



# Compatibility Testing On A New Ceramic Filler Material For Packed Bed Thermal Energy Storage

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**Abstract.** Thermal storages can contribute to decarbonization in storing energy of fluctuating renewables and waste heat of industrial processes by decoupling supply and demand. A great opportunity to decrease the cost of high temperature storage is given by single tank thermocline storage systems, where large fractions of cost-intensive high-temperature fluids are replaced by a low-cost filler material. Besides low cost, a suitable filler material must meet various other criteria such as suitable thermal conductivity, high heat capacity, high volumetric density of the packed bed of fillers, sufficient mechanical stability and compatibility with high temperature fluids in the temperature range of the application. For one promising type of ceramic filler, the compatibility with a solar salt mixture of 60 wt% sodium nitrate ( $\text{NaNO}_3$ ) and 40 wt% potassium nitrate ( $\text{KNO}_3$ ) was investigated. The filler is composed of additives, such as metallic slag and various other low-cost recycled materials, and a phosphatic binder resistant to the high temperatures. For testing, fillers were immersed in the corresponding fluids in a crucible under air atmosphere. The samples were thermally cycled in an oven up to the maximum foreseen temperature (250–550 °C for the salt). Overall, the ceramics tested show good compatibility with solar salt and have the potential to significantly reduce the cost of the storage systems.

**Keywords:** Thermal energy storage · Compatibility · Packed bed · Filler selection · Molten salt · Thermocline

## 1 Introduction

Different from conventional two-tank molten salt thermal energy storage systems, which represent the state of the art of storing high temperatures up to 560 °C, in thermocline storage systems the energy is not stored in two separate tanks, but in a single tank. The difference in density between the upper temperature level and the lower temperature level of the molten salt is utilized by causing a thermal stratification that separates the tank in two zones [1]. Advantages of this are the elimination of a second tank and the

resulting cost savings, a reduced floor space consumption and a simplification of gas management. The required pump shafts are shorter, which eliminates existing limitations regarding the height of the tanks. The main advantage, however, is the cost saving due to the introduction of cost-effective fillers, which substitute large portions of the more cost-intensive salt. By this investment cost can be reduced by 33% compared to a two-tank system [2].

Proposed filler materials are usually a natural type of rock or industrial waste products. Systematic studies to determine suitable regionally available material with the motivation to decrease cost and carbon emissions have been presented by several authors [3, 4]. Besides low cost, the selected material must meet the criteria of compatibility with the working fluid, resistance to thermal and mechanical stress, high heat capacity and suitable thermal conductivity. As raw materials do not fulfill respective criteria because they are contaminated with other materials, dirt and water they must be processed. The fact that the material has to be crushed, washed, screened and dried adds to the price. The shape of the particles is not optimized to a large surface to volume ratio for increased heat transfer but results from the treatment. Another approach is to manufacture filler particles tailored for a specific application. In this case the most relevant characteristic properties can be adjusted. One example for this is the ceramic filler tested in this study, which is composed of several low cost recycling materials and a phosphatic binder that gives flexibility in the shape and composition of the fillers to suit to requirements of the application.

The filler material tested was characterized to determine relevant properties of the filler and was measured for a brick 210 x 100 x 70 mm build of the material in temperature range of 300–500 °C. A density of ~ 2.00 g/cm<sup>3</sup>, thermal capacity of 1.2–1.28 J/kg K and thermal conductivity of 0.58–0.64 W/m K were found. Cost of filler aimed for is ~ 100 €/t [5] however, the materials used are also subject to the current price increase. In comparison the price for solar salt was quoted at 800–950 \$/t in 2017 [6]. Based on the current price development, this is to be considered as the lower limit.

To test if the fillers are compatible with molten solar salt as the high temperature heat transfer fluid, they were thermally cycled in contact with the fluid. Cycling gives the opportunity to investigate possible degradation of the material due to thermal stress and chemical interaction at the same time. As reference, specimens of filler only and fluid only were cycled in parallel. Afterwards fluid and fillers were analyzed to identify



**Fig. 1.** View into the crucibles after the end of the cycling experiment before sampling for further analysis.

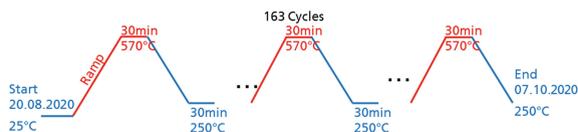
possible changes in the material and to be able to assess whether the material can be used as filler material in storage systems operated with solar salt.

