FACULTY OF TECHNOLOGY

Assessment of the potential of using phase change materials for latent heat thermal storage

Arttu Tamminen

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ABSTRACT

Assessment of the potential of using phase change materials for latent heat thermal storage

Arttu Tamminen

University of Oulu, Process Engineering

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Supervisor(s) at the university: Jean-Nicolas Louis, Eva Pongrácz

This work was part of the European Union's Horizon 2020 Making-City program aiming to increase information regarding the positive energy districts. In this study as part of the project, possible use of phase change materials (PCM) in thermal storage was studied to increase its capacity by adding encapsulated materials to the conventional hot water tank. Although the use of PCMs has been studied previously, there still is a need for more practical research regarding the increase of energy content and its discharge properties. Therefore, we studied the theory of PCMs, commercial availability, theoretical viability, and practical viability of PCMs to set up practical test equipment to monitor temperature changes and to measure their efficiency in increasing the energy content.

Four different PCM materials from multiple commercial PCM producers were tested in comparable tests with temperature measured in the function of time. Encapsulated PCMs were selected as they have been suggested to be the best option to nullify the negative properties according to previous studies. Increased energy gained by using the PCM was calculated for thermal storage based on previous studies and using commercial materials technical specifications. Finally, practical testing equipment was made to test PCMs and compare the results with theoretical calculations. All selected materials were tested in four different temperatures 70, 67.5, 65, and 62.5 °C with three cycles for each temperature. All experiments had consistent PCM/water volume relation (1/3) with additional control tests containing just water. After the automated test cycles were finished, temperature data with 30 seconds intervals were collected. Although, the mean temperature showed that all PCMs increased the overall energy content of the storage, the amount of latent energy stored differed as well as thermal conduction rates. The results revealed that compared to the control test with water, the highest calculated energy increase was in the range of 28 to 37 % depending on test temperature, which was lower in comparison to the expected theoretical energy increase.

Keywords: Energy, phase change material, PCM, thermal storage, hot water tank, Positive Energy District, PED, Flexibility, LHTES, latent heat

FOREWORD

I want to thank VTT Oy for this work, which was funded by European Union Making-City project. This made research and material testing possible along with a new outlook for the possible future for these materials as well as their possible role in PED concepts.

Thanks to the supervisors' D. Sc Jean-Nicolaus Louis and Professor Eva Pongrácz from the University of Oulu and principal scientist Klaus Känsälä from VTT for overseeing the thesis and guiding me during the work. Also, I want to extend my gratitude to my college senior research engineer Jari Rehu who was a great help in realizing the test environment, control systems, and data logging for it.

Oulu, 12.09.2021

Arttu Tamminen

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LIST OF ABBREVIATIONS

GHG	greenhouse gas
RES	Renewable energy sources
PCM	phase change material
SHTES	sensible heat thermal energy storage
LHTES	latent heat thermal energy storage
ZEB	zero-emission building
NZEB	near-zero-emission building
PED	positive energy district
c_v	specific heat capacity
K	kelvin
ΔT	temperature difference
m	mass
Q	heat energy
L	latent heat

1 INTRODUCTION

The way energy is produced and consumed is changing fast in the modern world. This is due to strives towards lower GHG emissions and increasing use of RES to prevent the world's average temperature from increasing by 1.5 °C according to the Paris agreement regarding the prevention of climate change (UNFCCC 2021). This has created expanding market for RES and for methods to use, shift, and store energy. In 2014, to hasten this process, European Union started large innovation and research program of Horizon 2020 that lasts until the end of 2020 to aid and create projects that drive economic growth and create jobs in a sustainable way (Europa 2021). As part of this, the project named Making-City was started, which belongs under a "Smart Cities & Communities" area of Horizon 2020. The city of Oulu located in northern Finland is one of two lighthouse cities along with the city of Groningen in the Netherlands. These lighthouse cities work as a testing ground for new technologies and innovations (MakingCity2021). These innovations are to act as a basis for ideas and technology to be used in housing improvements in Europe. In Oulu, multiple companies and organizations such as Arinan Kiinteistöt Oy, Oulu Sivakka OY, Jetitek Oy (Caverion presently), YIT Suomi Oy, Oulun Energia Oy, VTT Oy, and the University of Oulu started to work in close coordination to realize a plan for a PED of Kaukovainio district. The goal is to have a district that is energy neutral and provides surplus energy to the surrounding community. PV panels, use heat pumps to utilize waste energy sources in buildings with the addition of smart metering systems that would allow inhabitants to monitor their electricity consumption. The local market would use excess heat that the refrigeration system produces to supply energy to the district heating network.

As a part of this project, the possible utilization of phase change materials in thermal storage was studied. Theoretically, PCMs can be used to increase energy content with small temperature differences without increasing the storage volume due to their latent heat capabilities (Sharma et al. 2009). However, price and relatively poor heat conduction properties have prevented them from being utilized on a larger scale. Considering changing energy markets and demand for more flexible systems, it is now more viable to examine this method of storing energy.

PCMs have been studied extensively and multiple challenges are known regarding their poor heat conduction properties and issues with corrosion for metals and in some cases their lifetime (Sarbu and Dorca 2019). However, more practical studies were scarce and research with commercialized materials has got less attention. Therefore, small-scale heat storage was made to act as an environment where procured PCMs in multiple cycles are tested, to analyze their increase of thermal energy, and functionality. PCMs were chosen from manufacturers based on their working temperature range of 58 to 60 °C with the idea of using them in domestic hot water thermal storage. Prices and economic feasibility are left out of the studied scope.

This work aimed to study PCMs and their practical functionality in more realistic conditions. The main objective was to increase thermal energy content significantly in the test water tank using encapsulated PCMs and additionally, to provide an overview of the PCMs technological readiness level. Questions to study were; (I) How are PCMs available commercially today?; (II) How viable method are PCMs in increasing the thermal storage capacity?; and finally; (III) How do they function in realistic tests and how is their performance? The goal of this work was to gain present knowledge about PCMs and to utilize the knowledge gained for possible implementation to the positive energy district.

2 POSITIVE ENERGY DISTRICT AND FLEXIBILITY

Since the Paris Agreement in 2015, more international attention is focused on reducing carbon emissions and the United Nations published global sustainable development goals (SDGs), which include the goal 11 sustainable cities and communities goal (UN 2021a). SDGs are a measurement system to set goals and monitor various global challenges regarding social, technical, and political problems (UN 2021b).

The world is urbanizing at in fast rate as cities keep growing and new buildings are being constructed. This increases emissions and it has been estimated in Global Alliance for Buildings and Construction 2020 global report that buildings are responsible for about 38 % of CO2 emissions (United Nations Environment Programme 2020). This makes them one of the largest emission sources in the world and with a growing population and increasing housing demand, the ongoing reduction of emissions is an important and challenging task. These trends have increased research demand for low-energy buildings and cross-sector integration solutions. Heat and energy would be consumed and produced on-site utilizing more Power-to-Heat technologies coupled with aid of hydrogen technology (Hedman et al. 2021) in creating positive energy districts or near-zero-emission districts. Making-City projects are aimed to create replicable and scalable solutions and ideas that can be implemented around the cities in Europe (Alpagut et al. 2019).

Making-City project made it possible to try and utilize the expertise of several companies to realize a PED. In Finland, the city of Oulu is participating in the collaboration of different companies and organizations to implement ideas and technology to the upcoming positive energy district in the district of Kaukovainio. The goal is to have a district that acts as an energy integrated building cluster, which provides excess energy out of the system. New technologies were implemented in buildings to effectively utilize waste heat by using heat pumps. Apartments are equipped with photovoltaic panels that provide energy for the heat pumps when solar power is available. Data is being measured and logged from multiple different locations to give information regarding energy consumption and production. The local supermarket was fitted with the capability to provide energy to the district heating network by using high-temperature waste heat that refrigeration machines with CO_2 as a refrigerant create. Additionally, information for residents regarding their energy consumption is important to provide awareness of their consumption habits and possibly make people change their energy use if they wish.

2.1 Positive Energy District

Positive Energy District (PED) is described as a district that generates more energy than it consumes annually (Lindholm et al. 2021). For the project Making-City, it is defined as: "*an urban area with clear boundaries, consisting of buildings of different typologies that actively manage the energy flow between them and the larger energy system to reach annual positive energy balance*" (Alpagut et al. 2019). The main point for PED is not only to generate energy for the main grid but to minimize the production impact of RES on the main grid since the largest challenge is to control fluctuating power production from renewable energies. Furthermore, the ability to provide load-shifting properties in form of load control, long and short-term energy storage, and smart control systems will play an important role in the future as smart electric devices and grids develop to a commercial scale. Urban areas presently house around 67 % of the world's population with using about 70 % of energy in the world meaning that it is imperative to improve urban construction and deeper integration with other sectors like energy (Brozovsky et al. 2021).

Near-zero emission buildings (NZEB) and zero-emission buildings (ZEB) have been studied more and the concept is more familiar but combining it with the largest urban city planning, PED concept is new but research interest on the topic has been increasing steadily last 20 years (Hedman et al. 2021). Presently one of the tasks in the EU and globally is to find common standards for constructing PEDs since every country has its standards for buildings and energy systems. Additionally, ongoing and future public-funded projects need to be replicable, that the focus can shift from singular positive energy buildings or ZEBs to district-level planning of PEDs. This is known in the EU and many directives have been made and are underway to ensure a standardized way to measure and control energy (EU 2021). As of today, there are multiple ongoing projects regarding the implementation and research of PEDs. JPI Urban Europe has a list of ongoing and planned projects regarding PEDs in a booklet (JPI-UrbanEurope 2021) listing over 70 projects.

In Finland, district heating networks are a vital part of cities' heating systems and in 2019 around 34.5 % of living spaces and hot water were heated with energy from the DH network (Figure 1). As urban areas continue to develop and PEDs are expected to be part of the carbon-neutral solutions in Finland, sector coupling will be necessary to utilize the local heat and electricity sources using the DH network as a conduit to transfer thermal energy.

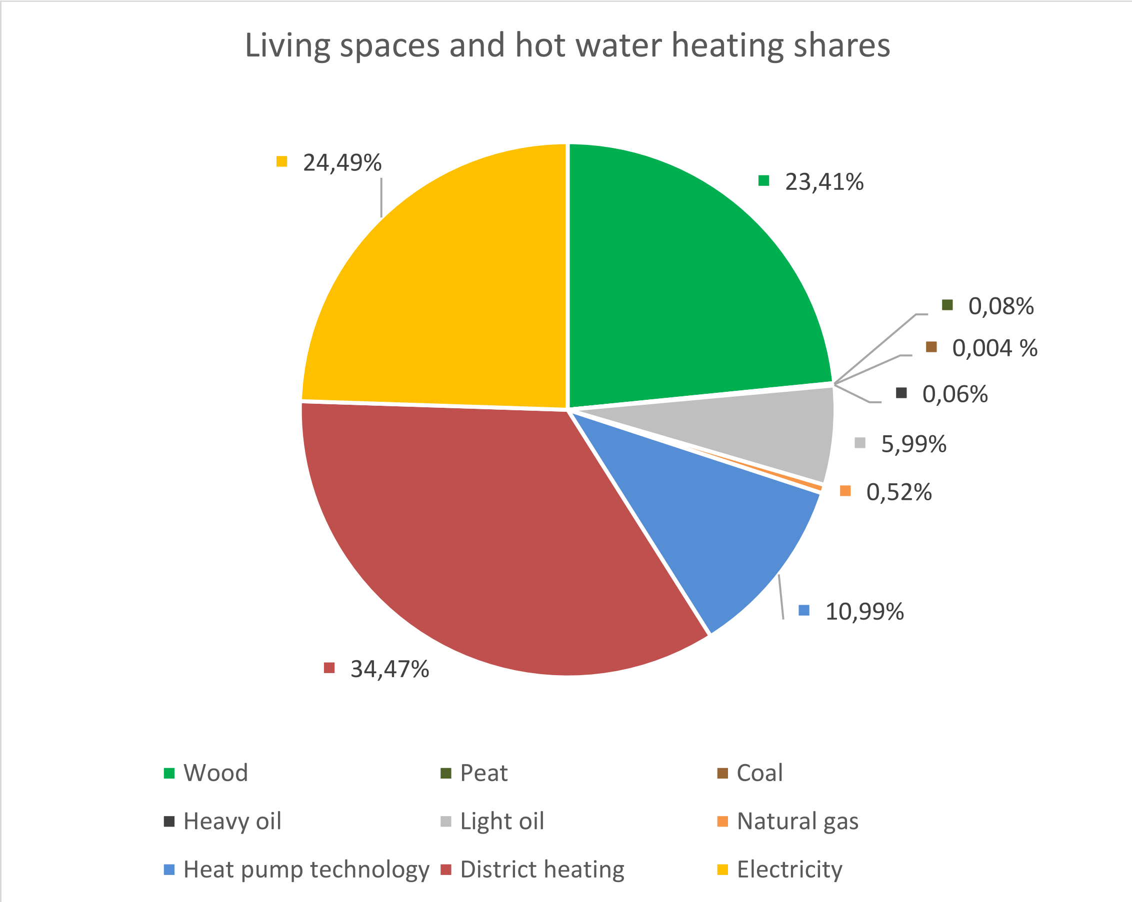

Figure 1 Living spaces and hot water energy shares in 2019 (Tilastokeskus 2019).

Ministry of employment and the economy of Finland set up a working committee in 2020 to compile ways and methods to deepen the integration of different sectors to increase efficiency and reduce emissions to achieve Carbon-Neutral Finland in 2035. Since heating is a major part of the everyday lives of Finnish people, the DH network is going to play a major part in sector integration and most likely in PED solutions. Currently, temperature levels in DH are too high for waste heat sources to be utilized properly especially during the wintertime when heating need is greatest. In the future, DH networks' temperature needs to be lowered and business models around them need reworking to enable more fluent interactions with companies and possibly future prosumers (Työ- ja elinkeinoministeriö 2021).

