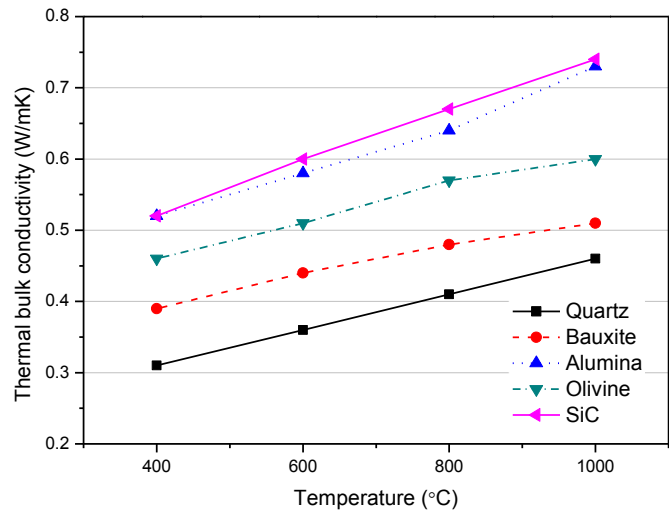


Figure 1: Thermal bulk conductivity estimated with the ZBS-model for different particle materials

3.2 Specific Heat Capacity

For optimal energy storage, a high density and high specific heat are required, resulting in a high volumetric heat density (kJ/m^3).

Additionally, a high C_p value results in increased heat transfer in a moving bed [5]. The specific heat C_p for the candidate particles can be found in VDI Heat Atlas [3] or is given by the manufacturer, and its temperature dependence is illustrated in Figure 2. The heat capacity of cristobalite is almost equal to quartz, apart from the peaks in specific heat due to phase transformations, as confirmed by Findikakis et al. [6]. Differences between values for pure SiO_2 and silica sand and quartz sand are not considered here, however they remain relatively small [5]. The specific heat capacity of graphite (carbon) is added as a basis of comparison: graphite can however not be used in air, and would require the use of an inert fluidization medium and air-free environment throughout the process.

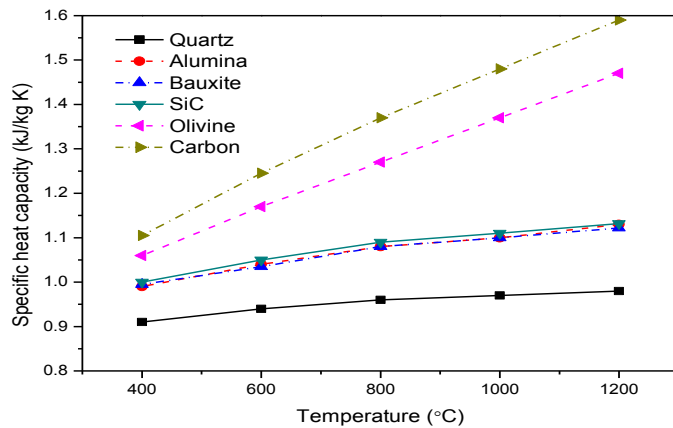


Figure 2: Specific heat capacity of candidate materials (the small peak due to phase transformation for quartz at ~ 573 C is not shown) [3]

4. Thermomechanical Properties

Internal stresses due to thermal shocks could lead to particle breakage and fines formation. Quartz and cristobalite are subject to crystalline inversions, which enhance this effect. The thermal shock resistance of various minerals and ceramics was evaluated by Baumann and Zunft, with an emphasis on high cooling rates [5]. Furthermore, the agglomeration propensity was reported as high in a Silicon Carbide thermal resistance test: SiC is known for its high thermal resistance and commonly employed for high temperature duties. However, few studies have been conducted on the agglomeration behaviour of SiC particles. In terms of the WP1, thermal cycling experiments with SiC and cristobalite were specifically performed and the agglomeration propensity is studied. Olivine is inert to agglomeration.

The density of the particulate feedstock is also important, since the product $\rho_p C_p$ determines the volumetric heat density, and hence the volume of the hot and cold storages. Densities are given in Table 1, and together with the C_p values, favour alumina and olivine.

Experimental study

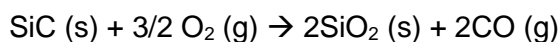
To study the thermal stability of SiC and cristobalite powder, a particle size analysis were performed on a sample undergoing multiple hot (800 °C) – cold (400 °C) cycling. 100 g of SiC and cristobalite powder – with an average particle diameter of 58 μm and 62 μm respectively – were placed in ceramic crucible, heated to 800 °C and then cooled to 400 °C. One complete cycle requires about 2 hours.