## 2 Experimental Setup

To be able to distinguish between effects of thermal stress or thermal decomposition separately from chemical interaction between filler and salt three different types of probes were prepared. A reference specimen of only fillers, a reference specimen of only salt and the compatibility specimen in which fillers and salt were combined (Fig. 1). Each of these specimens was doubled to check for continuity. Additionally, specimens of the filler material and the salt that were not thermally cycled were kept in their original state. Ortega describes a similar procedure for exposing specimens to molten salt but uses crucibles without welds and with an inlet to eliminate cross interaction with the high-alloy steel [7]. The crucibles used here were manufactured using stainless steel (1.4404), including welding seams and were closed under air atmosphere. Therefore, chemical interaction of salt with its surrounding wall and atmosphere was expected. On the one hand this leads to additional effort and makes the reference specimens necessary to be able to differentiate those to possible interactions with the fillers, but on the other hand helps to identify also possible cross interaction between gas atmosphere, tank material, fillers and salt as they may occur in a realistic scenario of application. The fillers and also the stainless steel parts of the crucibles were previously not heated before the tests as it would be the case when bringing the system to application, when heating large scale tanks is not possible and previous treatment of fillers would lead to undesired increase in cost of fillers. For pressurized systems nevertheless this might be necessary for safety reasons and in order to get rid of components fluctuating fast at the planned temperatures as inherent water.

### 2.1 Thermal Cycling Procedure

In total six crucibles were placed in the oven before the experiment and removed after the 163 cycles. A visualization of the cycling program run is given in Fig. 2. The test was started at ambient temperature. The ramps were run with maximal heating/cooling rate of the oven, which lead to duration of temperature ramps of  $\sim 180$  min. at the end of each ramp the maximum/minimum oven temperature was kept constant for 30 min. The maximum oven temperature was set to  $570^\circ\text{C}$  because a previous test with a thermocouple placed in a crucible showed that the maximum temperatures measured inside of it were always at least 20 K lower than the set oven temperature during this period of time. So the maximum temperature of exposure for salt and fillers was  $550^\circ\text{C}$ .



**Fig. 2.** Duration and cyclic temperature control (oven temperature) of the ageing tests of filler material in contact with solar salt.

**Table 1.** Mass balance of solar salt and filler after sampling. (A) and (B) indicate results of the two specimens from the same cycling test

Mass [g]	Filler only reference		Fluid only reference		Fluid + Fillers	
	A	B	A	B	A	B
$\Delta m_{\text{tot,av}}$	-1.3	-1.3	-1.9	-2.3	-3.5	-5.3
$m_{\text{fillers,start}}$	105.2	99.8	—	—	94.9	98.3
$\Delta m_{\text{fillers}}$	-0.9	-0.7	—	—	+ 29.0	+ 28.5
$m_{\text{salt,start}}$	—	—	200.3	200.5	199.7	199.9
$\Delta m_{\text{salt}}$	—	—	-2.3	-3.6	-33.0	-35.2
$\Delta m_{\text{salt}} + \Delta m_{\text{filler}}$	—	—	—	—	-4.0	-6.7

## 2.2 Sampling Procedure and Mass Balance

To be able to remove the fillers from the salt, sampling was done when the salt was still liquid. As a consequence liquid salt was attached to the surface of the fillers after sampling. In order to be able to calculate a mass balance for salt and fillers the salt was separated from the filler. This was done by placing the fillers again in the oven at moderate temperatures of  $\sim 300$  °C on an absorbent isolation layer of Insulfrax for several hours. The insulation was able to absorb the again liquified salt on the surface of the fillers (see Fig. 3). Fillers were removed from the insulation at high temperature to avoid any fibers of silicate wool sticking to them together with solidified salt. A detailed mass balance to identify changes in mass of fillers or salt is given in Table 1 considering changes of mass of all parts of the crucibles, tools used for sampling and the insulation layer that absorbed the attached salt of the fillers. Parameters of the used scale: 10000 g max.,  $d = 0.05$  g.

The total change of mass was calculated twice, once comparing the mass loss during the cycling for the sealed and once for the opened crucible. Given here is the average value of both  $\Delta m_{\text{tot,av}}$ . For all specimens a certain mass loss can be observed during the thermal cycling. Apparently, the crucibles were not absolutely leakproof. This was confirmed during subsequent tests regarding the gas tightness of the mica gasket.