2.2 Energy flexibility in buildings

As more energy-efficient solutions are required for the future for the European 2050 carbon-neutrality goal, the flexibility of energy use is more likely required. Peak demand is the highest electricity consumption point during the day, month, or year depending on the measuring scale (Susser 2021). The size of the peak is related to the geographical locations with outdoor temperature and amount of solar radiation as well as the time of the year. In Finland, the peak hours occur during morning hours and afternoon hours when people leave to work in the morning and come home in the afternoon (Motiva 2021a). During winter, energy for these peak hours is usually generated with fossil fuels power production or bought from abroad through the Nordpool electricity stock. This makes it expensive and pollutive and with the upcoming renewable energy age, it is important to dimmish these peak hours to a more manageable size. “Peak shaving” is one way to reduce the volume of energy production by moving the power consumption to a wider time frame. This lowers the energy consumptions peak meaning that energy can be produced with more eco-friendly means.

To use apartments as a way to add energy flexibility requires a large number of automated systems and permission from building residents, taking their privacy into account (Guhr et al. 2020). This works more for apartments and buildings that have heating systems based on electricity consumption such as air-, ground heat pumps and direct electric heating systems. Buildings that are in the district heating network have less potential in offering flexibility since their heat source is more of a byproduct of electricity production. By using the heating system in the building, it is possible to reduce heating energy during the peak hour thus decreasing the peak. One idea is to increase the indoor temperature slightly before the estimated peak hours. Electricity consumption increases before the peak but during it, buildings cool down to a set point naturally reducing the consumed electricity during peak hours (Junker et al. 2018). This has to be done in a way that does not disturb the residents or minimally affects their lives. With the successful implementation of this method, it would be possible to have a significant reduction in peak demands on a national scale. The economic incentive for residents would be possible savings when using the electricity before the peak demand hours since during peak time electricity costs are high. Due to the colder climate in Finland, these expenses are mostly part of heating costs meaning that heating uses most of the energy in the wintertime. In Finland, about 26 % of end energy is used for heating of buildings (Motiva 2021b) making it a challenging sector to save energy but also with high energy flexibility potential. Peak shaving in late autumn-, winter-, and early springtime is more challenging due to the constant need for thermal energy, making effective thermal storage and reliable forecasting for peak shifting, a necessary feature for possible flexibility during the colder period. In a study of energy flexibility, it was noted that peak shifting is possible with slightly higher use of energy and in some cases with lower production costs. Even though the amount of energy used will increase, it can be produced with a less emission-intensive method that has reduced impact on the environment. Extremely cold weather days are an exception to reduced costs of production due to high energy needs (Foteinaki et al. 2020). To increase the effectiveness of a flexible system would be to enhance the thermal storage capabilities of the building by increasing the energy capacity of hot water storage, constructing floor heating systems due to their properties to act as heat storage, and possibly installing PCM enhanced acoustic panels or wallboards for example (Frigione et al. 2019). A theoretical study by Najafian A. calculated that PCMs could be used to increase the effectiveness of peak shifting once optimized properly (Najafian et al. 2015). Additionally, when using PCMs in an optimized way, it can reduce heat losses from the water tank when charging for peak shifting due to higher energy capacity without the need to increase the temperature of the water. Since temperature does not need to be higher than usual before the peak hours, the heat losses due to high water temperature are reduced.

3 HEAT STORAGES

Heat has been stored in different mediums such as stone and water for a long time in history. Fireplaces and houses made of stone were used effectively to store heat to keep houses warm for a longer period and with the industrial age, water became the new medium for heat storage. Today hot water thermal storage is used in almost all buildings to buffer and store heat for daily use making them well-known technology. Thermal storage can be divided into two categories, which are sensible heat thermal storage (SHTS) and latent heat thermal storage (LHTS) (Cabeza 2014). SHTS is a conventional way to store energy by raising the temperature of the storage material. The material in the storage can be liquid, solid, or gas medium given that it has suitable properties. Water, for example, is a conventional medium used in households and industries to store energy. Water is cheap and is a thermodynamically well-known substance with additionally having a great heat storage density per weight and volume. Along with water, another popular storage medium is stone. Stone buildings and fireplaces are efficient in slowly absorbing and releasing heat making it good heat storage in buildings. In addition to storing heat, storage can also be made to store cold meaning that we want to cool down something depending on the usage. Basic principles work for both ways in TES and both SHTS and LHTS cases, storages will go through three steps: charge, storage, and discharge.

Today storing thermal energy improves the performance of systems, increases the reliability of the thermal system, and reduces deviances in the system's temperatures (Sarbu and Sebarchievici 2018). Size and energy medium are chosen when designing the storage.

3.1 Energy balance in a stationary closed system and steady-flow systems

Thermal energy storage can be thought of as a close system that has fixed mass with no work external work done meaning that its internal energy can be calculated by energy balance that does not have any energy changes caused by height or velocity, which can be seen in Eq. 1.

$$E_{in} - E_{out} = \Delta U = mc_v\Delta T \qquad \textit{Eq. 1}$$

With U being the internal energy [kJ] of the system, m being mass [kg] and c_v is the specific heat capacity of the material [kJ/kg·K]. ΔT [K]is the temperature difference of the system that shows how much energy is stored in the system compared to a certain temperature level. For example, between the normal temperature of 20 °C and 65 °C, which tells how much thermal energy potential there is when mass is heated or cooled by the amount of temperature difference. Since there is no work in the system, this can be further narrowed down to Eq. 2.

$$Q = mc_v\Delta T \qquad \textit{Eq. 2}$$

This is the most common way to calculate energy content in fixed mass or storage where Q is heat energy in kilojoules [kJ].

3.2 Thermodynamics of heat transfer

Heat is a form of thermal energy that can be transferred between systems caused by their difference in temperature (Yunus. A. Cengel and Afshin J. Ghajar 2015). This transfer of energy can happen in three ways: conduction, convection, or radiation. A temperature difference is needed for the thermal energy transfer to take place where higher temperature systems transfer energy to lower temperature systems until equilibrium, the balance of temperature is achieved.

Conduction is the energy transfer happening between materials or materials particles that are adjacent to each other. This is due to the vibration of atoms that have a higher temperature, which transfers energy to particles that have less energy. This transfer also happens through collisions and diffusions of the molecules. Conduction can happen in all three phases of the materials: solid, liquid, and gas. The rate of this energy transfer depends on the properties of the material or medium of which across energy transfers in addition to temperature difference and thickness of the material. This heat transfer rate can be calculated with the following Eq. 3

$$\dot{Q} = -kA\frac{\Delta T}{\Delta x} \qquad \textit{Eq. 3}$$

Where $\dot{Q}$ is heat transfer [W], k is thermal conductivity [$\frac{W}{m*K}$] of the material, A is the surface area [m^2] of the material, ΔT is temperature difference [K] and Δx is thickness [m] of the material that thermal energy passes through. This is called Fourier's law of heat conduction. Thermal conductivities are measured and available in engineering tables in various sources. Thermal conductivity is not a constant value and it is dependent on the temperature of the material. This however makes the calculations more complex so common practice is to treat it as a constant value if possible.

In convection, the heat transfers from solid higher temperature material, and transfer material are fluid, liquid, or gas. This causes motion to the liquid or gas, which transfers the heat. Heat conduction and motion of the fluid are combined in this method. Without the motion of fluid or gas, heat transfer would be only conduction-based. Convection can be divided into forced convection and natural convection. With forced convection, the motion of the fluid is caused artificially by pumps, fans, wind, or other devices. In natural convection, movement is caused by the increase or decrease of temperature. Change in temperature makes a slight variation to the density of the fluid. For example, floor heating causes air near the floor to increase in temperature, which makes the air warmer and lighter causing it to rise upwards and being replaced with cooler and heavier air. Since there are motions of fluid present, calculating them would prove to be a complex task. However, convection heat transfer is proportional to temperature difference as seen in Newton's law of cooling (Eq. 4).

$$\dot{Q} = hA_s(T_s - T_\infty) \qquad \textit{Eq. 4}$$

$\dot{Q}$ is the rate of convection heat transfer [W] and h is convection heat transfer coefficient [$\frac{W}{m^2*K}$]. A_s is the surface area from where convection heat transfer takes place. T_s [K]is the surface temperature and T_∞ [K] is the temperature measurement point that is some distance away from the surface e.g., air temperature. In this case, the fluid temperature near solid material or temperature source is thought to be the same in similar temperatures. Convection heat transfer coefficient h is determined experimentally and is affected by many different variables such as the shape of the heat source or fluid motions nature meaning that it is not specifically a fluid property.

Thermal energy can also be transmitted by thermal radiation. Energy is transmitted in this case in form of electromagnetic waves also known as photons. Thermal radiation does not require adjacent materials or mediums to transfer heat and pass through a vacuum. The best example of this is the thermal radiation of the sun. Everything that is above the temperature of absolute zero emits this thermal radiation. The highest rate of thermal radiation can be calculated by using Stefan-Boltzmann's law (Eq. 5).

$$\dot{Q} = \varepsilon \sigma A_s T_s^4 \qquad \textit{Eq. 5}$$

Where $\dot{Q}$ is the rate of thermal radiation [W] that the surface can emit in temperature of T_s [K]. $\sigma = 5.670 * 10^{-8}$ [$\frac{W}{m^2 * K^4}$] is Stefan-Boltzmann constant, A_s is the surface area [m^2] of the object and ε is the emissivity of the surface material. Emissivity is the measure of how close the material is to the blackbody, which has an emissivity of $\varepsilon = 1$. A blackbody has the highest rate of thermal radiation that can be achieved at certain temperatures. Calculating thermal radiation rate is a more complex matter than conduction or convection due to the nature of radiation where it bounces around from surrounding materials and fluids.

Calculation of thermal energy transfer in solid is in reality, a complex combination of conduction and radiation. Alternatively, if fluid is present, the convection factors are taken into account instead of conduction. These three methods cannot exist at the same time during heat transfer. In solids, conduction happens in materials that are not transparent but with transparent materials, there exists also radiation alongside conduction. Additionally, on the surface, heat transfer can happen with convection and radiation. This means that convection heat transfer cannot happen in solid materials as stated previously but it can be calculated as conduction, but this means that motion is not present at the fluid. In the case of thermal radiation vacuum is the only medium that has heat transferred only by radiation (Yunus. A. Cengel and Afshin J. Ghajar 2015).

3.3 Sensible heat storage

Sensible heat storage is the conventional way of storing thermal energy by increasing the temperature of the storage medium without causing the material to change its phase. Water, bedrock, oil, air, or bricks can act as a medium. A suitable material is usually chosen by the purpose of the storage, heat capacity of the material, and space available. Water has excellent qualities regarding thermal storage capabilities as seen in Table 1 with considerably high qualities in both specific heat capacity and heat content. Water storages are found in every apartment buildings and houses that have domestic hot water lines. It stabilizes the consumption and acts as a buffer enabling pleasant usage of warm water and more adding more control for the heating system. (Sarbu and Sebarchievici 2018)

Table 1 Specific heat and heat content per volumes of certain materials (Cabeza 2014)

Material	Specific heat [kJ/(kg*K)]	Heat content per volume [MJ/(m^3*K)]
Water	4.2	4.2
Oil	2	1.7
Concrete	0.8	2.1
Granite	0.8	2.1
Steel	0.5	3.6
Paraffin	2.9	2.6
Wood	1.8	0.9

Sensible heat storages have almost linear temperature curves depending on the storage medium used in them. In Figure 2 sensible heat energy increase is illustrated as a linear line comparing it to latent heat energy. In SHS volume and material types are only things contributing to the energy intensity of the storage.

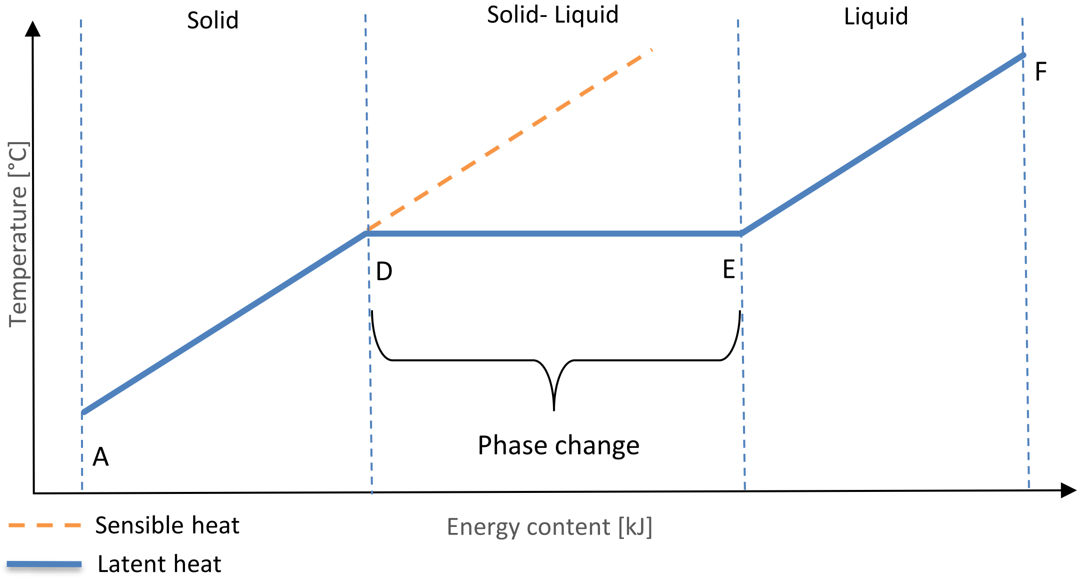

Figure 2 Sensible and Latent heat's increase of energy content (Regin et al. 2008)

3.4 Latent heat storage

In latent heat storage, energy is stored by utilizing the materials change of phase phenomenon. Water is a familiar substance that shows all three states of solid, liquid, and gas in our everyday lives. In temperatures between 0 °C and 100 °C water is in a liquid state at normal atmospheric pressure of 101 kPa and below 0 °C it solidifies or freezes. Above 100 °C water starts to vaporize and changes its phase to gas or steam in case of water. During these changes, the temperature does not change but energy is being absorbed or released by the molecules (Wu 2010). This makes it possible to utilize this feature as an energy-enhancing method. The material used in the storage is chosen by melting or solidification temperature needed. In theory, once the temperature increases to the melting point of the chosen material, the increase in temperature stops until complete melting of the material has been reached. This means that a larger amount of thermal energy can be stored in the same temperature difference compared to sensible heat storage. With water, between solid and liquid states, for one kilogram of water, 334 kJ of energy is needed to transition between states (Legates 2005). Even though in theory, the temperature does not change during this process, in reality, this applies only in close proximity of material meaning that temperature fluctuation is present. The energy required for the material to change its state is called latent heat. Latent heat is much greater than the specific heat of substances meaning that utilizing the latent heat, a larger energy amount can be stored (Wu 2010). Materials that are designed to be used as latent heat storage are called phase change materials (PCMs) and they can be classified as organic, inorganic, and eutectics. This classification was made by Abhat A, (Abhat 1983). Each type has advantages and disadvantages depending on the intended use. Even though PCMs latent heat is much larger than the specific heat of other storage mediums such as water, their specific heat themselves is usually much lower meaning that as temperature difference increases, the latent heat energy can negatively affect the thermal storage.

