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Summary

The deliverable deals with the development of the latent thermal energy storage for the geothermal power plant Kizildere-2, Turkey. The storage is meant to increase the flexibility of the power plant by storing heat from the brine after the steam separation. Latent thermal energy storages use phase change materials (PCM) to store heat as latent heat at an almost constant temperature. Therefore, the storage system is called a PCM module hereafter. The storage will be charged and discharged by an immersed heat exchanger within the PCM. The main objective of this deliverable is the selection of suitable PCMs for the storage as well as the selection of proper materials for the heat exchanger and storage container.

For the PCM selection multiple materials were selected based on a literature review. The materials were experimentally characterized to identify their melting temperature, phase change enthalpy, cycle stability and hysteresis between melting and crystallization of the PCM. The organic adipic acid and the inorganic salt mixture HITEC were identified as most promising materials. As organic materials suffer from thermal degradation an ageing test was performed for adipic acid.

A finned-tube heat exchanger will be designed for the PCM Module. As tube materials carbon steel and stainless steel were considered. Additionally, aluminium is possible for the fins. To identify proper materials for the heat exchanger corrosion tests were performed for both PCMs.

As adipic acid degrades and leads to a higher corrosion rate for the preferable low cost carbon steel, HITEC was selected as PCM material. For the salt mixture, the material selection tests led to the conclusion that the heat exchanger could be made of carbon steel with aluminium fins or stainless steel with aluminium fins.

Objectives Met

With this deliverable, the following work package objective was achieved:

- Selection of the most appropriate phase change storage media for each of the case study examples.
- Selection of the most appropriate metallic alloys in contact with the PCMs.

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1. INTRODUCTION

Within the GeoSmart project thermal energy storage systems are developed and designed for geothermal power plants Insheim, Germany and Kizildere-2, Turkey. For Insheim a water thermocline storage is considered. For Kizildere-2 a steam storage as well as a latent thermal energy storage are planned. As water thermocline and steam storages are more common technologies this deliverable contributes to the development of the PCM module.

On the Kizildere-2 site belonging to Zorlu, the steam is separated from the liquid brine on different pressure levels by flashing and sent to a high, medium and low pressure turbine for electricity production. One of the objectives of the project is to store a part of the steam and the heat of the brine after the first separation to adapt the production of electricity to the demand. The steam will be stored in a steam accumulator.

For storing the brine heat, a latent thermal energy storage will be developed. These latent heat storage systems use phase change materials (PCM) to store heat at an almost constant temperature level through a phase transition. For this reason, the storage is called a PCM module in the rest of the document. When the demand is low, steam and heat are stored. When the demand is high, steam is released from the steam accumulator and sent to the low-pressure turbine. The PCM module is used to heat up the brine coming from the outlet of the low pressure separator. After the PCM module, steam is generated by flashing for the low-pressure turbine. The PCM module will operate between 107 and 165°C.

The objective of this deliverable is the selection of possible PCMs that could be suitable for the Kizildere plant, and the selection of metallic alloys that are compatible with the PCM. The selection follows several steps:

- A list of suitable PCM products are established from a literature review.
- Suitable PCM products are selected according to various criteria such as physical properties, non-hazardous products and cost.
- The PCM materials are tested at lab scale, to confirm or understand their physical properties and their stability under thermal cycling.
- The chemical compatibility of the PCM materials with the other materials of the module (for instance metallic alloys) are tested at lab scale, the main objective is to assess the interaction between the PCM materials and other constituents.

2. BEHAVIOUR OF A PCM MODULE

The PCM module is similar to a vertical single pass shell-and-tube heat exchanger with no tubulars on the shell side (Figure 1).

Its constituents are:

- Two top and bottom collectors, with distribution plates.
- The shell filled of PCM.
- The finned tubes, the fins enhancing the conductive heat transfer to the PCM side, this is actually the more standard and industrial way to achieve it, even if there are many other methods - foams, composites, conductive particles in the PCM etc.
- Inserts in the tubes on the brine side, enhancing the convective heat transfer.
- A gas sky to accommodate the volume change consecutive to the phase change of the PCM. This gas sky can be open to the atmosphere, tight with dry air or tight with an inert gas such as Nitrogen, depending on the PCM compatibility and stability with oxygen and moisture.