After a predefined number of cycles (250, 500, 1000) the samples were removed, allowed to cool and then a sieve analysis was performed using Tyler sieves of 3, 14, 48 and 150 mesh, equivalent to 6680, 1168, 295 and 104 μm , resulting in the cumulative weight fraction passing the different sieve sizes, as shown in Figure 3.

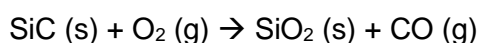
For silicon carbide (SiC), agglomeration occurred, forming large, hard lumps (1 to over 3 cm) with increasing number of cycles. In the cristobalite sample, some small agglomerates were observed. However, these agglomerates could be crushed effortlessly between ones' fingers, disintegrating immediately with the slightest pressure applied.

The agglomeration behaviour of SiC can be explained by the surface oxidation of the particles leading to the formation of low-melting SiO_2 that bridges SiC particles. The purity of the SiC particles plays an important role in the agglomeration behaviour because Si is the main impurity of industrial SiC. To gain insight in the formation of an oxide layer on the particle surface, the thermodynamics of the SiC oxidation were studied.

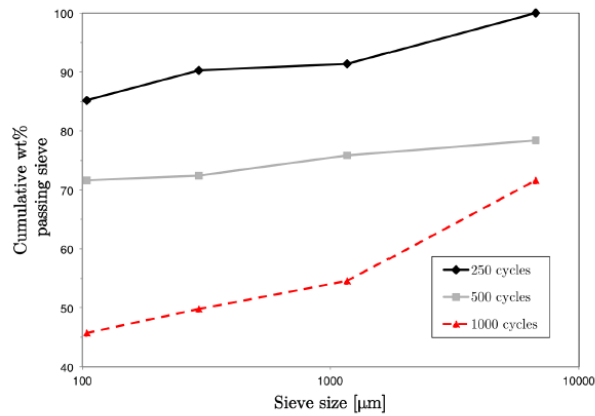
Passive oxidation



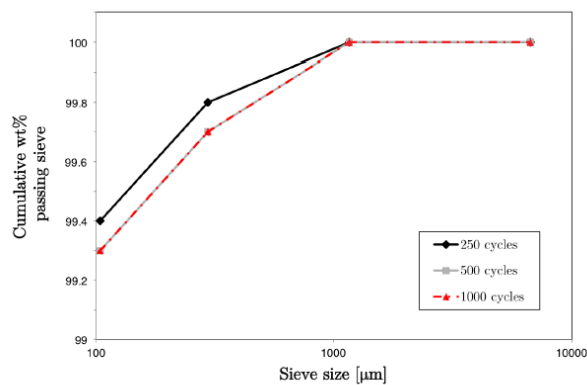
Active oxidation



The reaction is controlled by the diffusion of O_2 through the surface SiO_2 layer, which grows at the Si-SiC interface. SiC contains a limited amount of impurities, mostly Na, Al, K and free-Si. After SiO_2 is formed, low-temperature eutectic melts can occur, even at temperatures as low as 737 °C (as $\text{Na}_2\text{O} \cdot 2\text{SiO}_2$), as reported by Van Caneghem et al. [7]. Cristobalite remains inert up to 1200 °C as specified by its manufacturer, Sibelco-Belgium.



(a)



(b)

Figure 3: Agglomeration of (a) silicon carbide (SiC) and (b) cristobalite particles upon multiple hot (800 °C) - cold (400 °C) cycling. Sieve analysis with 4 different sieves with mesh sizes 104, 295, 1168 and 6680 μm (Initial particle size SiC: 58 μm; cristobalite: 62 μm)

The thermomechanical behaviour of quartz sand (mean particle size of 0.80 mm) was investigated by Baumann and Zunft [5]. Quartz displayed significant granular disintegration after thermal cycling. This effect was enforced by quartz inversion occurring at approximately 573°C.