4 PHASE CHANGE MATERIALS

Phase change materials (PCM) are materials, which are used to store energy across two phases. Material molecules have bond energy that keeps the molecules together in solid or liquid form. Once enough energy is fed to the material in form of heat, for example, molecules will overcome binding energy and the material will change its phase to gas. This is called latent energy and it does not change the composition of the material but instead will absorb or release energy without changing temperature during this change of phase (Yunus. A. Cengel and Afshin J. Ghajar 2015). Several aspects should be taken into consideration in PCMs that are desired working temperature and large latent heat fusion per mass to achieve more dense energy storage. Other things that also need to be taken into consideration are heat conduction capabilities of the material, possible supercooling, long-lasting phase change repeatability, and amount of volume change during the change of phase.

Phase change materials can be classified by in which phase the material works (Cabeza 2014). For example, solid-solid, solid-liquid solid-gas, or liquid-gas. Solid to liquid and back is the most used one with the most information available due to its simplicity and high latent heat. PCMs can be categorized into two sections by their material to organic and inorganic. Organic materials are, for example, paraffin and non-paraffin fatty acids. Inorganic materials are different salt hydrates, saline composites, and metal alloys. Classification of PCMs can be seen in Figure 3.

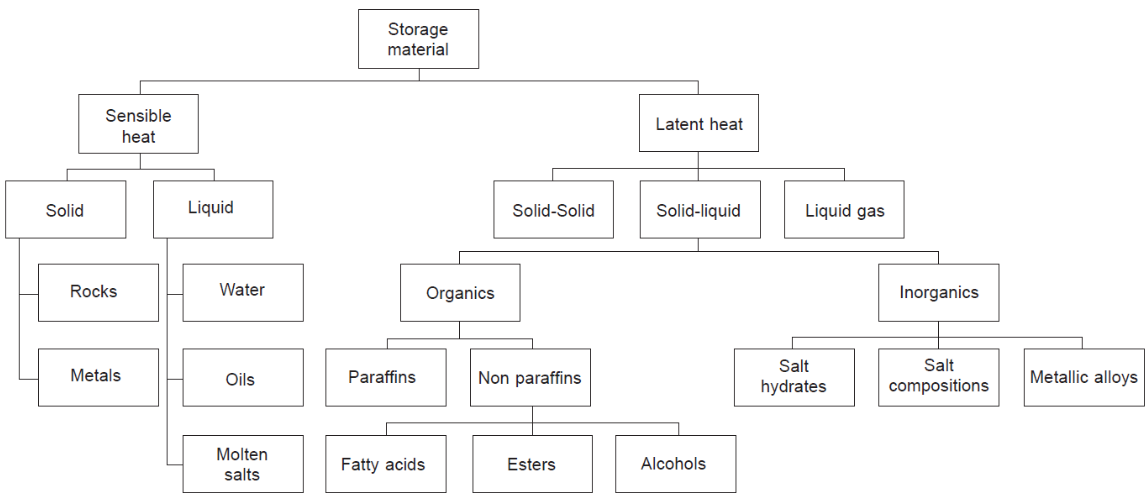

Figure 3 Categorization of phase change materials (Cárdenas and León 2013)

In lower temperature applications, the choice is usually between organic and inorganic compounds when choosing the storage material.

4.1 Organic PCM

Organic materials are further divided between paraffin and non-paraffin. Organic materials have consistent melting and solidifying without any phase segregation or loss of latent heat fusion properties (Alkan 2006). Additionally, they solidify with little to no supercooling and are often non-corrosive. Supercooling can be seen especially with some inorganic materials where the material is in a liquid state and temperature decreases lower than solidifying temperature before starting to release heat and in some materials, this can negate the wanted benefits if supercooling is too effective. Paraffin is mostly made up of mixtures of straight n- alkanes (Figure 4) with crystallization of the CH chain releasing a large amount of heat. As the number of carbon atoms and length of the chain increases, so does melting point and latent heat fusion energy. This means that material is available in considerable temperature ranges.

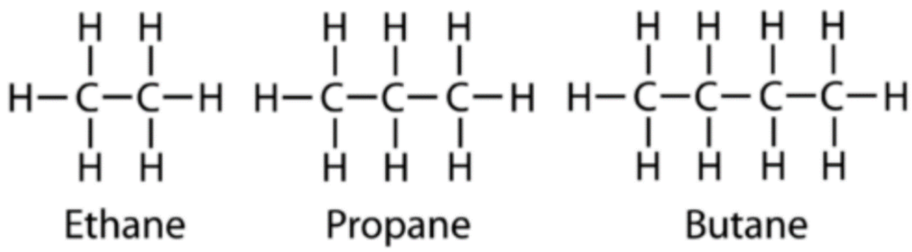

Figure 4 example of alkene chain (Bewick et al. 2021)

With good properties regarding congruent melting, a long lifetime without degradation, and being ecologically friendly, they are not without issues. Undesired properties are moderate flammability, compatibility issues with plastic containers in some cases, and the largest of all being low thermal conductivity. These challenges can be overcome with the modification of the wax and the way the storage is built.

Non-paraffin organic PCMs are numerous with great varieties in their performances. They consist of different esters, fatty acids, alcohols, and glycols. They are usually more flammable which must be taken into consideration when choosing the material. The properties of these materials show are flammability, poor thermal conduction, low flash points, toxicity in some cases, instability in higher temperatures, and high heat fusion energy (Sarbu and Sebarchievici 2018). Fatty acids have better latent heat capabilities than paraffin and additionally, they have non-degrading melting and solidifying cycles and show no supercooling during solidification. Lastly, the most important factor regarding fatty acids is their cost which can be twice or more expensive than paraffin and they have some corrosive properties for metals. Some of them have supercooling properties as well as some degradation in melting and freezing cycles. This is not the case with all of them and they are sold commercially with a promise of not losing their functionality during thousands of cycles.

4.2 Inorganic PCM

Inorganic PCMs are salt hydrates, salt compositions, and different metallic alloys. Salt hydrates are the most used ones in this category with the best example of being the hand warmer pouches that release heat when the metal disc is snapped causing the exothermic reaction of sodium acetate solidifying. Salts crystalline with water to solidify and solid to liquid transformation is dehydration of salt and this process resembles changing of phase thermodynamically. The problem with the salt-based materials is that due to the nature of the process of hydrate crystals breaking into anhydrous salt and water this can cause incongruent melting causing segregation of the material. This shows as particles forming in the bottom of the container due to density differences of solid and liquid, and ultimately lowering the efficiency of the storage. Salt hydrates are also showing supercooling of liquid before solidification, but the issue can be addressed by modifying the mixture with a nucleating agent.

Salt hydrates are the most interesting group of PCMs in lower temperatures (under 100 °C) because of their high latent heat fusion, low volume change during the melting-freezing cycle, and good thermal conductivity compared to paraffin. They are slightly corrosive for metals due to salt but are compatible with plastics and are eco-friendly. They are also economically more reasonably priced (Sharma et al. 2009). Major issues with suitable salt hydrates are the mentioned incongruent melting where salt is not entirely soluble in water hydration at the melting point. Moles of water in hydration are not enough to dissolve one mole of salt. The result of this causes particles of salt to settle on the bottom of the container and it stays crystallized during the melting period, which means loss of latent heat capacity. This process is irreversible, and it will deteriorate the thermal energy capacity of PCM. These issues can be fixed with nucleating agents and creating mixtures that improve the properties of the material. Other methods are mechanical stirring, thickening of the material preventing the segregation, adding more water to the solution to increasing solubility but this reduces latent heat capacity. Lastly encapsulating the material to reduce the separation of salt in the material. This is done by companies that sell these materials commercially, however, these actions increase the cost of the material making it more difficult to be economically viable.

Metallic alloys are low melting metals and metal mixes (eutectics) that can work as an energy storage medium. Metallic material's good properties are high latent heat energy per unit volume additionally with very high thermal conductivity and low vapor pressure. Some metallic PCMs can have low melting points around 30 °C making them possible to be used in low-temperature applications such as apartment heating or district heating systems.

Eutectic materials are in minimum composed of two or more materials that solidify and melt congruently. This kind of composition prevents segregation of the materials since they solidify into a dense crystal mixture and melt together simultaneously without separating (Sharma et al. 2009).

4.3 Calculating the energy of PCM

When calculating the energy of PCM it is important to consider the thermal energy that is stored in the material when it is solid and liquid. This can be calculated with Eq. 6, which was obtained from (Regin et al. 2008).

$$Q = m * \left[\int_{T_A}^{T_D} C_{ps} * dT + L_p + L + \int_{T_E}^{T_F} C_{pl} * dT \right] \qquad \textit{Eq. 6}$$

Where *m* is the mass of PCM in [kg], and T_A is the starting temperature of solid material. T_D is the temperature when the material starts to melt. T_E is the temperature when the material has melted, and T_F is the final temperature of liquid with all temperatures being at °C. C_{ps} is the specific heat of solid material and C_{pl} is the specific heat of material in liquid form [kJ/kg*ºC]. L_p is the materials solid-solid latent heat [kJ/kg]if present and *L* is the latent heat for solid-liquid in [kJ/kg]. These temperature points are visualized in Figure 2 presented in section 2.3.

During cooling the same amount of energy is released but it can differ in form of supercooling when the material starts to solidify. Energy could be calculated by simply multiplying the latent heat capabilities given by the manufacturer with a wanted mass, but it is not an accurate method. This does not take into consideration the heat conductivity of the material.

4.4 Supercooling

One aspect of the PCMs is supercooling or subcooling which, depending on the usage of the material can be a positive or negative feature (Safari et al. 2017). Supercooling can be thought of as delay of solidification and it always takes place when PCM goes through a change of phase from liquid to solid. At this moment solidifying does not happen when solidification temperature is reached instead temperature will go below that level before crystallization occurs. In this brief moment, the material is in a metastable state. Some sensible energy is lost on this process, but the major issue lies with the larger temperature difference needed for the utilization of the latent heat with PCM. Since the major benefits come from the small temperature difference needed for a large portion of the energy stored than in SHTES, supercooling phenomena increases the temperature difference required hence making it less efficient technology. In case of wanted effect, a good example is a hand warmer sold in stores where PCM is in liquid form, and once the metal disk inside the bag is “clicked” the rapid crystallization occurs releasing heat. Figure 5 presents different temperature curves involving supercooling.

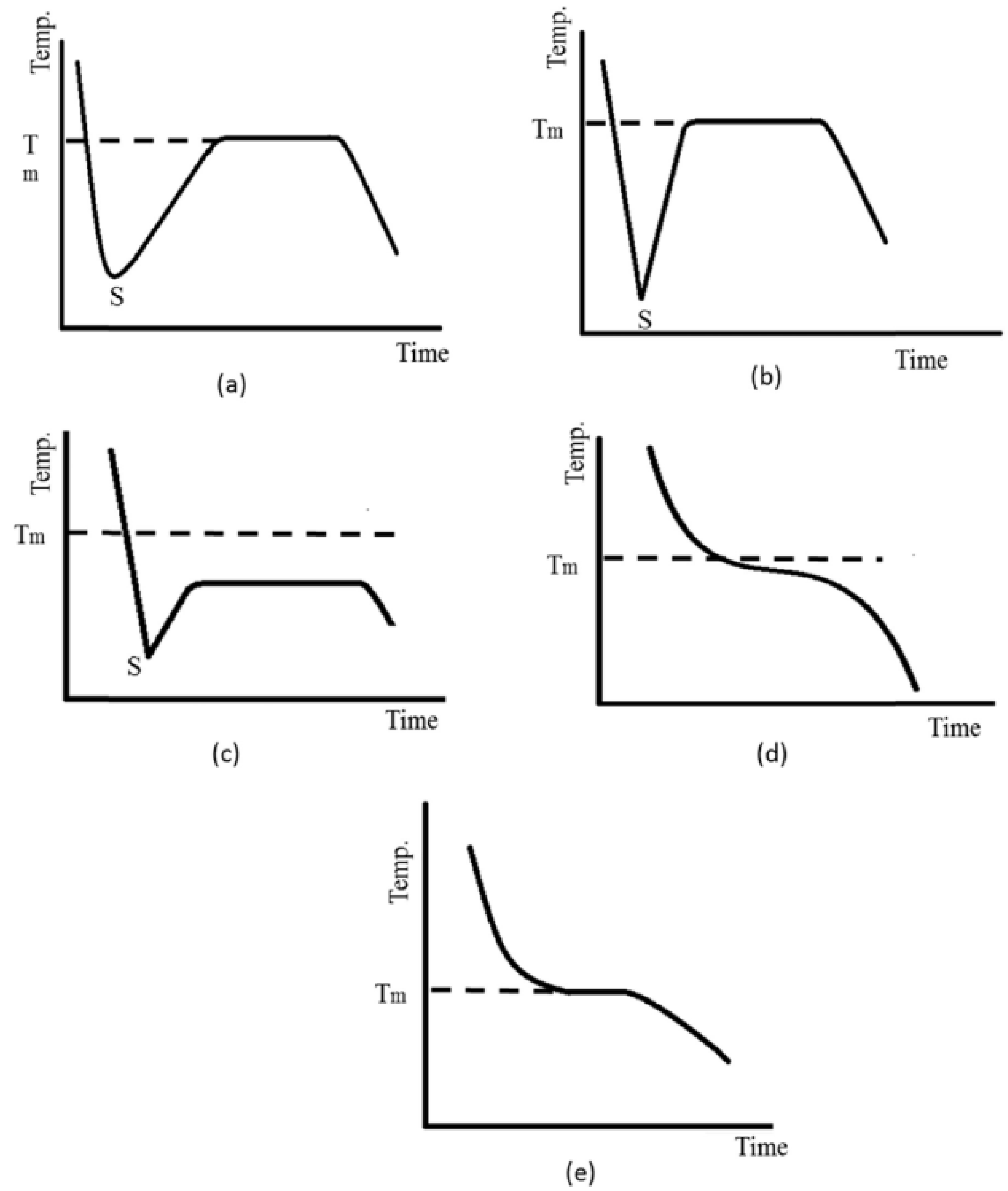

Figure 5 Supercooling curves (Garg et al. 1985)

In Figure 5, graphs a) and b) supercooling happens due to poor nucleation of the material meaning that crystallization does not occur immediately but once it starts the temperature increases to T_m level. In graph b) the material has better thermal diffusivity than in a). Graph c) supercooling is caused by poor crystal growth rate even though nucleation started. This problem presents itself by temperature unable to reach T_m level. Graph d) shows that solidification starts at T_m level but supercools slowly most likely due to high rate of heat removal meaning that heat conduction from the material is too low for the amount of heat removed. in final graph e) implies low thermal diffusivity in liquid and temperature takes longer time to reach T_m level meaning that material starts to solidify further away from the temperature sensor. Ideally, graph e) is showing the wanted result from PCM storage where the temperature is stable through the phase change of the material with no supercooling present.