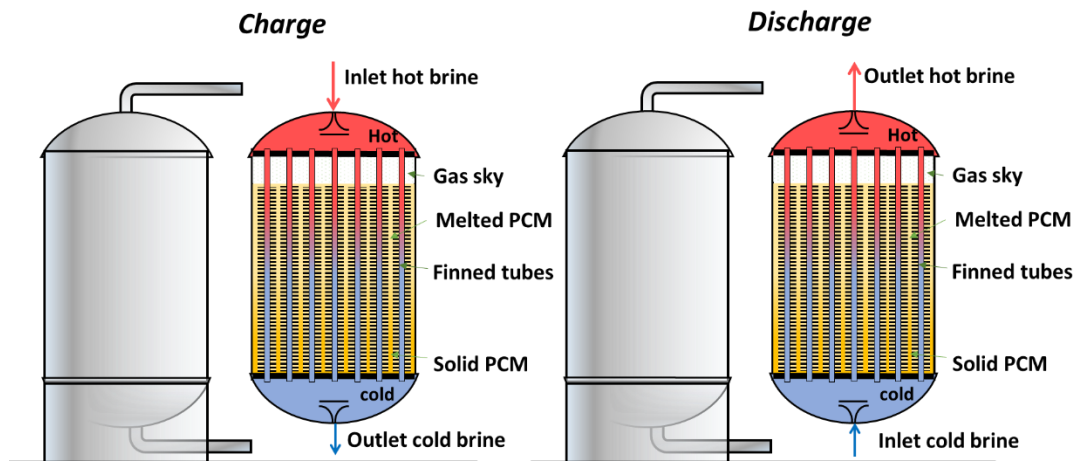


Figure 1. PCM heat storage operation principle

Thermally, the PCM storage functions as follows:

- During the charge, the “heat transfer hot fluid” (whose temperature is higher than the PCM melting temperature) enters by the top collector and flows inside the finned tubes, transfers its heat to the PCM, and goes out colder by the bottom collector. During this phase, the solid PCM melts and becomes liquid. The melting front is complex because the PCM melts faster between the fins than in the free PCM.
- During the discharge, the “heat transfer cold fluid” (whose temperature is lower than the PCM freezing temperature) enters by the bottom collector and flows inside the finned tubes, recovers the heat from the PCM, and goes out hotter by the top collector. The temperature of the heat transfer fluid at the output during the discharge is close to the melting temperature of the PCM. During this phase, the PCM changes its state from liquid to solid.

3. PCM PRE-SELECTION

Based on a literature review, several PCMs were identified. The main criterion for selection was a melting temperature within the operation temperature range from 107 to 165 °C. It was decided to choose a material with a melting temperature below 160 °C to store the heat near the maximum temperature, but also to ensure a sufficient temperature difference for the heat transfer. Also, the material should have less subcooling, this means the material should crystallize at a temperature close to the melting temperature. Regarding the stability a high cycle stability and low degradation rate is needed. According to the KPIs defined in deliverable 1.8 a maximum Reduction Rate of Melting Enthalpy (RRME) of 10% per year is permissible for the demonstration. Furthermore, the material should be compatible to the heat exchanger and vessel materials. The identified materials are listed in Table 1. The comments on the materials are based on the information in the literature and are verified by material tests (see section 4).

Table 1. Identified materials with properties from literature sorted by melting temperature

Material	Melting Temperature °C	Melting Enthalpy kJ/kg	Density kg/m ³	Comment	Source
Benzanilide	161	129-162	-	High melting temperature	[1-4]
Salicylic acid	157-161.1	199	1440-1443	High melting temperature, low cycle stability	[1-3,5]
LiNO ₃ -NaNO ₃ -KCl 45-50-5	160.0	266	2297	High melting temperature, hygroscopic, corrosion possible	[6]
Mannitol-Dulcitol 70-30	152.0	260	1300	Supercooling	[7]
Adipic acid	150-155	213-260	1360-1370	Low pH, corrosion possible	[1,4,6,8]
KNO ₃ -Ca(NO ₃) ₂ 67-33	150.0	72.0	2000	Hygroscopic	
KNO ₃ -NaNO ₃ -NaNO ₂ 53-7-40 (HITEC) or 53-6-41	142	80-110	1899-2006	Common as heat transfer fluid, corrosion possible, hygroscopic	[3,4,6,9-11]
LiNO ₃ -NaNO ₂ 62-38	156	233	2296	Very hygroscopic, expensive	[6]
Sebacic acid	130 - 135	228	1270	Degradation possible, low cycle stability	[1,4,9]

4. PCM MATERIALS TESTS

4.1 Material Characterization

The identified materials listed in Table 1 are characterized by differential scanning calorimetry (DSC) to identify the melting and crystallization temperature as well as the melting enthalpy. Further, the materials were thermally cycled in the DSC to prove the cycle stability. Organic compounds were cycled in air while inorganic mixtures, due to their hygroscopic nature, were cycled under nitrogen atmosphere. Different DSC's are used as some materials tend to supercooling for smaller sample mass. TA Instruments Discovery Q2500 and Q200 DSC as well as a Setaram Instrumentation SensysEvo DSC were used. The latter allows a sample mass about five times larger. Materials were cycled between 50 and 100 times.