The mass loss of the fillers only reference specimens ( $\Delta m_{\text{fillers}}$ ) is in the same range as the associated total mass loss and is reasonable as it could be reproduced in a testing of drying the fillers in an oven at 350 °C of (-4.2% for the cubes and -0.6% for the spheres) [5]. It could be concluded that the original presumably dry fillers still included water or other volatile substances that fluctuate at lower temperatures.

Also the mass loss of the fluid only reference ( $\Delta m_{\text{salt}}$ ) is in the same range as the total mass loss. The used mica gaskets are best suited in combination with molten salt. However, this does not mean absolute tightness over a period of  $> 1000$  h. A thin layer of yellowish deposits observed after the end of the experiment on the inner wall of the oven and on the outside of the crucibles makes the mass loss plausible and fits to the detected minor salt leakage.



**Fig. 3.** Optical modification of fillers cycled with solar salt, a) before in contact with salt, b) after sampling, c) after separation of salt in oven

For the actual compatibility specimens of Filler + Fluid, an increase of mass of fillers and decrease of mass of salt could be observed. In the overall balance ( $\Delta m_{\text{tot,av}}$ ), however, the magnitude of the mass change is in agreement with the mass change of the fluid added to the one of the fillers. The enlarged mass decrease in this case can be explained by the superposition of both effects described above (mass loss of filler + salt loss).

The enlarged increase in the mass of the fillers and, to the same extent, decrease in the mass of the salt compared to the reference specimens suggest that salt remains in the pore space of the fillers despite the separation of the salt adhesion by tempering in the oven on absorbent insulating material, which is considered in the mass balance of the salt. This conclusion is particularly plausible since the fillers have been shown to have an inter porous void fraction, which could be verified by observed gas leakage in a previous test in which the fillers were placed in fluid.

For the sake of completeness, it should be mentioned here that the sample arrangement always consisted of spherical and cubic fillers, which were, however, identical in their material composition. The reason for this was that an increased gain in knowledge was expected to be derived from this in the mechanical stability tests that followed the compatibility exposure tests. The classical crush strength tests carried out at room temperature, however, showed a four times higher mechanical stability for the fillers which were precipitated in salt. This is mainly due to the salt deposited in the pore space of the fillers, which apparently strengthens the filler structure at this temperature in the hardened state. So, these tests were not conclusive. Conclusive tests would have to be repeated at temperatures at which the salt is molten.

**Table 2.** Ion concentration of the solar salt in mg/kg. “control” is uncycled, (A) and (B) indicate analysis results of doubled samples from the same cycling test.

Ion	Control sample	Fluid only reference		Fluid + Fillers	
		A	B	A	B
Al	13.9	14.4	16.3	29.7	27.7
Ca	6	27.3	11.6	61	50.7
Cr total	< 1	17.1	15.1	254.3	208.1
Cr (VI)	< 0.01	17.10	15.10	226.60	197.10
Cu	14.3	10.6	9.3	10.3	9.5
Fe	< 1	< 1	< 1	< 1	2.2
K	113000	91840	196000	100880	137740
Mg	7.8	5.4	5	5.5	6.5
Mn	< 1	< 1	< 1	< 1	< 1
Na	137000	121540	242400	127460	170900
Ni	1.6	2.1	1.8	1.6	< 1
P	1.9	< 1	< 1	6.7	< 1
S	21.7	21	21	436.6	356.6
Ti	1.9	1.8	1.8	2.1	2.1
NO <sub>2</sub>	< 10	9200	6000	10200	10600
NO <sub>3</sub>	564000	670000	640000	684000	682000

### 3 Analysis of Salt

In general, an increase in the concentrations of individual ions compared with the reference sample can be caused by two different reasons.

- Reason No.1 would be that the corresponding elements are actually contained in the filler material and are present in solvable form, so that they pass into the salt over time.
- Reason No.2 might be that the corresponding elements are not present in the filler material, but in the alloyed stainless steel (1.4404) of the crucibles and the welds therein, and that the redox chemistry is accelerated by the fillers. As a consequence the salt decomposes more quickly and the redox reactions with the crucible are accelerated. The porosity of the fillers and the increased corresponding surface area are reinforcing this effect.

The subsequent chemical analysis given in Table 2 was carried out using inductively coupled plasma mass spectrometry (ICP-MS). The concentration of iron, copper, magnesium, manganese, nickel and titanium is constant in all cases.