Cristobalite undergoes a displacive transformation to tetragonal α -cristobalite between 275 and 200°C [8]. This transformation induces thermal expansion, leading to internal stresses inside the particles and an increased amount of fines. The β to α onset temperature varies widely and is assumed dependent on the thermal history of the material [9]. Furthermore, Breneman and Halloran observed hysteresis upon repeated cycling through the β to α cristobalite transformation; an effect which was found particle to be size dependent [8]. To prevent excessive particle breakage and fines generation it is important to suppress the crystalline inversion with a lower working temperature well above 200 °C. Suppressing the β to α transition is possible with chemically stabilized cristobalite [10]. The metastable retention of β - cristobalite was demonstrated for a $\text{Na}_2\text{O}-\text{Al}_2\text{O}_3-\text{SiO}_2$ system [11]. It is expected that the additions are not disadvantageous for the thermophysical properties and the attrition behaviour. This should be confirmed in a further study.

5. Attrition Behaviour

Dust emissions are inherently related to fluidized bed operations, where fines are formed due to particle breakage or attrition in the fluidized bed. In this section a means of comparison between

different particulate materials is targeted. Zhang et al. focused on attrition in powder circulation systems specifically for thermal energy capture and storage [12]. As mentioned by Zhang et al., it is crucial to evaluate the attrition behaviour (i) to limit fines' generation, (ii) to minimize erosion, (iii) to maintain the desired size distribution, (iv) to ensure safety of the workers, (v) to respect the environmental regulation regarding dust emissions, and (vi) to reduce losses of the costly material [12].

Erosion aspects are detailed by Vandewalle and Baeyens [13]; concluding that the erosion impact is limited for superficial gas velocities and associated particle flow below 10 m/s and 200 kg/(m²s) respectively. For the UBFB receiver, both bubble-induced attrition, and attrition related to the nozzle injection (secondary air up the riser) are considered.

Through experimental analysis Zhang et al. introduced a correlation for attrition in fluidized beds, including all relevant operating characteristics, as given in the equation below [12].

The attrition rate is defined as the sum of contributions of bubble-induced and jet-induced effects.

$$R_t = f \left(\left(\begin{matrix} \text{visible} \\ \text{bubble} \\ \text{flow rate} \end{matrix} \right), \left(\begin{matrix} \text{weight to} \\ \text{bed diameter} \\ \text{ratio} \end{matrix} \right) + \left(\begin{matrix} \text{orifice} \\ \text{number} \end{matrix} \right), \left(\begin{matrix} \text{orifice} \\ \text{velocity} \end{matrix} \right), \left(\begin{matrix} \text{orifice} \\ \text{diameter} \end{matrix} \right) \right)$$

Adding the contribution of jet-induced attrition leads to the correlation as presented by Zhang et al. [12].

$$R_t = K_1 \left[\gamma (U - U_{mf}) \frac{W}{D} \right] + K_2 [n_{or} d_{or}^2 U_{or}^2]$$

where W is the bed weight, D the diameter of the bed, n_{or} , d_{or} the number and inside diameter of the orifices respectively and U_{or} the orifice velocity. The effect of the particle nature is included in the intrinsic attrition constants, K_1 and K_2 .

To assess the differences between various particulate materials, Zhang et al. [12] suggested applying the powder-specific Abrasive Index Number (AI), as presented by the Conveying Equipment Manufacturers Association (CEMA), to express the attrition nature of particles [14]. These tentative AI-influences were experimentally confirmed, with K_1 increasing for materials more prone to attrition. Although, a complete fitting of K_1 in terms of AI, was not achieved, Zhang et al. concludes that the AI-value is an adequate tentative indication towards particle attrition [12], with high AI-values indicative for desirable low attrition rates. The AI values along with the CEMA impact factors are given in Table 2. Since the largest contribution of AI is granted to the hardness of the material, this will be the focus for comparison, since AI is an index of abrasion of equipment components by powders and only a secondary means to compare attrition.

In view of generation and emission of fines, it is also important to connect all air exhausts to a high efficiency filter. In view of the temperatures encountered (300 - 850 °C), the use of ceramic or sintered metal filters is proposed, as a result of previous experimental work, reported by Smolders and Baeyens [15] and Schildermans et al. [16].

Towards hardness and equipment erosion, silica sand, quartz, cristobalite and olivine are preferred. All selected powders are however powders are however prone to a limited attrition.