4.2 Aging Test

From the identified PCMs, adipic acid and HITEC are selected as the most promising PCMs. The selection was made based on the relevant material properties for a PCM module, namely melting temperature, melting enthalpy, cycle stability and supercooling (see section 5.1). HITEC is already used in industrial applications as a heat transfer fluid for temperatures up to 620°C [11]. Thus, the stability of the material is not further investigated. Adipic acid is mentioned in literature as a potential PCM but is not investigated in detail. In particular, the degradation of the material must be investigated since a slight reduction of the melting enthalpy is observed during thermal cycling.

For organic PCMs like Erythritol and D-Mannitol, an oxygen based degradation mechanism is proposed in literature if the materials are kept in their liquid state [12,13]. Therefore, it is suspected that adipic acid will also degrade. An ageing test was performed under air and inert atmosphere; for the latter argon is used. For each atmosphere 18 samples of 80g were prepared in closed bottles (see Figure 2). The bottles are stored in an oven in their liquid state, at the maximum operation temperature of 165°C for up to 1000 hours.



Figure 2. Adipic acid samples for ageing test

After each of the six time intervals three bottles were taken from the oven. Three samples were taken from each of two bottles and characterized by DSC to identify the melting and crystallization temperature as well as the melting enthalpy.

Attempts were made to analyse the degradation mechanism using Raman spectroscopy and attenuated total reflectance (ATR). The measurements were performed for samples from the different time intervals. Both methods are complementary and can provide information on the molecular structure and functional groups of adipic acid.

4.3 Material Compatibility

4.3.1 Alloy selection

A material selection analysis was conducted to identify the optimum materials combinations to be used in the PCM heat exchanger. Based on the options available and offered by the different heat exchanger manufacturers, a few materials were selected and tested in combination with the two PCMs selected arising from the initial literature review and aging tests, namely adipic acid and HITEC salt.

Three different alloys were initially tested to assess their corrosion performance when in contact with adipic acid: SA 179, 316L stainless steel and Al1050. In the case of HITEC salt Al1050 and SA 179 were tested.

Table 2. Tests conducted for PCM/metals compatibility

PCM	Tested alloy
Adipic acid	SA 179
	316L
	Al1050
HITEC salt	SA 179
	Al1050

Carbon steel SA 179 is a structural alloy widely used in the manufacture of boilers, storage tanks and pressure vessels as well as heat exchangers. Suitable at low and moderate temperature, this alloy is an affordable material with a relatively high thermal conductivity compared with other Fe-rich alloys.

316L is an austenitic stainless steel, with low carbon content (<0.03%wt), high corrosion resistance and low stress corrosion cracking susceptibility. Therefore, it is a suitable material for heat exchangers, it is however a more expensive alloy with a lower thermal conductivity than carbon steel.

Al 1050 (99.5% Al), was identified as the most suitable material for the fins of the heat exchanger due to its high thermal conductivity, it was the third material tested for compatibility with both selected PCMs.

4.3.2 Test setup

4.3.2.1 Tests – adipic acid

Tests to validate the compatibility of adipic acid with the potential alloys to use for the heat exchanger were conducted. 2L flat flange beakers were used, with 4-neck lids, surrounded by heating jackets for a controlled temperature (see Figure 3). Type K thermocouples were used to both control the temperature of the jackets and the temperature inside the vessels.

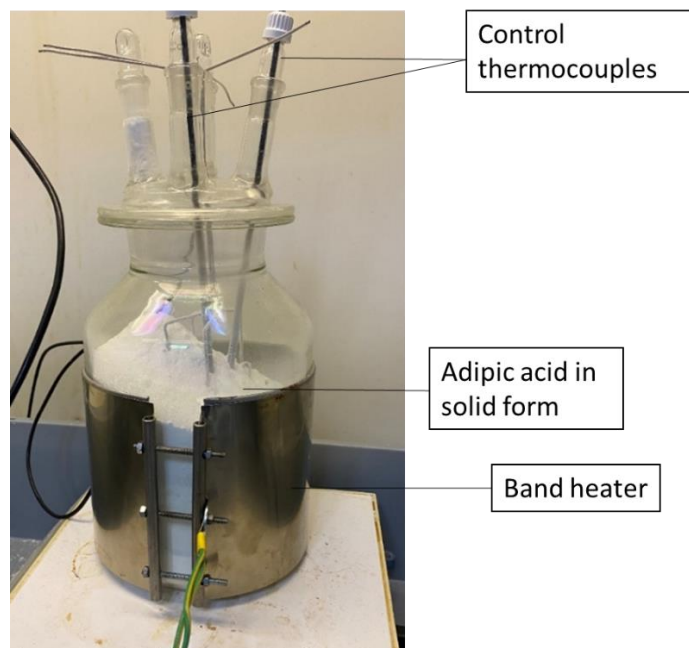


Figure 3. 2L glass vessels containing adipic acid and samples prior to testing. The vessel is wrapped in a heating jacket, and two thermocouples are located inside the vessel for temperature control