For aluminum, calcium and chromium ions, there is an increase in concentration in the reference samples, which is also evident in the samples that were in contact with the

fillers, where it is even higher. Aluminum and calcium are present in the base mixture of the fillers in relevant amounts, thus allowing a conclusion to be drawn for reason No.1. Their increase is not considered critical for compatibility. The content of chromium in the base compound of the fillers is  $< 0.2$  wt%; related to 100 g of filler material, the maximum soluble amount of chromium is thus  $< 0.2$  g. In the salt, a concentration averaging 225 mg/kg is found. For 200 g salt, this corresponds mathematically to 0.045 g chromium. So the increase here could also be due to reason No.1. Since the high-alloy steel has a chromium content of around 16.5% by weight, however, reason No.2 is more likely.

In general, the increase in the ion concentration of chromium is to be considered critical because the oxidation state chromium (VI) has multiple properties of concern. Chromium (VI) oxide is the only stable form in salt. Therefore, it was recorded separately. In fact, analysis shows that the chromium is almost completely present as chromium (VI). An indication of an increased concentration of chromium (VI) in the salt is also the yellowish coloration, whereby a slight yellow tint is also caused to the formation of nitrite. However, the concentration of chromium (VI) is below the considered limit values. After 1128 h, max. 17 and 250 mg/kg were determined without and with fillers, respectively. For comparison, the limit value for the classification of waste requiring monitoring is for chromium (VI) on the one hand:- according to AVV (European Waste Catalogue Ordinance) for substances of group H7 (carcinogenic according to category 1 or 2) individually 1000 mg/kg OS (original substance) - on the other hand according to VwV (General Administrative Regulation for AVV) for substances of group H14 (environmentally hazardous according to R50/53) in total (here relevant Cr + Ni) 2500 mg/kg OS [8]. Therefore, the concentration is not rated as critical.

The phosphate analysis showed no to very little increase in phosphate ions in the solar salt. The phosphate binder is an elementary component of the filler material mixture. According to the data sheet, the stainless steel of the crucibles contains max. 0.045 wt.% phosphate. The increase is therefore assumed to be due to reason No.1. An increase in concentration to a significant degree would have been considered critical. Thus, it can be assumed that the filler would remain stable over a longer period of time. The increased ion concentration of sulfur is also more likely to be caused due to reason No.1, since according to the data sheet max. 0.03 wt.% sulfur is contained in the steel of the crucibles and no increase was observed in the reference sample. For a more detailed discussion the elemental analysis of the fillers must be considered. However, the detectable concentrations of sulfur in the compatibility specimens are considered harmless for the intended technical process since severe precipitation reactions are not to be expected.

Sodium and potassium, just like phosphate, are glass formers, so a decrease of the ion concentration in the solar salt was expected in combination with the siliceous packing. The smaller sodium atom was considered to be more reactive than the larger potassium atom. However, the analysis does not show a clear trend in this direction. No clear trend can be identified for neither the compatibility specimens nor the fluid only reference specimens. The duplicated specimens increased in concentration in one case and decreased in the other.

The stability of solar salt is directly related to the stability of nitrate, which is known to dissociate into nitrite and oxygen according to Eq. (1). The resulting balance depends on pressure and temperature. It is known that dinitrogen, nitrous oxides or oxygen species can also form from the nitrite. The oxygen species can lead to increased corrosion of the stainless steel tank walls. [9]



A separate overview of the nitrite/nitrate concentration of the samples is given in Table 3. In the original salt sample nitrite was not present. However, nitrite formation occurred in all four samples. For the pressure vessels and maximum temperatures of the aging tests, the nitrite/nitrate equilibrium was expected to be in the range of ~ 98 mol % nitrate ions/ ~ 2 mol % nitrite ions according to [7, 9]. The calculated ratio agrees with this and is in the same range for the reference samples as well as for the salt that was in contact with the fillers. Thus, no significant difference in comparison to the Fluid only references can be seen and the fillers can be excluded as a driving factor for nitrite formation.

## 4 Analysis of Fillers

The subsequent chemical analysis given in Table 4 was carried out using inductively coupled plasma optical emission spectrometry (ICP-OES) for the fillers. The significant increase in concentration of potassium and sodium ions, as well as nitrite and nitrate in samples A and B, confirms the inclusion of salt in the pores of the fillers identified from the mass balance. As a consequence, their share was subtracted from the analyses under the assumption that the total increase is caused by inclusions only. By this correction the share of the other ions is increased (Fluid + Fillers corr.).