In Solar Power Tower applications, fluidization velocities will be low, typically $\leq 4U_{mf}$. Only low velocity secondary air injection is used. The critical gas velocity (≥ 30 m/s) to provoke fragmentation, as illustrated by Taylor [17], Ghadiri [18] and Geldart and Baeyens [19], will certainly not be reached. Henceforward attrition is mostly due to grinding small chips of large particles. If we assume a typical bed weight of approximately 10 tons for a total of ≥ 7000 tons of powder in the circuit (most of it in the storage hoppers), the attrition rate, in SPT plants, will be $< 0.5 \times 10^{-6}$ kg/s for a bed of 1.5 kg or $< 3.3 \times 10^{-3}$ kg/s for a total bed weight of ≥ 10 ton.

For an annual operation of 7000 h/yr, the total attrition loss will be less than 85 ton/yr, meaning that about 1.2 % of the powders inventory will need to be replaced.

Table 2: CEMA abrasive index of different bed materials [12] Physical Characteristics Value CEMA impact factor

Physical characteristics	Value	CEMA impact factor
Silica sand		
Hardness, HM	6	36
Particle shape	Sub-rounded	1.5
Bulk material density (kg/m ³)	≥ 1300	1.1
Particle size	Group A powder	1.0
Abrasive index AI 68.3		
Quartz sand		
Hardness, HM	6	36
Particle shape	Sub-rounded to rounded	1.0-1.5
Bulk material density	~ 1300	1.1
Particle size	Group A powder	1.0
Abrasive index AI 68.3		
Cristobalite		
Hardness, HM	7	49
Particle shape	Sub-angular	1.5
Bulk material density	1398	1.1
Particle size	Group A powder	1.0
Abrasive index AI 80.9		
Alumina		
Hardness, HM	9	81
Particle shape	Sub-angular	1.5
Bulk material density	~ 1400	1.1
Particle size	Group A powder	1.0
Abrasive index AI 133.7		
Sintered bauxite		
Hardness, HM	9	81
Particle shape	Sub-rounded	1.5
Bulk material density	~ 2000	1.2
Particle size	Group A powder	1.0
Abrasive index AI 145.8		
SiC		
Hardness, HM	9	81
Particle shape	Sharp	2.0
Bulk material density	1900-2000	1.2

Particle size	Group A powder	1.0
Abrasive index AI 194		
Olivine		
Hardness, HM	7	55
Particle shape	Sub-rounded	1.5
Bulk material density	~2000	1.2
Particle size	Group A powder	1.0
Abrasive index AI 99		

6. Health, Safety and Environmental Aspects

When working with powders, exposure occurs via inhalation. Therefore, exposure thresholds often focus on the respirable fraction of the considered particles. The respirable dust fraction corresponds to the proportion of airborne particles, which penetrates into the pulmonary alveolar region of the lungs. Fine particles, present in the bulk, may become airborne during handling and use in fluidized beds, in which case they may become respirable particles. In addition, fines are generated due to attrition and abrasion, as described in section 5, making health and safety hazards of the particle materials largely dependent upon attrition behaviour.

The occupational inhalation exposure limits of the considered particle materials are expressed through the Threshold Limit Value (TLV) and the Permissible Exposure Limit (PEL), set by the American Conference of Governmental Industrial Hygienists (ACGIH) and the Occupational Safety and Health Administration (OSHA) of the United States Department of Labour respectively.

For crystalline silica quartz, the TLV is set at 0.025 mg/m³, the 8-hour time weighed averaged (TWA) PEL for respirable quartz is

$$\text{OSHA PEL respirable quartz} = \frac{10 \text{ mg/m}^3}{\%SiO_2 + 2}$$

$$\text{OSHA PEL total quartz} = \frac{30 \text{ mg/m}^3}{\%SiO_2 + 2}$$

For cristobalite, the occupational standards are more severe, as illustrated in the Safety Data Sheets. Its TLV is equal to quartz. The Occupational Exposure Limit (8-hour TWA) for cristobalite powder is half the value of quartz: 5 mg/m³ divided by "%SiO₂+2" for respirable cristobalite. Long-term or repeated inhalation exposure to crystalline silica (both quartz and cristobalite) may cause fibrosis or "silicosis" and is considered carcinogenic to humans according to the National Institute for Occupational Safety and Health (NIOSH) [20,21]. Furthermore, the ACGIH labelled both quartz and cristobalite as a suspected human carcinogen [22].

Silicon carbide powders are considered without harm to humans, provided concentrations in the human environment are below the OSHA PEL of 15 mg/m³ for SiC. The ACGIH fixed the TLV at 10 mg/m³ for SiC. Only SiC fibres have been linked to an increased cancer incidence [23], however far below the incidence of asbestos fibres.