5 IMPROVING HEAT TRANSFER OF PCM AND CURRENT COMMERCIAL LEVEL

One of the largest issues regarding the use of PCMs is their lack of proper heat conduction. There are many papers written regarding this issue with proposed solutions including drawbacks of these methods. During the heating of the storage with PCM, the thermal transfer starts with conduction but quickly turns to convection heat transfer due to the melting of the material next to the heat source, which means that heat is transferred by liquid moving due to convection forces. During this time, the heat transfer by convection is a generally faster process than solidification but still can be a relatively slow process, especially with larger volumes. When energy is taken out of the storage, the process happens in reverse order. Thermal energy is first transferred via conduction since material solidifies to the lower temperature surface area creating a “protective layer” to it reducing the heat transfer rate. Heat transfer by convection gets gradually smaller and more heat is transferred via conduction in solid material. Thermal conductivity can be improved by either improving the heat transfer area of the storage or increasing the thermal conductivity of the PCM in the storage (Cabeza 2014).

5.1 Compact latent heat storage and additional heat transfer area

Compact latent heat storages have a higher volumetric amount of PCM than in systems using encapsulated PCM, which in theory contains a higher amount of energy and longer output of energy. Challenge in these storage types is the low heat transfer rate with a large bulk amount of PCM. Such tanks can be tube-and-shell type solutions or U-tube heat-exchanger type such as Figure 6 presents by (Nakaso et al. 2008) where steel pipes go through PCM-filled containers. Such latent heat thermal energy storage (LHTES) requires structural designs to compensate for the poor heat conduction properties of PCM.

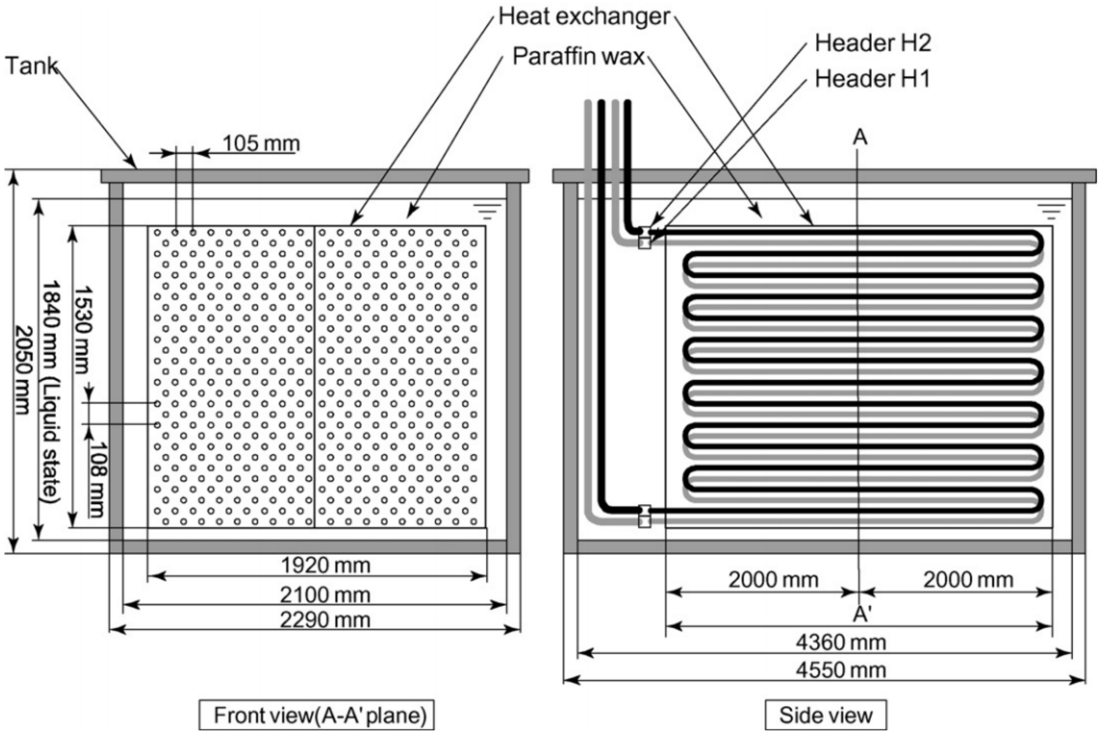

Figure 6 Schematic of LHTES tank by (Nakaso et al. 2008)

One way to increase the heat transfer rate is to add more surface area that is in contact with the PCM that is creating thermal energy transfer resistance on itself. One of the topics for research has been different ways to increase the heat conductive properties of PCMs. One of the original driving forces for the utilization of PCMs in energy and temperature control was NASA during its Space Program (Fan and Khodadadi 2011). The enhancement for thermal conductivity was done in the 1970s by Humphries and Griggs by embedding metallic fillers to PCM such as metallic wool, foam, and honeycomb structures (Humphries and Griggs 1977). This increases the heat conduction properties of the material due to increased surface area that transfers heat faster. The downside to this is the reduced amount of PCM caused by filling material and the increase in weight and volume. For the metallic filling, they settled upon the honeycomb structure due to its versatility in expanding conditions. Another studied method was adding fins in radial or axial direction on the tube containing HTF surrounded by PCM (Figure 7). This method has been studied extensively because of its simplicity and low cost. One note was that fins in the tube should be relatively thick since with thin fins the heat transfer increase was extremely low (Chow et al. 1996). When compared to the unfinned tube versus the thick finned tube the heat transfer coefficient was twice higher with a finned tube. Results of how these fins affect the thermal transfer depend on their configuration or placement in the storage, for example, their shape, size, and the number of fins added. The material of these fins is also important regarding their heat transfer coefficient, density, resistance to corrosion, and costs.

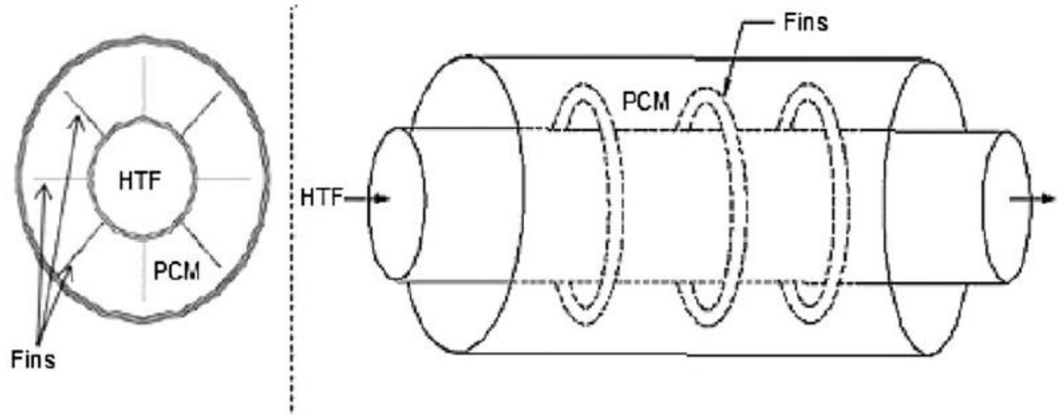

Figure 7 Longitudinal fins (left) and annular fins (right) (Sarbu and Dorca 2019)

Later other studies have been conducted relating to Humphries and Griggs studies by adding materials to enhance heat conductivity. Added material can be thought of as “porous” material and its porosity affects how well it transfers heat. This idea can be used for the numerical study of the subject (Mesalhy et al. 2005). Material matrix also affects the system in terms of effectiveness and costs. Adding the heat conduction material to the PCM reduces buoyancy greatly due it is hindering liquid material’s convection flow, but overall increasing the heat transfer (Epstein and Cheung 1983). In addition to metal matrices mentioned earlier, there have been studies with metal rings or containers that have been built in such a way where the surface has been increased for better conductions properties. Since adding metal increases the weight of the storage and takes up the volume from PCM itself other materials have been investigated such as carbon fibers around the copper tube (Nakaso et al. 2008) which contains HTF (Figure 8). Carbon fibers are non-corrosive meaning that they could be utilized with salt-based materials, which are highly corrosive to metals. Additionally, carbon fibers have a lower density than metals which means that they take less volume and weigh less, which can be desirable properties.

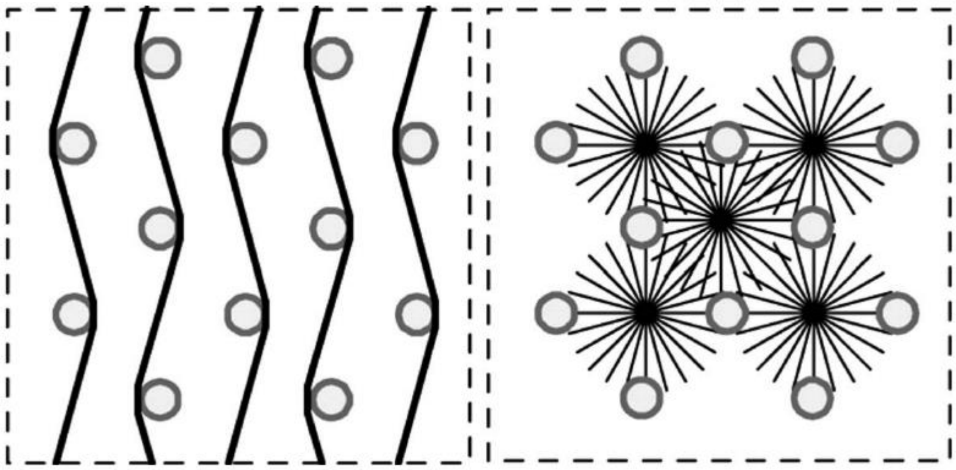

Figure 8 Thermal capacity enhancement using carbon fiber cloth (left) and brushes (right) with copper tubes (Nakaso et al. 2008).

Adding Lessing rings (Figure 9) also proved to be a working way in increasing thermal conductivity (Velraj et al. 1999). Adding these metal rings provided 10 times better thermal conductivity than without rings.

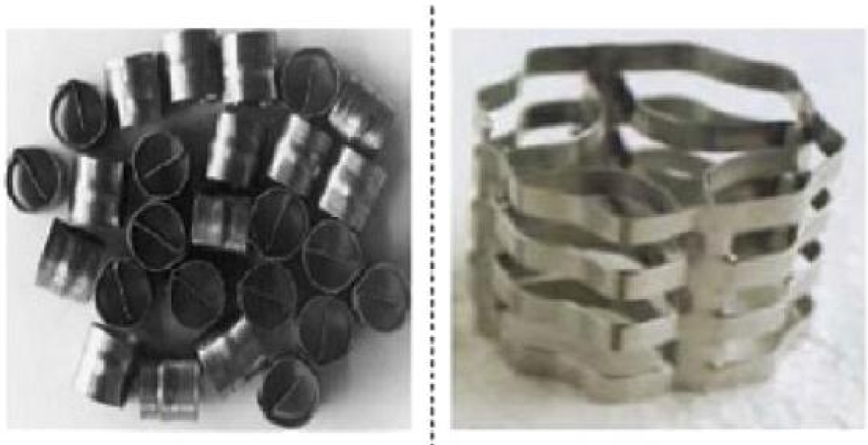

Figure 9 Metallic Lessing rings that have been tested to increase thermal conduction rate (Sarbu and Dorca 2019).

5.2 Incorporating PCM into a porous material

Impregnation of porous material with the PCM has also been studied. This would increase the thermal conductivity of the material and in some cases also negate volume change during cycles. In one study by (Sari and Karaipekli 2007) pure paraffin and impregnated paraffin expanded graphite composite was compared. Impregnated composite proved to increase thermal conductivity but because of the inconsistencies in its structure, the transfer of heat was not continuous. It was noted in another study (Zhou and Zhao 2011) that adding metal foam also increased thermal conduction well and additionally the rate of heat transferred was better because of more consistent structure. These methods improve heat conduction, but it also decreases drastically the latent heat capacity due to the lower volume of PCM and investment costs increase when using PCM/graphite. (Yin et al. 2008) they studied a composite of paraffin and exfoliated graphite nanoplatelets (xGnP^{TM}) which increased thermal conductivity and possessed the same latent heat capacity as paraffin on its own. Composite can be absorbed also with fatty acids or inorganic salts.

5.3 Encapsulation of the PCM

Encapsulation of the material is also a viable way to negate the unwanted effects and reinforce wanted properties that are the longevity of the material with inorganic PCM and increase in heat conduction rate due to increased surface. Encapsulation can be divided into macro-, micro-, and nanoencapsulation, which refer to the size of the capsules for the material. Microcapsules diameter can vary from 1 mm to 1 μm and smaller than that are defined as nanocapsules. Microcapsules are coated with encapsulation material that is usually polymer-like material (Kuznik et al. 2011). Encapsulation helps the PCM to be incorporated directly into building materials to add cooling capacity to buildings or in larger macrocapsules, which can be used to increase energy content in the storage.