In comparison with the control sample the concentration of copper and nickel remained constant during the tests. The sulfur values decreased slightly. Thus, the detected slight increase in sulfate in the salt can be attributed to the interaction with the fillers. However, this is not considered critical. For chromium and titanium contradictory values were determined in specimen A compared to specimen B for the corrected values. Although the external conditions were identical once an increase of concentration and in the other case a decrease compared to the control sample is observed. The resulting different concentrations might be caused by not fully homogeneous recycling raw materials used for the production of the fillers. For the different powder materials, the compositions vary considerably. A Gaussian particle size distribution is given for each powder. It has been shown that different grain sizes exhibit strong differences regarding the contained ions.

For example, the concentration of silicon in the 1.2 mm grain size is three times higher than in the 0.25–0.5 mm grain size of the same sample of raw material. The same applies to the iron and the manganese content, as shown by the increase for the corrected values. Since the fillers are small compared to the grain size of the raw materials difference might be caused by a few grains more or less of the bigger particles contained in the respective filler. The deviation found is therefore within the expected range.

**Table 3.** Ratio between nitrite and nitrate mass in the solar salt samples.

Sample	$[NO_2^-]/[NO_3^-]$
Control	0
Fluid only reference (A)	1.37%
Fluid only reference (B)	0.94%
Fluid + Fillers (A)	1.49%
Fluid + Fillers (B)	1.55%

**Table 4.** Elemental composition of the filler samples after the exposure in solar salt in mg/kg. "control" is uncycled, (A) and (B) indicate analysis results of doubled samples from the same cycling test.

Ion	Control sample	Fluid + Fillers		Fluid + Fillers corr.	
		A	B	A	B
Al	93200	55300	64900	70049	81784
Ca	126500	97100	96700	122997	121857
Cr	600	300	800	380	1008
Cu	< 100	< 100	< 100		
Fe	50300	42900	57600	54342	72585
K	1600	29300	31200		
Mg	8300	6400	7300	8107	9199
Mn	7950	7000	9000	8867	11341
Na	26150	55600	57500		
Ni	< 100	< 100	< 100		
P	34300	26000	26900	32934	33898
S	3250	2100	2100	2660	2646
Ti	3650	2200	2900	2787	3654
NO <sub>2</sub>	< 100	1400	1500		
NO <sub>3</sub>	< 100	152000	144000		

Significant losses in concentration were found for aluminum and calcium ions. Since both ions, bound as metallic oxides, are present in the raw materials in relevant quantities, the result correlates with the increase of concentration in the salt identified in the chemical analysis of the solar salt. The analysis also showed a slight decrease in phosphate ions in the fillers exposed to the salt. This was expected to a certain extent, since production requires a slight excess of phosphate binder, leaving a surplus of unbound binder which could then be transferred to the molten salt. In the corresponding analysis of the salt however only one of the two specimens showed an increase in phosphate ion

concentration and the decrease in fillers is relatively low, especially if the corrected value is considered. On the basis of the investigations carried out, no clear statement can be made as to whether parts of the phosphate are dissolved exclusively in the salt or also might interact with the stainless steel crucible.

## 5 Conclusion and Outlook

A new ceramic filler material was exposed to a mixture of solar salt and both, salt and filler where afterwards analysed to evaluate the compatibility of both. Based on a mass balance for the fluid and solid corresponding to the tests, an internal porosity of the fillers was identified. It gives potential for further optimization regarding volumetric density of the packed bed. The decomposition of the solar salt in nitrite is not driven by the fillers but thermally. For the compatibility tests discussed in the present paper the chemical interaction between solar salt and filler is within acceptable limits. In a follow up step towards application the fillers are tested in a prototype setup with a storage volume of  $\sim 0.9 \text{ m}^3$ . It is expected that the interaction of salt and steel wall of the tank encouraged by the fillers does not occur to the same extent, since the surface-to-volume ratio is significantly more favourable in this case. Tests will be run with lower maximum temperatures. By this the overall reactivity is decreasing, thus the concentration of chromium in the salt is inhibited. Additional samples will be analysed also from this setup to monitor the ion concentration in the salt. Based on the monitoring, measures can be recommended for the application, such as appropriate coatings for the steel or the selection of an alternative steel. Although additional specimens that are sampled at previous timesteps would have been helpful for better understanding if the occurred decomposition is unlimited or if the chemical interaction is limited to a certain concentration and stops at a certain point, the suitability of the material is not questioned by the results presented.

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