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An appropriate volume of adipic acid was initially inserted in the vessels. Metallic samples due to be tested in the vessels were photographed and weighed prior to testing. These samples were placed in glass baskets and inserted in the test vessel. The temperature of the vessel was raised to 165°C at a controlled rate of 50K/hour to avoid an overshoot of the temperature due to latent heat. The temperature was maintained at 165°C for the duration of the tests.

In order to facilitate the removal of the samples, the glass baskets were lifted from the molten material by the means of a metallic wire through the vessel lid. Once the material solidified, the vessel was opened and the samples were collected to be then weighed, photographed and characterised.

4.3.2.2 Tests – HITEC salt

Tests with HITEC salt were conducted using a setup similar to the adipic acid tests. In this case, two tests were conducted in parallel, where 2L/min of compressed air were constantly sent to the vessel for moisture sweeping (see Figure 4). Two control thermocouples inside the vessel were used to both record the temperature profile and control the potential overheating of the system.

The use of HITEC salt [14,15] as a PCM showed that common oxidants in molten salts such as O₂, H₂O and H⁺ can accelerate corrosion processes and accelerate the degradation of metals.

It was shown that a heat treatment of the PCM prior to corrosion testing could result in an improvement in corrosion resistance of the exposed steel. To conduct a similar process and in an attempt to reduce the moisture content in the salts, the temperature was maintained at 120°C for 4 hours before being raised to 165°C to melt the salts.

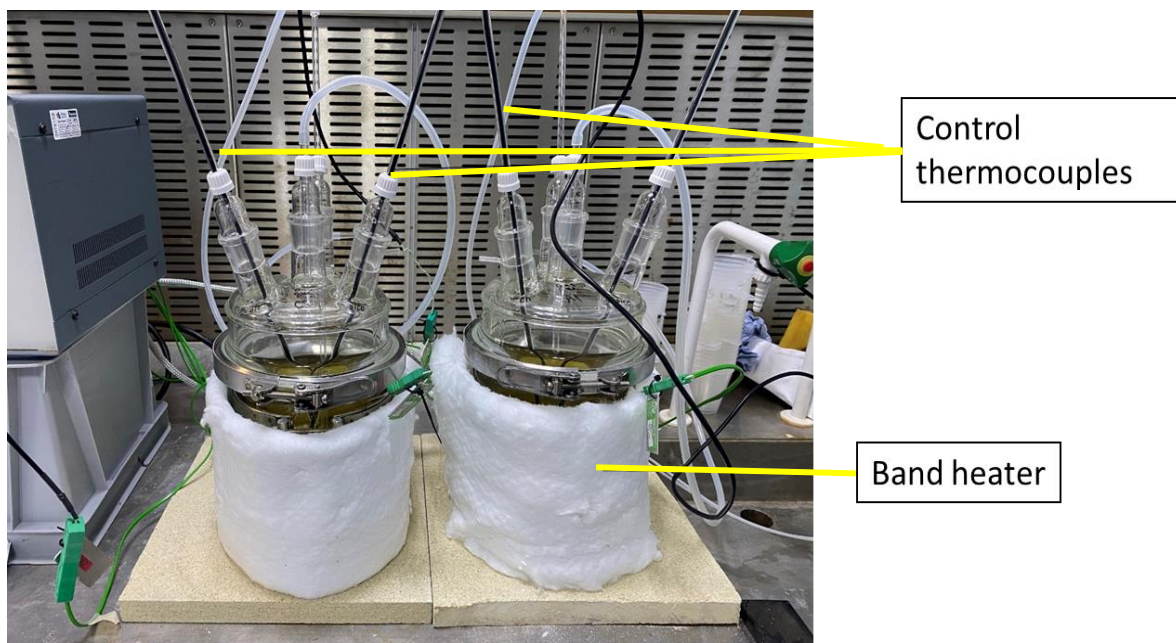


Figure 4. Test setup for HITEC salt compatibility experiments including compressed air connections, band heaters and temperature controllers

4.3.2.3 Sample characterisation

All the samples were photographed, measured and weighed prior to testing in order to have full control of the behaviour of the tested alloys. At the end of each test, samples were rinsed with deionised water and IMS in order to remove the excess of acid/salts on the sample surface. Subsequently, two out of four samples were cleaned following ASTM G1 [16].