As mentioned, PCMs have usually a rather low heat conduction rate and additionally, some have corrosive effects on common steels and metals. Organic materials have low heat conduction but good longevity regarding many heating and cooling cycles. As in inorganic materials, especially salt-based materials have better heat conduction than organic ones and possess generally repeatable melting and solidifying cycles. However, salt-based materials are prone to have segregation in a long term causing the material to lose its thermal capacity. Encapsulation can help to mitigate these issues since this increases heat conduction rate meaning more effective charge and discharge of energy. Longevity also increases with inorganic materials since segregation can be negated with a small volume of capsules. This also helps with corrosion problems with conventional water storage. Encapsulation means that material can be added afterward to the older conventional water tank to increase energy density meaning that it can be possible to enhance existing thermal storage. This however requires a superb quality capsulation to ensure long maintenance-free usage, which in turn increases overall costs.

All methods that aim to increase the thermal conductivity in the storage by increasing the surface for better heat conduction are to a certain point beneficial. However, adding extra material to the storage reduces its energy capacity, and to make up for the loss of capacity the volume of the storage needs to be increased. Originally one of the good properties for latent heat storage is its lower volume compared to conventional sensible heat storage. These additions for thermal conductivity also increase the complexity of the storage, which directly affects the investment cost.

5.4 Cascade storage

When working with larger temperature differences, the advantages of the latent heat system diminish, meaning that sensible heat storage can be a more reasonable choice. This can be fixed by utilizing a cascade type storage where PCMs with different melting and solidifying temperatures are used. The flow of the HTF must be thought out to make sure that heat flux to the materials is constant.

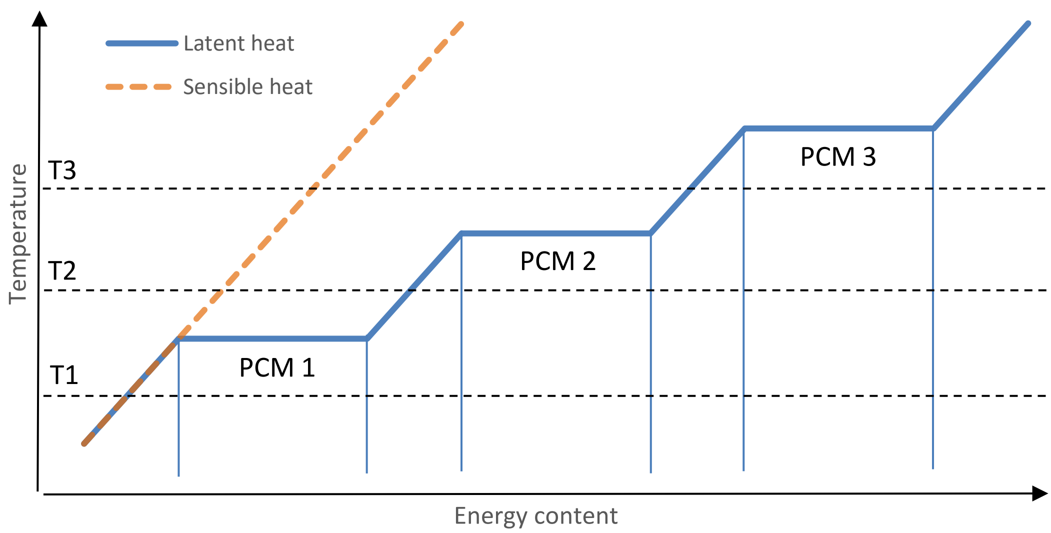

Figure 10 SHS storage compared to cascaded LHS (Sarbu and Dorca 2019)

During the melting process, the hot HTF is fed into the storage where the first material has melting point at that temperature. As the HTF transfers heat to melting material its temperature drops down to the level of the next material in storage that continues to absorb heat energy. Cascade storage can contain any number of materials long as suitable ones are found. Well-designed cascade storage with properly chosen materials can display good performance.

5.5 Commercial producers

For the project's needs, it was necessary to use commercial producers of PCMs instead of developing something new. In addition, to avoid additional expenses, encapsulated material was looked into. Otherwise, storage itself would have needed to be redesigned to work without capsules. The end goal was to implement the storage to fit the needs of the apartment building and local market, so it was crucial to use proven technology in the storage tank. Encapsulated material would be easy to insert into the conventional water storage and if needed, changing the material or removing it completely is possible. Reading publications regarding the subject, multiple manufacturers were found. Criteria for material were encapsulation, functionality in the hot water storage tank, and proper working temperature range. After a search, five companies that had PCMs available replied to our inquiries, had encapsulations available with suitable temperatures were chosen.

(i) Axiotherm GmbH is a German company with a wide range of PCMs and they offer encapsulation services alongside storage tanks. The range of their products goes from -40 to 120 Celsius.

(ii) Rubitherm GmbH is also a German company that focuses on PCMs with more focus on lower temperature applications. They offer encapsulation for the lower temperature materials in form of macro encapsulation and microencapsulation. The range of materials varies between -10 to 90 Celsius.

(iii) Climator Sweden AB Company is located in Sweden and it focuses on energy storing PCMs. Their products range from -21 up to 70 Celsius. They also offer encapsulation of materials in aluminum packages.

(iv) Croda is a British chemical company that produces various chemicals with CrodaTherm™ being their brand for PCMs. All their products are bio-based waxes that are not salt, or paraffin-based. Their products range from -22 to 60 Celsius with a focus on lower temperatures. They do not offer encapsulation services, but they have collaboration with Axiotherm that offers encapsulations for the materials.

(v) Global-E-Systems Europe B.V is a Dutch company that also produces PCMs with their encapsulation services. The range of temperatures is from 7 to 89.3 Celsius with mostly inorganic salt-based materials.

These companies provided some test materials for the project for testing. There were other companies also found, which are: PureTemp LLC located in the USA, Sunamp Ltd located in Scotland, Phase Change Energy Solutions Inc. located in the USA, RGEES LLC located in the USA that is also working with Pluss Advanced Technologies B.V. that is an Indian company. All of these companies were contacted but they did not have suitable material for the tests or some did not reply for contacts.

5.6 Technological readiness level

Technological readiness level (TRL) is a way to assess the maturity of the technology in easier terms. The origin of TRL is from NASA in the 1970s to assess levels of space technologies (Tzinis 2015; TWI-Global 2021) but it is used to assess other technologies as well. There are 9 levels in measurement as defined by NASA (Figure 11) with level 1 being a theoretical idea and 9 being most mature with the real operating system. For example, mono- and polycrystalline photovoltaic solar panels are in level 9 with commercialized systems.

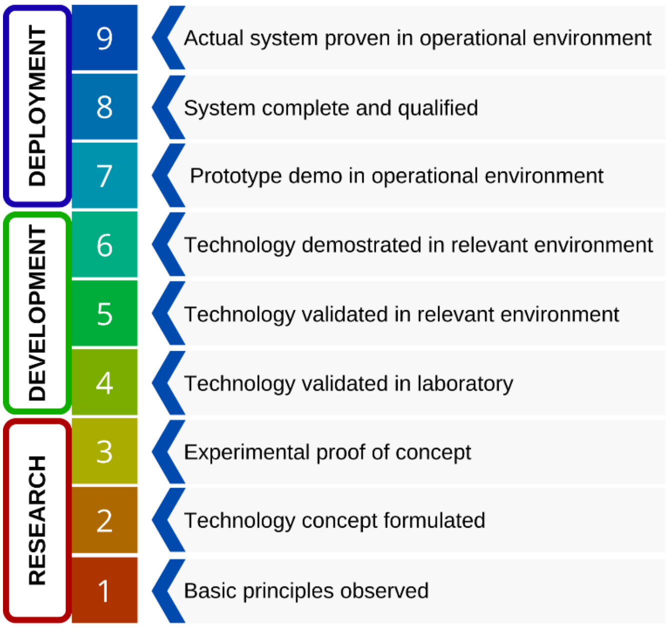

Figure 11 Technological readiness level table (TWI-Global 2021)

Levels 1 to 4 are usually government-funded and Universities focus their studies on these levels. In these first stages, concepts are on a theoretical level where it is not usually economically profitable to invest. For this reason, the private sector mostly focuses on levels 7 – 9 where technology has been demonstrated to be functional and potentially has promise. Between these levels from 4 to 6 is the most difficult part where neither private nor university side focus too much leading many technology developments ending on these stages. Since the material for testing is purchased from commercial producers, the TRL for these materials can be thought to be in the deployment section.

5.7 PCMs in a test environment

There has been much research done to increase heat conduction properties of PCMs and studies where different PCMs are tested in built testing environments. The University of Lleida and the University of South Australia have done such a study for static thermal energy storage (Gasia et al. 2018; Tay et al. 2018) meaning that PCM is stationery and energy is transferred to material using a heat transfer fluid (HTF). Research equipment manufactured at the University of Lleida in 2008 was compact LHTES made to withstand higher pressure and temperatures to allow a high variety of different PCMS to be tested. Three tanks were made containing shell-and-tube heat exchangers (Figure 12) with structural differences between them such as added square fins to increase heat transfer area (Tank 2). Tank 3 has different tube dimensions and distance between tubes and rounded end of the container.

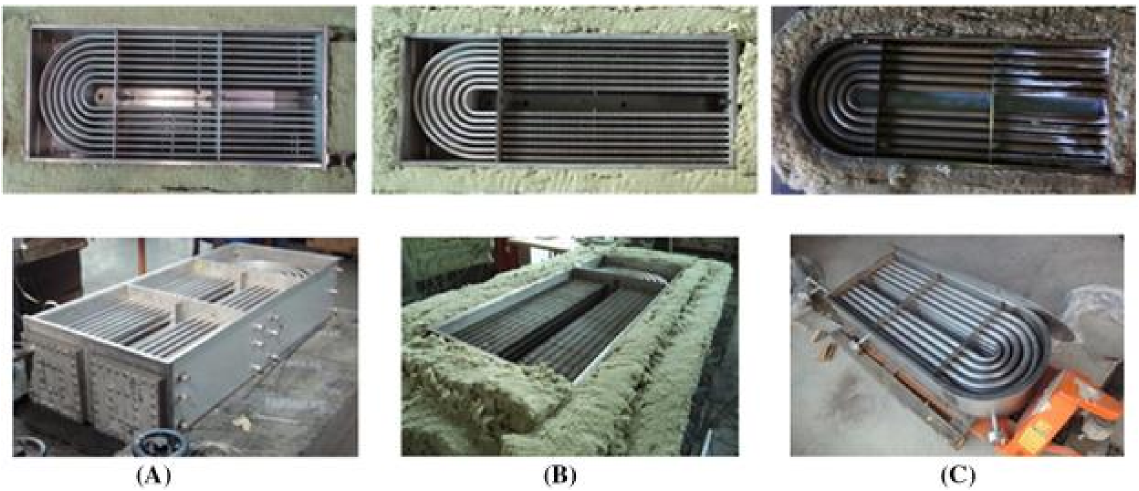

Figure 12 Storage systems made in the University of Lleida. (A) Tank 1; (B) Tank 2; (C) Tank 3 (Gasia et al. 2018).

Their focus was on higher temperature PCMs with a temperature range between 58 to 301 °C. The lower temperature material was RT58 that was ordered from Rubitherm GmbH, which was also looked into when selecting our test materials. They did not offer encapsulation service meant for water tank, so they were left out of our tests. Test results for RT58 material can be used as a reference for the results of this work.

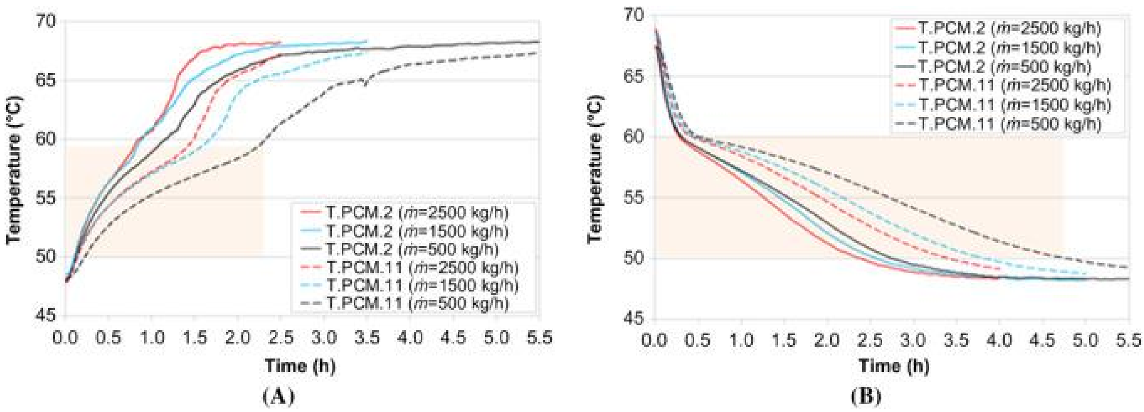

Figure 13 Temperature profiles for different flow rates using RT58 in Tank 1. Charging (A) and Discharging (B) (Gasia et al. 2018).

As seen in Figure 13 changing variable was flow rates of HTF, which can be seen in the temperature curves of the graph. Higher flow transfer energy from the system causes charging and discharging of the TES to be faster. HTF in this case was Syltherm 800 heat transfer fluid (DOW 2021). Unfortunately, there was no reference to measure the gained energy content by using PCMs.

University of South Australia's research regarding PCM was a more modeling-based study where prototype tube-and-shell was used to validate their ε-NTU numerical model (Tay et al. 2018). PCM used in the study was water to validate the model by freezing and melting water and in larger-scale tests, sodium nitrate was used which is salt-based material. Due to higher temperatures compared to our domestic hot water level temperatures, the results are not comparable. However, the temperature graph from the high-temperature PCM prototype is valuable in seeing how PCMs functionality can be seen. In Figure 14, PCM can be seen affecting the temperature of the air fed into the system. This test was performed with high-temperature equipment making the result not directly relatable to domestic hot water temperature tests.