The TWA TLV and respiratory PEL for alumina and olivine are set at 10 mg/m³ and 5 mg/m³. Neither alumina nor olivine are classifiable as a human carcinogen provided the particulate matter contains

no asbestos and less than 1% crystalline silica. Long-term or repeated exposure via inhalation of alumina aerosol may have effects on the central nervous system [24].

Although high-efficiency filtration will limit the exhaust concentration of the fine dust to $\ll 0.1 \text{ mg/m}^3$ [15,16], occupational hazards should still be considered in the working environment, and the use of air filtration masks is required.

7. Economic Assessment

The minerals like silica sand based particles are favourable over heat-treated minerals and technical ceramics from an economical point of view. Silica sand (30-100 μm) requires the lowest financial investment ($< 50 \text{ €/ton}$). Quartz and cristobalite powder ($\pm 60 \mu\text{m}$) are also cheap (100-200 €/ton) compared to similar size SiC powder (1100-2700 €/ton). Alumina powder is commercially available starting from 300 €/ton . Olivine is quoted at 200 – 250 €/ton .

8. Conclusions

The considered minerals can theoretically withstand temperatures up to and well above 1000 $^\circ\text{C}$. None of the materials fully matches the earlier described desired criteria, so a tentative weighted scoring for each characteristic is applied, giving a more firm ground to base a decision upon. This is presented in Table 3. The weighting is indicated in the Table.

Table 3: Tentative overall comparison of particle properties

Characteristic	Silica sand	Quartz	Cristobalite	Alumina	Sintered bauxite	SiC	Olivine
Specific heat capacity [/10]	6	6	6	6	6	6	8
Thermal bulk conductivity [/10]	5	5	5	7	6-7	8	5-6
Thermal stress [/10]	2-4	4	6-7	8	10	10	10
Agglomeration propensity [/10]	0	4	10	10	10	2	10
Attrition behaviour [/5]	4	4	5	7	8	7	7
Rheological properties [/5]	3	3	3	3	3	3	3
Environmental aspects [/5]	4	4	4	4	4	4	4
Health and safety aspects [/5]	2	1-2	1-2	3	3-4	2	4
Total [/75]	36-38	40-41	46-48	50	52-54	42-43	57-58
Total	48-51	53-55	61-64	67	69-72	56-57	76-78

The scoring is scaled from 0 (worst) to 5 or 10 (best)

Alumina and silicon carbide showed the highest thermal bulk conductivity. No significant difference in specific heat capacity was observed, with a slightly lower value for crystalline silica like materials.

Relating to the thermomechanical behaviour, thermally-induced internal stresses were discussed. Crystalline SiO₂ – silica sand, quartz and cristobalite – suffer from the crystalline inversion, causing thermal expansion. The agglomeration behaviour was experimentally determined for silicon carbide and cristobalite, through multiple hot (800 °C) - cold (400 °C) cycling.

Cristobalite is favoured over silicon carbide, where particle agglomeration occurred upon cycling. The latter is caused by surface oxidation of the particles leading to the formation of low-melting SiO₂ that bridges SiC particles. The purity of the SiC particles plays an important role in the agglomeration behaviour.

Propensity towards agglomeration can be significantly reduced if high-purity SiC powders are used. Cristobalite remained nearly inert up to 1000 cycles. To compare attrition behaviour of the various particulate materials, the CEMA abrasion index of the particles was suggested by Zhang et al. [12]. The CEMA AI considers particle size, particle bulk density, its shape and the Moh hardness, with the highest contribution from the latter. The hardest materials – alumina, sintered bauxite and silicon carbide – show the lowest attrition rates. Cristobalite and quartz are considered carcinogenic substances, and form the greatest hazard of the considered materials. The environmental impact of the various powders is related to particle attrition. To limit the emission of fines, materials with low attrition rates are favourable, and fluidized bed systems should be equipped with adequate dust collection systems in accordance with local environmental regulations. The natural minerals are preferred over technical ceramics from a cost perspective. Cristobalite offers a substantial cost advantage compared to similar sized silicon carbide.

Although, the weighted scoring never rules out subjectivity, significant differences can be observed between the various materials. Olivine has the most favourable characteristics, followed by alumina and cristobalite, making olivine the particulate material of choice for solid/gas suspension HTFs and TES material.

If cristobalite were to be applied, it is important to suppress the crystalline inversion through using chemically stabilized cristobalite or through operating the system above the phase transition temperature at 270 °C.

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