In between each cleaning cycle, samples were weighed to control the weight loss/gain and each value was recorded.

4.3.2.4 Corrosion rate measurements

Corrosion rates were estimated from the mass loss measured after exposure of the PCM in controlled atmosphere and at controlled temperature. The following equation was used to determine the corrosion rates.

$$CR = \frac{(K \times W)}{A \times T \times D}$$

T is the time of exposure in hours

A is the area in cm²

W is the mass loss in grams following the cleaning cycles

D is the density in g/cm³

K is a constant

5. RESULTS AND DISCUSSION

5.1 Material Characterization

The results of the material characterization are described qualitatively in Table 3. According to the table, only adipic acid, HITEC and LiNO₃-NaNO₂ provide suitable properties. As the melting temperature of the LiNO₃-NaNO₂ is lower compared to HITEC it will not be considered. Finally, the organic material adipic acid and the inorganic salt mixture HITEC were identified as possible materials for the PCM module.

Table 3. Qualitatively characterization results, very good ++, good +, neutral o, bad -

Material	Melting Temperature	Melting Enthalpy	Cycle Stability	Supercooling	Suitable
Benzanilide	-	+	++	+	no
Salicylic acid	-	+	-	-	no
LiNO ₃ -NaNO ₃ -KCl 45-50-5	-	+	++	+	no
Mannitol-Dulcitol 70-30	++	++	-	-	no
Adipic acid	++	++	+	++	yes
KNO ₃ -Ca(NO ₃) ₂ 67-33	not rateable	not rateable	not rateable	not rateable	no
KNO ₃ -NaNO ₃ -NaNO ₂ 53-7-40 (HITEC)	+	+	++	++	yes
LiNO ₃ -NaNO ₂ 62-38	+	+	++	++	yes
Sebacic acid	+	+	-	+	no

5.2 Ageing test

For both atmospheres a similarly pronounced degradation of adipic acid occurs. The reduction of the melting enthalpy compared to the unaged material is shown in Figure 5. The melting enthalpy is reduced but tends to stabilize at 1000h at around 10% reduction. In deliverable 1.8 the Reduction Rate of Melting Enthalpy (RRME) is defined as a KPI. A maximum reduction of 10% per year is acceptable. For the application at the Kızıldere-2 power plant the PCM is assumed to be liquid 10 h per day for 360 days per year. If the degradation rate stabilizes after 1000h a RRME of 10% will be reached after 100 days. Beyond 100 days the RRME is expected to be lower.

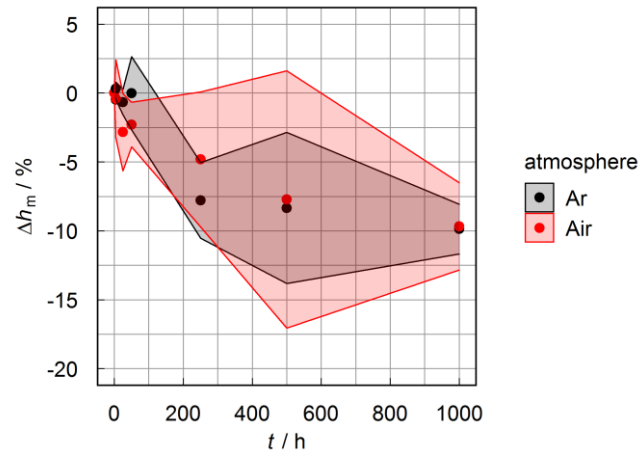


Figure 5. Reduction of melting enthalpy of adipic acid for argon and air atmosphere

The melting and crystallization temperature slightly shifts to lower temperatures. Additionally, the colour of the samples changed to dark brown (see Figure 6). The resulting spectra of the ATR and Raman spectroscopy do not show any significant change between unaged and aged samples. As degradation is detected by DSC measurements, it is assumed that either the structural changes lead to similar structures compared to the original molecular or the changes are minor and superimposed. Thus, the degradation mechanism cannot be explained by the applied methods. Due to the unexplained degradation and colour change of the sample, adipic acid was classified as unsuitable for the PCM module.



Figure 6. Aged adipic acid samples after 1000 h at 165 °C

5.3 Material compatibility

5.3.1 Compatibility of metallic alloys with adipic acid as PCM

As detailed in Table 2, tests in adipic acid were conducted on Al1050, SA 179 and 316L. Figure 7 shows a photograph of one of the test reactors, showing that vapour solidified on the upper part of the vessel and the lid (areas not covered by the band heater).