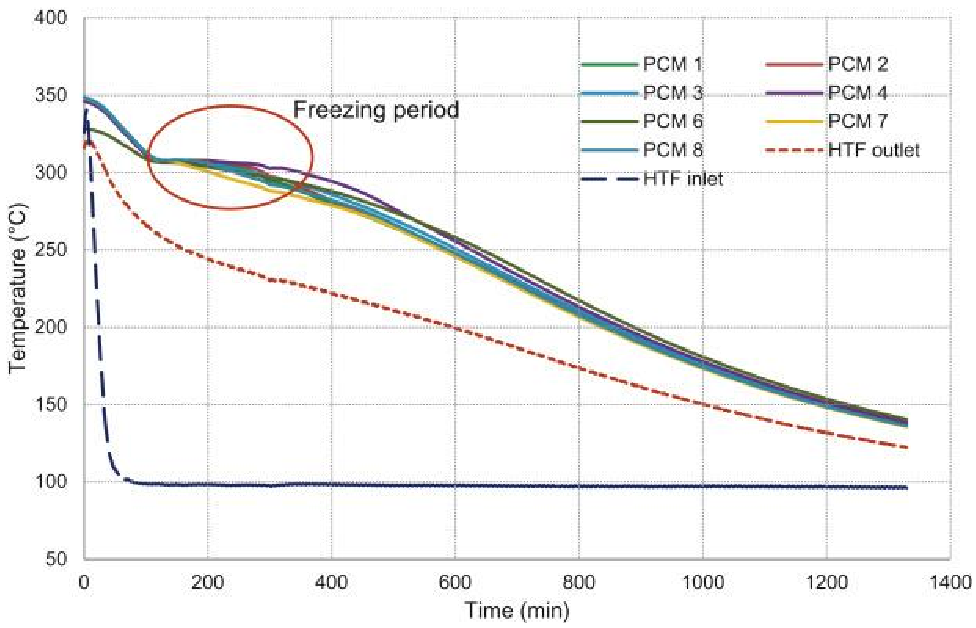

Figure 14 Temperature of PCM and HTF in time axis with air as HTF (0.01 kg/s) and inlet temperature of 100 °C (Tay et al. 2018)

Kamil Kaygusuz & Ahmet Sari had a study performed in 2005 where paraffin wax was tested as LHTES material (Kaygusuz and Sari 2005). Their test equipment was a tube-and-shell type heat storage (Figure 15) with one HTF pipe flowing through the center of the tank. Data collected were time, temperature, and flow rate with the focus being the melting, solidification, and performance of the PCM. Paraffin used had a melting temperature of 37.99 – 42.42 °C and latent heat of 163.2 kJ/kg.

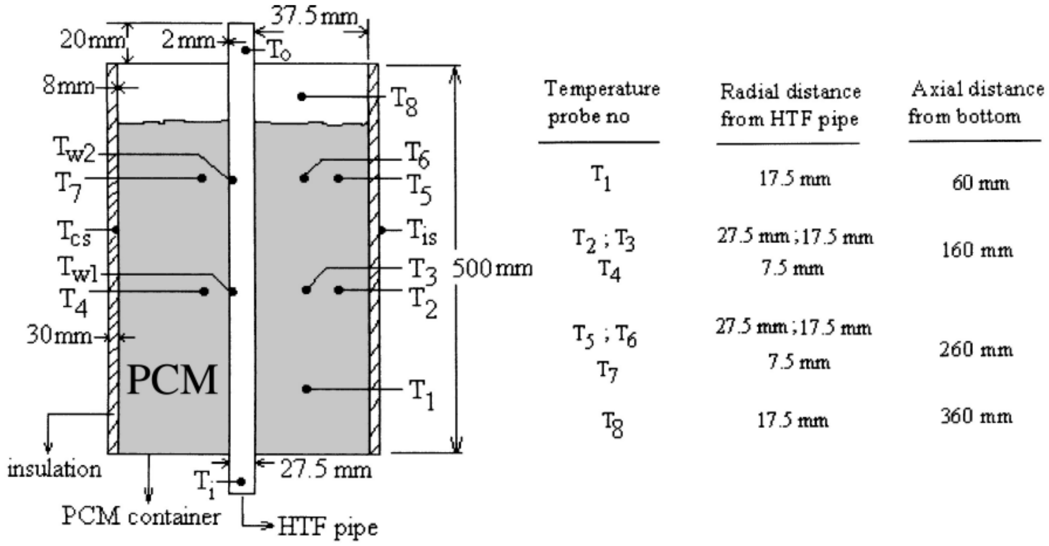

Figure 15 Diagram of heat storage unit used and temperature sensor locations (Kaygusuz and Sari 2005)

Paraffin wax they used show a good response for the temperature drop with little supercooling occurring. The focus was to see how different mass flow rates affect the melting of the PCM in the system. Energy calculations have not been performed nor has it been compared to SHTES with water.

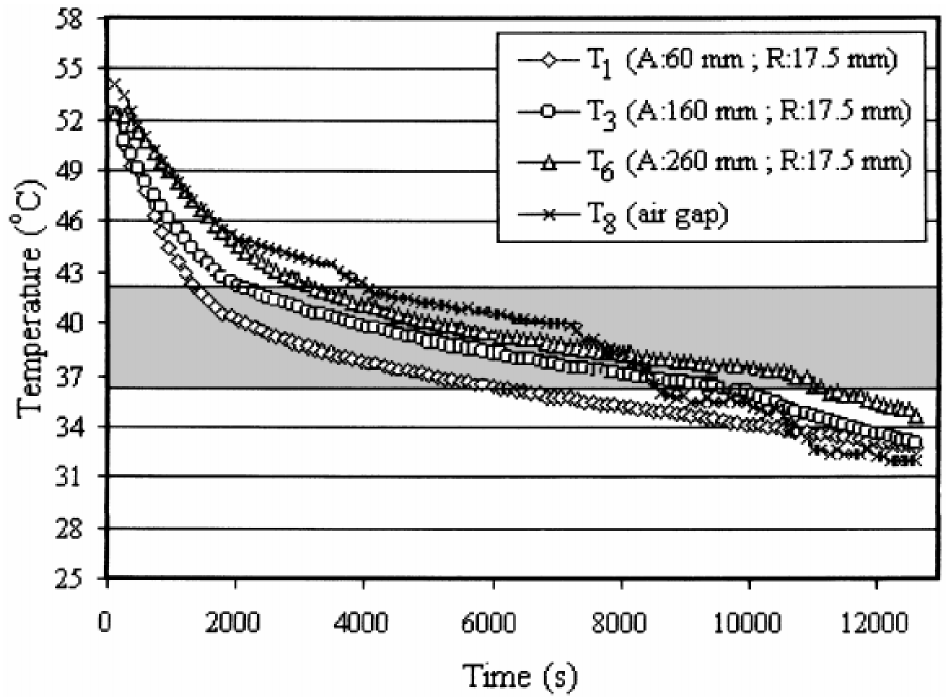

Figure 16 Temperatures measured in different radial distances from the HTF (water) pipe with an inlet temperature of 32 °C and mass flow of 0.016 kg/s (Kaygusuz and Sari 2005)

Tests for commercial materials have been done to some extend. For example, Porteiro J from the University of Vigo made a study regarding the behavior of different PCMs in a hot water storage tank in 2016 (Porteiro et al. 2016). These three commercial PCMs were tested in relatively small quantities to see how they fare against different thermal demands.

In 2019 Xu T. had research, where thermal storage was tested with macro encapsulated PCMs for purpose of using the energy for heating spaces (Xu et al. 2019). A larger mass of 154 kg of Climsel C48 was used capsulated in cylinders.

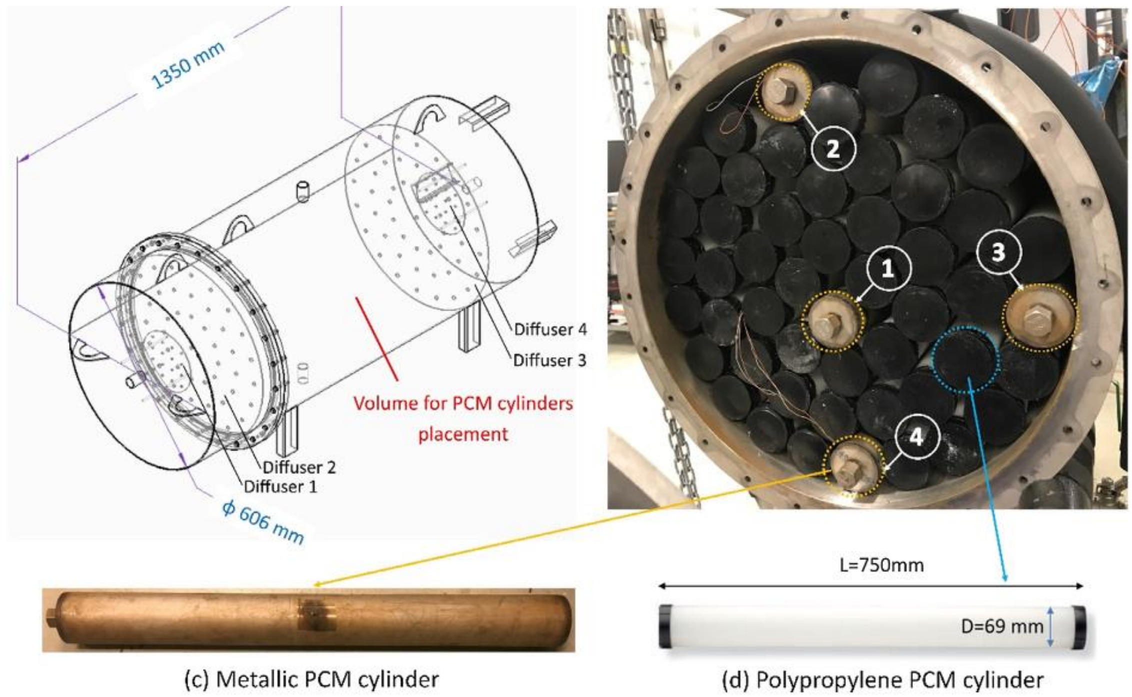

Figure 17 LHTES unit in Xu T. research (Xu et al. 2019).

In their research, the performance of PCMs in horizontal and vertical positions was looked into as well as the effects of temperature and HTF flow rates. 37 % of total storages volume was used by encapsulated PCMs. Steel cylinders were used to monitor the state of PCMs inside the containers. Steel containers increase the melting and solidifying time of PCM in their approximation by about 3 %. The vertical position of the tank creates a better flow distribution in the system and while charging the storage, increased temperature and flow decreases the time to charge as seen with temperatures in Figure 18. Respectively lowering the discharging temperature reduces the time to discharge the energy from storage. In their tests, it was noted that charging and discharging times reduce dramatically if only 75 % of calculated energy capacity is used instead of 99 %.

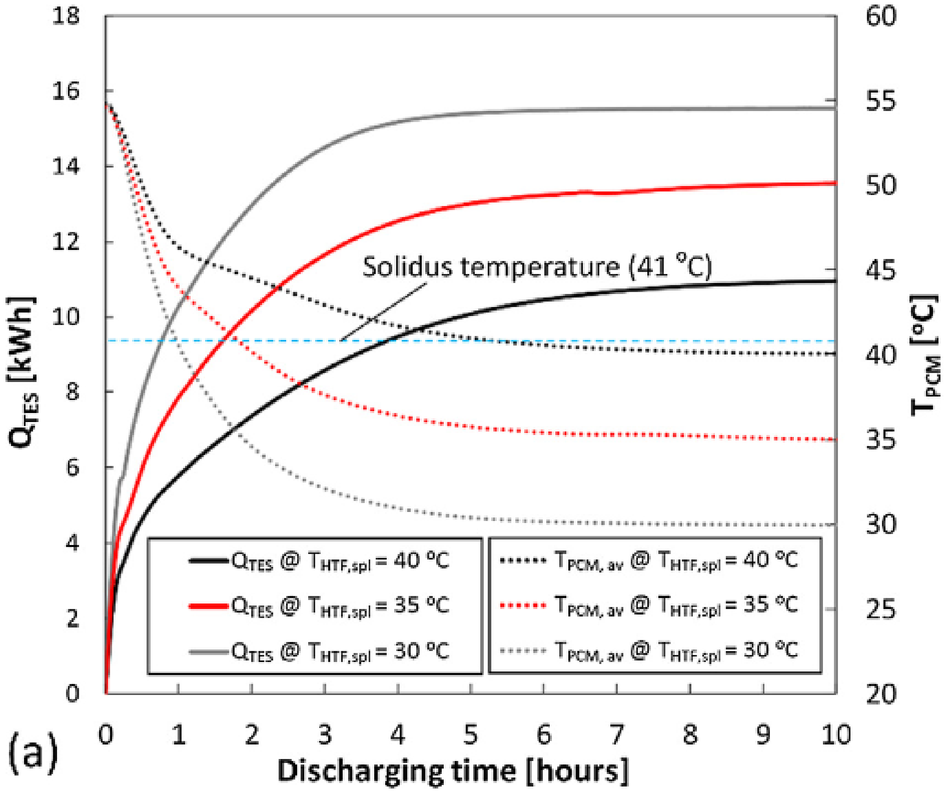

Figure 18 Discharging thermal energy in a vertical position with a steady flow rate of 1.5 m³/h. HTF input temperature varied until inlet and outlet temperatures reached equilibrium (Xu et al. 2019).

6 METHODOLOGY AND MATERIALS

Phase change materials have been utilized and tested since the 1800s (He and Setterwall 2002) without them getting more mainstream attention or usage. With the energy positive district project, it was possible to test these phase change materials and create a small test thermal energy storage, which would give insight into how it would perform with . Plenty of material exists in forms of studies and publishes, where this topic is discussed but few actual implementations with details of the technology or process are presented. Additionally, this project provided a good opportunity to get more familiar with PCM technology, where it is at on the TRL chart and to compare commercial producers what they are offering and how good are their encapsulation methods. Testing was to be conducted with flowing water acting as a heat transfer fluid (HTF) with a heating circuit working as a heat source and a cooling circuit being the load of the system where heat is transferred. Temperature range for the material needed to be between 55 – 60 °C. This specific temperature range was selected since a possible position for thermal storage was in a renovated apartment building in the new energy district at Kaukovainio. Hot water would be used for the apartment's domestic hot water. Regulations (1047/2017) set by the Ministry of Environment (FINLEX 2021) in Finland require that the water temperature needs to be always above 55 °C in the hot water line to prevent Legionella bacteria or the development of other organic matter. This meant that materials needed to be selected based on these restrictions. All the commercial producers for the encapsulated materials had a catalog for their materials for different temperatures. However, all of them had only one suitable material for the intended temperature level. Test materials were chosen as three for 58 °C levels and one organic material for 60 °C levels. The research was done following several thought-out research questions. The flowchart in Figure 19 presents the questions and methods for possible answers. From published papers and general information available on the internet, manufacturers were looked up. Some of the manufacturers that were mentioned had stopped production or companies did not exist anymore.