Figure 7. Glass reactor during tests, showing that high partial pressure led to a solidification of the vapour on the upper part of the vessel and the lid

Initial tests on SA 179 samples (25mm OD, 20mm ID) showed that it was very sensitive to corrosion when exposed to molten adipic acid. Figure 8 (b) shows that the samples following ASTM-G1 cleaning still show the effect of corrosion on their surfaces. Corrosion rates of $6220\mu\text{m}/\text{year}$ were measured for both cleaned samples, highlighting the potential issues to be faced if using this combination of material for the heat exchanger.

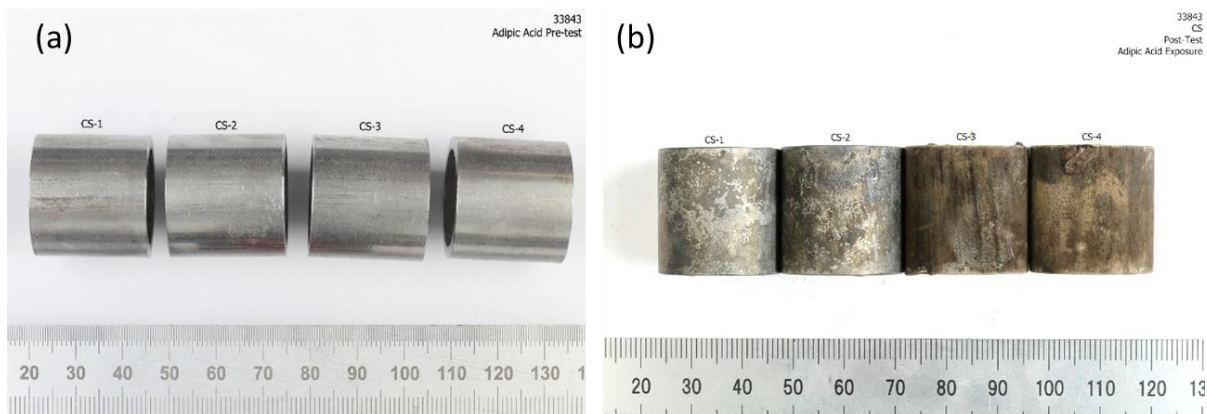


Figure 8. SA 179 samples before (a) and after (b) exposure in adipic acid for 7 days. CS-1 and CS-2 were cleaned following ASTM-G1 and CS-3 and CS-4 are covered in corrosion products

In the case of Al coupons, similar tests were conducted. Experiments with different durations, from 3 to 20 days exposure. Figure 9 shows the samples before and after exposure to adipic acid at 165°C. Samples Al-5 and Al-6 were cleaned following ASTM-G1 and weighed after each cycle to estimate their corrosion rates. Values measured after the 7 days tests estimated corrosion rates of around 120µm/year, and later tests conducted for 20 days estimated values closer to 100µm/year. Both these values remain high for the use of Al fins in the PCM module heat exchanger.

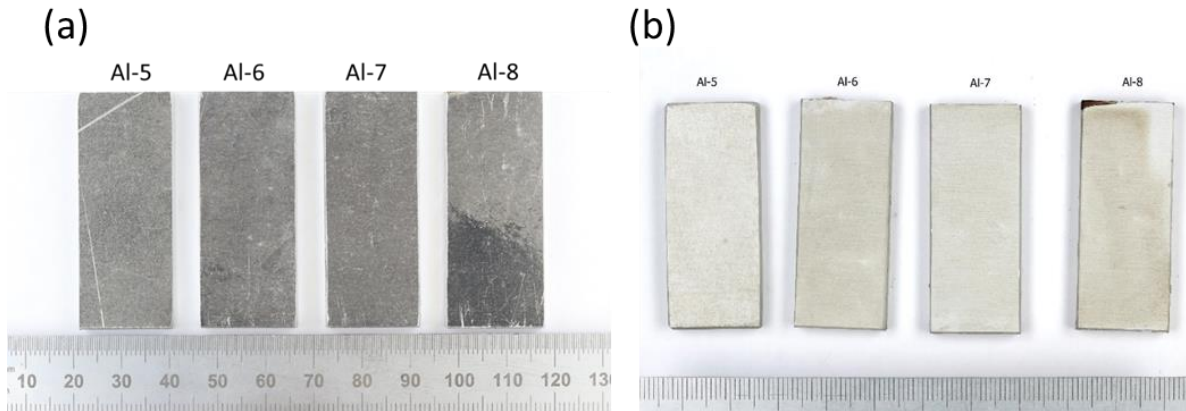


Figure 9. Al1050 samples before (a) and after (b) exposure in adipic acid for 7 days. CS-1 and CS-2 were cleaned following ASTM-G1 and CS-3 and CS-4 are covered in corrosion products

Tests conducted on 316L coupons (Figure 10) showed that adipic acid has a limited effect on this alloy. A corrosion rate of around 50µm/year was measured for both coupons cleaned following ASTM G1, suggesting that 316L remains an option for the manufacture of the PCM module heat exchanger.