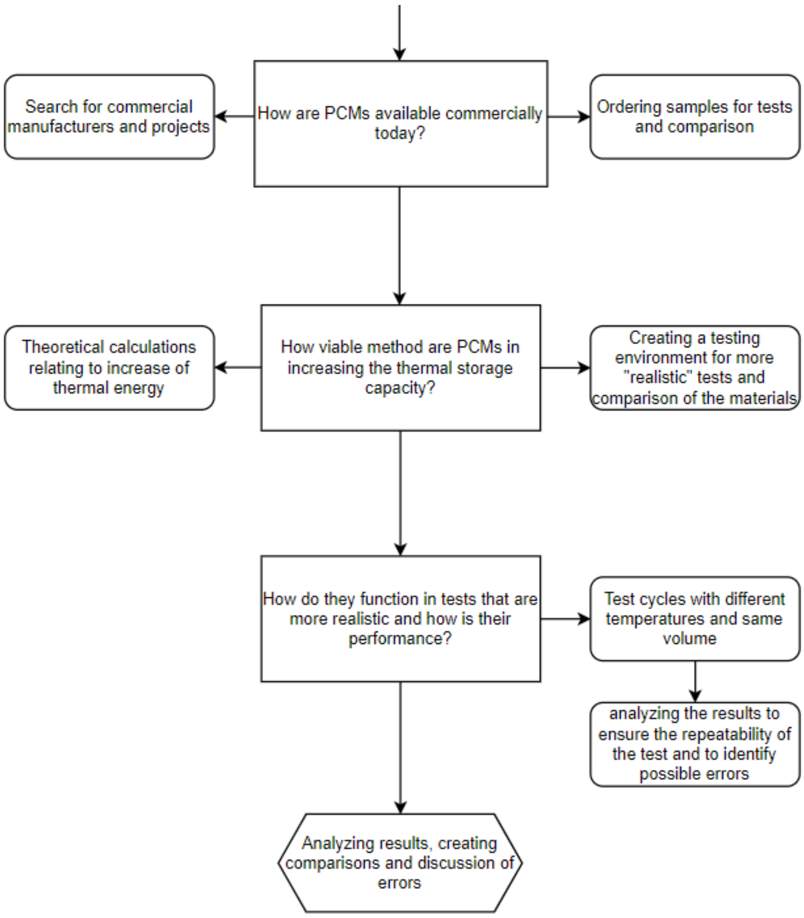

Figure 19 Flowchart for questions and methods

6.1 Theoretical calculations

Product sheets were downloaded from the product manufacturer's website that gave useful information and Eq. 2 and Eq. 6 were utilized for calculating the energy for latent heat storage. As mentioned, this does not consider the heat conductivity of the material meaning that output energy depending on the system's condition might differ.

Theoretical calculations for the added energy by PCM were calculated using the previously presented formula. There were four different test materials each with their properties as presented in Table 3. Temperatures for the tests are in four different levels, 70, 67.5, 65, and 62.5 °C with all having a minimum temperature of 40 °C meaning that the temperature difference will be between 30 and -22.5 °C. These temperatures were chosen to ensure that materials would melt properly. For all materials, manufacturers recommend a higher temperature for melting than the specifications mention. This is due to possible supercooling which means that material needs higher or lower temperature to initiate phase change. This can be referred to also as material hysteresis. Hysteresis is a sort of "latency" in the system but is usually referred to in magnetic fields but is used to describe lag in other systems (Britannica 2021).

Firstly, thermal energy for 30 liters of water is calculated without any PCMs added. Presented 30 liters come from the planned size of testing equipment. Calculated energy for liters of water for different temperatures is presented in Table 2. Eq. 2 is utilized for this calculation, where waters specific heat capacity 4.17 kJ/(kg·K) was used, which in reality does change in function of temperature but in this case, this is not taken into account due to small change of results. Specific density is 0.999 kg/liter, which also changes in different temperatures, but differences are small at this scale and are neglected.

Table 2 Thermal energy in 30 liters of water in different temperature differences

Temperature difference ΔT [°C]	Energy [kJ]
30	3745.5
27.5	3433.4
25	3121.3
22.5	2809.1

To compare PCM enhanced storage with water to storage with only water, simple calculations were made to estimate added energy. Using the previously discussed in Eq. 6 regarding the calculation of energy potential in PCM, with specs for materials obtained from manufacturers that are listed in Table 3.

Table 3 Properties of test PCM.

	Units	ATS 58	ClimSel C58	GAIA HS PCM58	ATP 60
Melting temperature	°C	56 - 58	58	58	57 - 60
Solidification temperature	°C	54 - 57	55	52	57 - 60
Latent heat energy	kJ/kg	240	260	226	230
Specific heat capacity	kJ/kg·K	3	-	2.5	2
Density (Solid)	kg/l	-	-	-	-
Density (Liquid)	kg/l	1.28	1.4	1.45	0.8
Heat conductivity: Solid	W/(m·K)	0.6	0.57	1.02	0.2
Heat conductivity: Liquid	W/(m·K)	-	0.47	0.54	-
Volume expansion	%	>6	-	-	>10

Energy for temperature differences was calculated for a 30-liter container with 2/3 of volume being water and 1/3 of being test PCM. This relation comes from the size of the testing equipment and the size of the encapsulated materials. A smaller amount of water was not possible and a larger volume requires more samples from companies. ½ relation would have been more nominal but this was not possible with the container used. Results are compiled in Table 4 accompanied by bar charts in Figure 20 with separated energy content for all substances.

Table 4 Theoretical energy contents with 20 liters of water and 10 liters of PCM

Temperature difference ΔT [°C]	Energy in Water [kJ]	ATS 58 [kJ]	ClimSel C58 [kJ]	ATP 60 [kJ]	GAIA HS 58 [kJ]
30	2502	4224	4480	2255	4364.5
27.5	2293.5	4128	4410	2214	4273.9
25	2085	4032	4340	2173	4183.3
22.5	1876.5	3936	4270	2132	4092.6

From these calculations, it can be seen that energy content increases at least by 40 % with salt-based materials that are ATS 58, C58, and GAIA HS 58. With organic-based ATP 60 increase is around 20 to 30 % depending on maximum temperature. These values can be seen in Table 5.

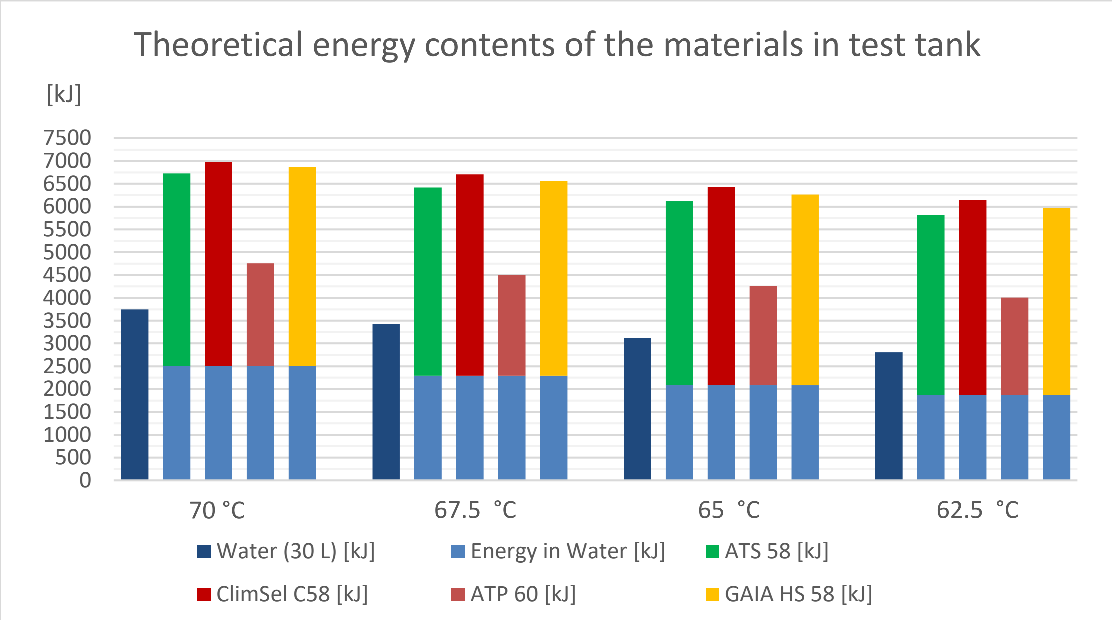

Figure 20 Theoretical energy contents for each material with water's energy as a reference

From a theoretical point of view, there is potential in increasing the thermal energy content of storage using these materials. These calculations however did not consider possible poor thermal conduction properties of the material during storing and releasing of energy.

Table 5 Theoretical % increase in thermal energy compared to only water (30 liters)

Max Temperature [°C]	ATS 58	ClimSel C58	GAIA HS 58	ATP 60
70	44 %	46 %	45 %	21 %
67.5	47 %	49 %	48 %	24 %
65	49 %	51 %	50 %	27 %
62.5	52 %	54 %	53 %	30 %

6.2 Testing protocol

Different methods for testing the materials were looked at in the papers to give some insight on how they have been tested and what look forward to. Most of the testing in papers focused on heat conduction properties of the materials and in small-scale testing equipment where heat conduction can be monitored. On the other hand, few of the papers focused on the functionality of materials, and none compared materials to each other in a consistent way and there were not found examples of detailed real applications of the PCMs in thermal storage.

Since chosen temperature range was 55 – 60 °C the testing cycles were set to be 70, 67.5, 65, and 62.5 °C. Even though materials had a melting temperature of about 57 °C, the higher temperature was used to ensure the proper melting of the material. To ensure the repeatability of the test cycles, each temperature got three heating and cooling cycles except for the first cycle getting one extra to ensure that the remaining three cycles are near identical as seen in Table 6. The most important aspect of PCMs was temperature measurements. If heating and cooling power are known, then measuring the temperatures as a function of time gives a better picture of the properties of the materials. Since the materials have their specified melting temperatures given it was also interesting to see if energy storing and releasing temperature changed when maximum temperature was changed.

In theory, it seems that there are some possibilities of using these materials but in the end, the price of the materials, their functionality and lifetime dictates how economically feasible it is.

Table 6 Test plan for the materials.

		No. Of cycles in test temperatures			
Volume (ratio)	Material	70 °C	67.5 °C	65 °C	62.5 °C
1	Water	4	3	3	3
1/3	ATS58	4	3	3	3
1/3	ClimSel C58	4	3	3	3
1/3	GAIA HS 58	4	3	3	3
1/3	ATP60	4	3	3	3

Multiple temperature sensors are used to measure data from the water tank to reduce possible measured error in 30-second intervals. Heating and cooling cycles are automated using computer software that uses the data from temperature sensors to control the system.

6.3 Materials and their preparation

Since materials are from different producers and they mostly have their method of encapsulating the material. To ensure that tests are consistent and comparable the volume of material needs to be the same in all tests for each material. These steps were done to each material at the start of the test to ensure that consistency stays.

First weighing capsules to see their weight and this also works as a check-up that capsules are intact and there are no observable problems. Then calculating and measuring the volume of one capsule. An effective method to measure the volume is to use a water measuring dish and measure the volume of water that the capsule displaces. Weighing pieces and seeing their weight distribution also indicates the quality of encapsulation.

The first set of materials were obtained from Germany, Netherlands, Sweden, and United Kingdom. Properties for all the materials were presented in Table 3 with theoretical calculations. Climator Sweden AB in Sweden was the first one to ship a small number of samples to get to know their product. Their product of ClimSel C58 was packaged in an aluminum shell as seen in Figure 21. An aluminum foil-like shell allows the material to expand more freely and most likely acts to increase its thermal conductivity.

Axiotherm GmbH provided samples of ATS 58, an inorganic PCM with capsulation to plastic shell in form of disc and stick. This material was packed into their HeatStixx™ form. Global-E-Systems B.V. shipped their encapsulated products of GAIA HS PCM 58, which was capsulated in plastics spheres Figure 21. Spheres seemed to be sealed by plastic welding the cap shut. Croda LCD provided organic-based PCM that had a working range of 60 °C. They did not provide encapsulation themselves but instead, they had a collaboration with Axiotherm that encapsulated the material for them meaning that encapsulation is the same as previously mentioned ATS 58 had. The material was packed in the same HeatStixx™ as seen in Figure 21 below. A measuring tape is placed as a reference for the size in Figure 21.

Figure 21 Acquired materials for testing with measurement tape for scale. Materials from left to right: Axiotherm HeatStixx capsule (ATS58 and ATP60), Global-E-System B.V. Gaia HS PCM58, and Climator AB ClimSel C58.

To test the properties and functionality of encapsulated PCMs a testing environment was needed. This required some planning on what kind of equipment was necessary for the test. The first idea was to use a laboratory hot water bath to determine the energy contents of the materials by measuring the water temperature when water in the bath cooled down. There was an issue with utilizing the hot water bath since cooling of water was a very slow process meaning that it would take a long time for the test cycles and it did not show materials working in a more realistic environment. For these reasons, controlled cooling was needed. It was decided that a controlled heater and cooler were needed for the test as well as a tank for water. The idea was to keep it relatively simple and clean, to see the functionalities of the materials with temperature measurements in a function of time.