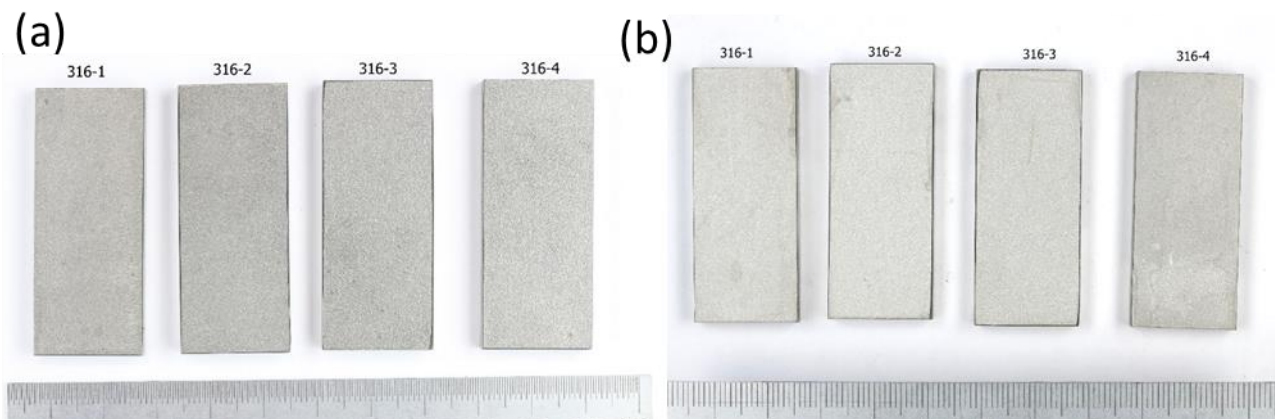


Figure 10. 316L samples before and after exposure in adipic acid for 7 days. 316-1 and 316-2 were cleaned following ASTM-G1 and 316-3 and 316-4 were untouched.

A summary of the tests conducted on adipic acid is shown in Table 4

Table 4. Summary of corrosion rate measurements for the tests conducted in adipic acid

PCM	Substrate	Duration	Corrosion rate ($\mu\text{m}/\text{year}$)
Adipic acid	SA 179	7 days	6220
	316L	7 days	50
	Al 1050	3 days	188
		7 days	120
		20 days	99

5.3.2 Compatibility of metallic alloys with HITEC salt as PCM

Following the tests conducted on adipic acid, further tests were conducted to evaluate the compatibility of HITEC salts as a PCM in a metallic module. Initial tests on SA 179 coupons were conducted (see Figure 11), and corrosion rates of $140\mu\text{m}/\text{year}$ were measured for the three samples following ASTM G1 cleaning. However, SA 179 can face corrosion when exposed to air, and a proportion of this mass loss could be attributed to the corrosion under air. To mitigate these numbers, a reference sample was also cleaned following G1 without being tested in HITEC salt. A mass loss corresponding to $120\mu\text{m}/\text{year}$ was measured, suggesting that a majority of the mass lost during cleaning was due to the surface oxidation of the specimens. Such results suggest that SA 179 is a valid option for material selection in the design of a metallic PCM module.

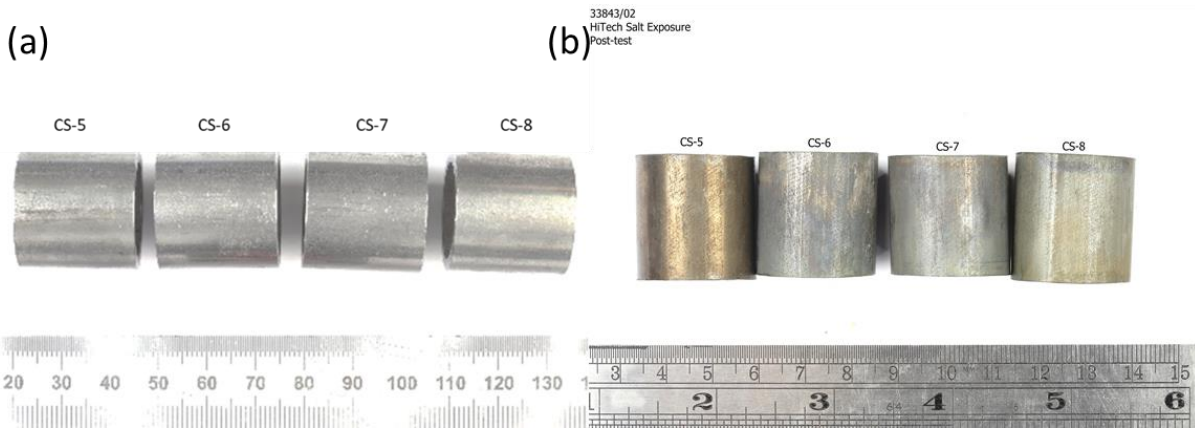


Figure 11. SA 179 coupons before (a) and after (b) exposure in HITEC salt for 7 days. CS-6, CS-7 and CS-8 were cleaned following ASTM-G1 and CS-6 is still covered in corrosion products from the tests.

Tests were also conducted on Al 1050 specimens. Figure 12 shows the photographs of the coupons prior to (a) and after the tests (b). Limited effect on the samples' surfaces can be seen, which was confirmed by the mass loss measurements. Minimal mass loss was measured, and corrosion rates of less than $4\mu\text{m}/\text{year}$ were estimated based on the 7-day tests. A summary of the tests conducted on HITEC salts is shown in Table 5.

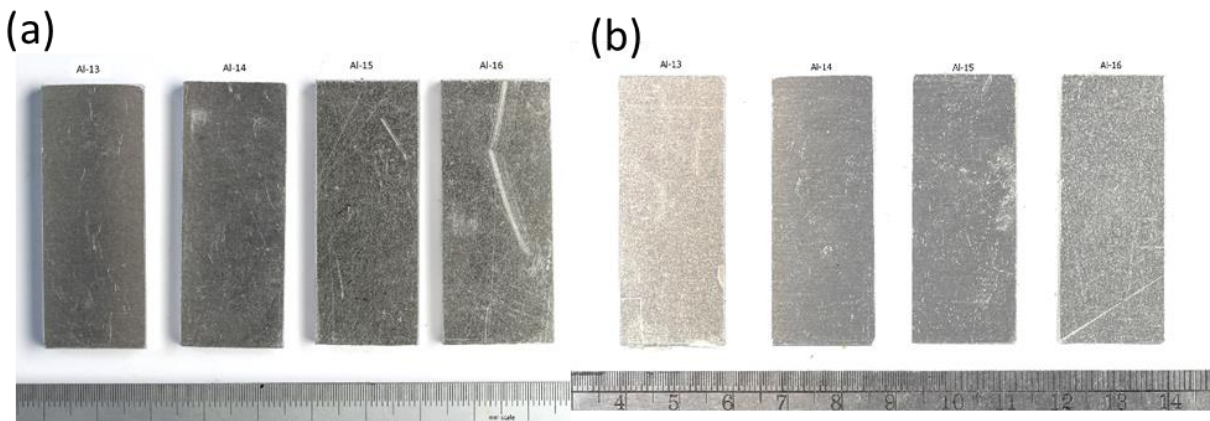


Figure 12. Al 1050 coupons before (a) and after (b) exposure in HITEC salt for 7 days. Al-14, Al-15 and Al-16 were cleaned following ASTM-G1 and Al-13 is untouched.

Table 5. Summary of corrosion rate measurements for the tests conducted in HITEC salt

PCM	Substrate	Duration	Corrosion rate ($\mu\text{m}/\text{year}$)
HITEC salt	Al 1050	7 days	4
	SA 179	7 days	20

6. CONCLUSIONS

The main findings can be summarized as follows:

- Based on literature review possible phase change materials (PCM) were selected in the temperature range from 107 to 165°C.
- Nine materials were characterized by DSC measurement to identify the melting and crystallization temperature, melting enthalpy, cycle stability and subcooling. The organic material adipic acid and the salt mixture HITEC KNO₃-NaNO₃-NaNO₂ 53-7-40 provide the most promising properties.
- An ageing test for adipic acid exposed a stabilizing reduction of melting enthalpy of up to 10% after 1000
- SA 179 was identified as a potential material for the tubes of the PCM module, being a standard material for heat exchangers, condensers or heat transfer equipment in various media including brine.

Based on these findings the salt mixture HITEC KNO₃-NaNO₃-NaNO₂ 53-7-40 has been selected as the PCM. The heat exchanger tubes will be made from low-cost carbon steel. The fins will be made from aluminium.

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