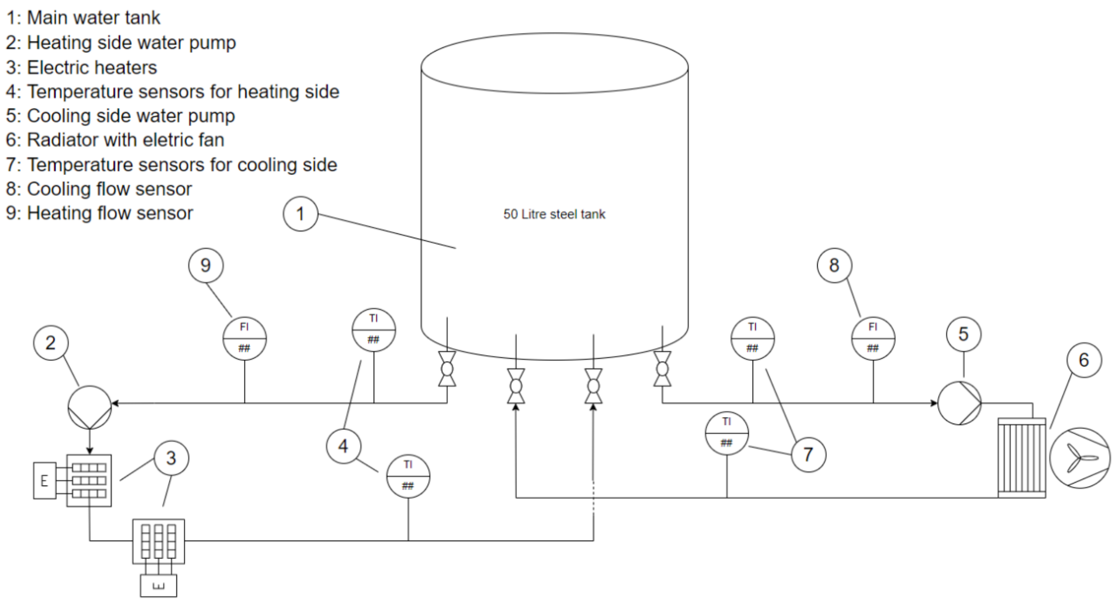

Figure 22 Testing equipment diagram.

The water tank is cylinder-shaped with a diameter of 395 mm and height of 440 mm and it is made of stainless steel with the bottom made of 5 mm thick basic steel. In the tank, there are 8 outputs/inputs with valves and a fitting for a 19 mm diameter hose. There is also one additional smaller output for easier emptying of the system in the bottom level of the tank. Inlets for the tank are installed to have four in the lower part of the tank and the other 4 in the upper section of the tank. This gives the option to have cross-flow in the tank if a higher water level is utilized. The maximum amount of water for a full tank is 50 liters. Despite outputs and inputs being near the bottom of the tank water stratification is not an issue due to the relatively small size of the tank.

For the piping of the system, a 19 mm radiator rubber hose was used since it can fit most of the valves, pumps, or radiators that would be used. In addition, it is easy to work with and is available easily. For turns and lengthening needs, 90-degree turns, T-sections, and extensions made of plastics were used with hose clamps. Heating was done by using heating coils made for an engine's cooling system that did not have an internal thermostat to have full control over the heating process. Heaters are made by DEFA with a heating power of 550 W. Two of these kinds of heaters were used to have total heating power of around 1100 W as seen in figure 9. Fluid circulation was done with a 12V centrifugal circulation pump in each circuit.

Figure 23 Heating with two 550W heaters and pump. Heaters have overheating protection on them.

Cooling was handled with a small aluminum radiator, two 12V system fans, and a 12V water pump. Tests were done with only one fan in operation to prevent cooling power from being too effective. The reduction of cooling power was made due to the low heat conduction properties of PCM. Meaning that too high cooling power might make PCMs thermal energy release less noticeable in data.

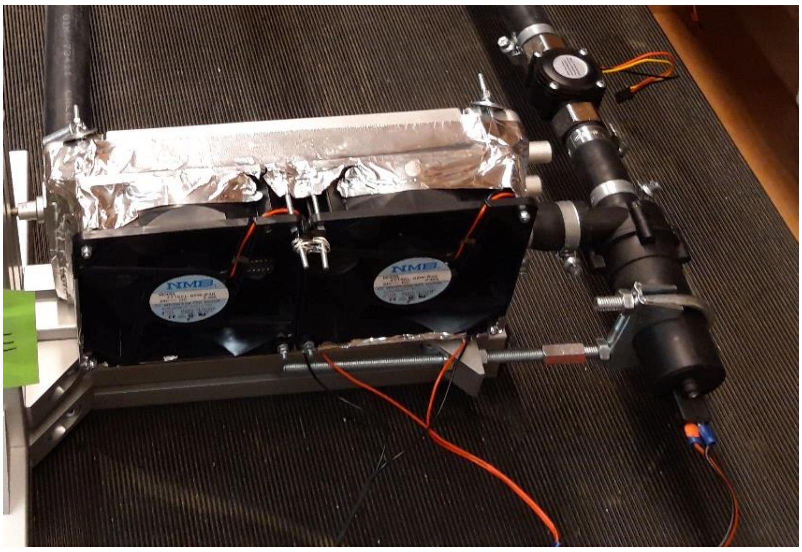

Figure 24 Cooling radiator with fans, flow sensor, and 12V water pump.

The Control system was done by using remote Z-wave sockets since all the pumps and fans drew power from electric sockets. The use of Z-wave sockets was possible since there was an extensive amount of them available due to relation to another ongoing project. Z-wave (SmartHome 2021) is a control protocol that uses low energy radio waves at 863.42 MHz in Europe to help various smart appliances communicate with each other. It was first developed in Denmark by Zensys in 2001 until released in 2004. Z-wave uses very little energy to work and its communication is always two-way, meaning that it confirms every signal send and received making it a very reliable system. Z-wave is also a standardized protocol, which means that integration is easy to various devices through standard language. Z-wave devices utilize meshing when sending signals meaning that each device looks out for other devices in the vicinity that creates an efficient way to pass commands and information. Signal jumps from device to device and the number of these 'hops' is limited to 4 from each device which keeps the transfer of signals clearer. Due to their reliability and simplicity to use, they were to be utilized in this task. Raspberry Pi 3 (Figure 25) was acquired to function as a signal transmitter with added Z-wave RaZberry module. Raspberry Pi is a credit-card-sized computer developed and made in the UK. Its design started in the University of Cambridge's Computer Laboratory in 2006 for purpose of educating students about computing and programming (Packt 2021).

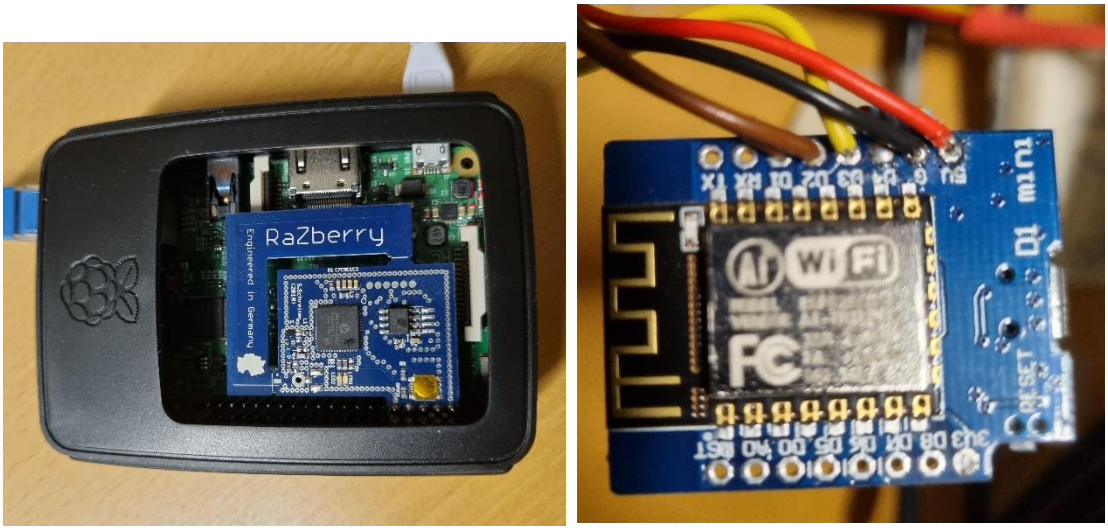

Figure 25 Raspberry Pi 3 and ESP8266 chip.

For temperature measurements, DS18B20 digital temperature sensors from Sparkfun were used, which provides 9 to 12-bit temperature readings (Semiconductor Corp). These sensors work with a 1-Wire interface meaning that only one wire additionally with a ground needs to be connected. Data is read and written from a single data line and it can alternatively draw power from it. Power can also be supplied externally by a 3 - 5.5-volt supply. Since the sensor has a unique silicon serial number, multiple sensors can be placed on the same 1-Wire bus and each sensor can be identified by the computer. Their working temperature is reported by the manufacturer to be from -55 to 125 °C and between -10 °C to 85 °C with an accuracy of ±0.5 °C. Data can be obtained from sensors every 750 ms or at a slower frequency. In this case, data is read every 30 seconds from sensors and 12-bit resolution is used to read data. Sensors are also waterproof meaning that they are ideal for measurements underwater. For both the cooling and warming side, temperature sensors were added to the inlet and outlet to measure temperature differences before and after. Additionally, both circuits were equipped with a simple centrifugal flow sensor with pulse measurement (Figure 26). This sensor was made by Seeedstudio, which produces all kinds of small and medium IoT equipment. The sensor was easy to set up with the temperature sensors. Data from the flow sensor is read with the same 30-second interval as temperature sensors.

Figure 26 Flowsensor (left) and Z-Wave socket

Sensors are connected to the ESP8266 chip (Figure 25) that acts as reader and uploader of data as read data is uploaded to cloud server through Wi-Fi in the testing room. Cloud service is provided by Thingspeak (ThingSpeak 2021) that is an IoT analytics platform, which makes data visualization easy, allows data storage and a quick simple download possibility for data in CSV file form. Raspberry Pi accesses the Thingspeak cloud and reads the temperature of the water in the tank. Based on the wanted temperatures, Raspberry Pi operates the Z-wave sockets that control the pumps, heaters, and cooler fans. Python is used as a coding language for the sensors and Raspberry Pi because it is open source, and it is very compatible with other languages.

7 RESULTS

For finding a suitable material for the testing and the possible future larger prototype, a search for commercial materials was done and multiple companies were found that provided PCMs with encapsulation for a higher temperature range of 30 – 85 °C with the addition of some low-temperature level PCMs as well. Data was imported from the cloud server, analyzed for possible errors, and averaged. Overall, all the materials showed some change in the slope of cooling and heating with other materials performing better than others. For these tests, the cooling phenomenon was more interesting to monitor since the usual release of heat is more crucial in thermal storage for the feasibility of the system. However, in more intensive usage, charging speed can not be left out. These aspects were interesting since PCMs have usually low heat conduction properties.

7.1 Commercial availability of PCMs

The main source for phase change material came first from published research papers and some of the manufacturers mentioned were no longer in operation. After more search, it turned out that there are still multiple companies that are researching and producing materials for the construction, pharmaceutical, food industry, and energy sector. The highest interest for companies seemed to be in the construction, food, and pharmaceutical sectors. This is due to PCM's good properties in stabilizing temperatures. They can be used in transportation tasks that require low, stable temperature for preservation, and in the construction business, PCMs are incorporated into wall or ceiling elements. These solutions are regarded as passive methods where the material is used to control the temperature without active energy transfer methods. Higher temperature ranges that could be used in hot water containers in domestic or larger industrial applications seem to be still uncommon with more scare temperature ranges. One of the best methods for utilization of PCM is the encapsulation of the material, which can be likely reason for the small number of suppliers since encapsulation requires more advanced methods for effective encapsulation. Companies that do offer these higher temperature materials most have a way to provide encapsulation for the material. Overall, PCM applications have increased in recent years (Pielichowska and Pielichowski 2014). PCMs have also been thought to increase the efficiency of PV panels by incorporating PCM into them, which keeps temperature levels lower thus preventing efficiency lowering due to the high temperature of the panel. This same idea can also function with electronics since some devices may require temperature control that could be achieved by applying PCMs to it. Development of PCM usage depends highly on the price range of the materials and plays a key role in regards to generalizing the product with addition to the proper functionality of materials. Due to the need for more precise energy control, PCMs might get more attention in the building sector to increase thermal energy mass in apartments in form of low-temperature materials in walls for example, or high-temperature materials in water storages to allow more control over temperature.

7.2 Their functionality and performance

The purpose of making the test equipment relatively simple was to see if the materials function as they are marketed. With added PCM, it was expected to see how material releases thermal energy into the HTF. All materials showed an increase in thermal energy, which can be observed as a longer cooling time, down to selected 40 °C. After running the tests and visually confirming the repeatability of the tests, the data needed to be filtered and handled properly to reduce errors and ensure the quality of data. During the data analysis, possible errors in sensors were looked for. First Figure 27 presents water test cycles. These act as a base to which compare the water with added PCM.

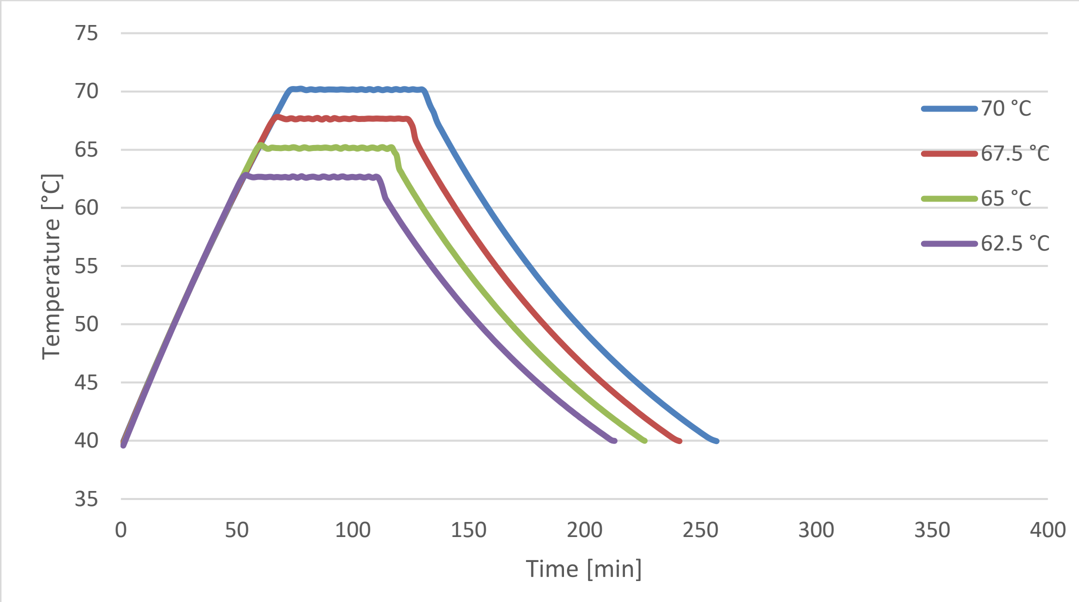

Figure 27 Temperature curves in the function of time for water

Tests with water were functioning as expected with results showing smooth temperature curves which imply successful measuring. Next are temperature curves with water that has 1/3 of volume PCM.

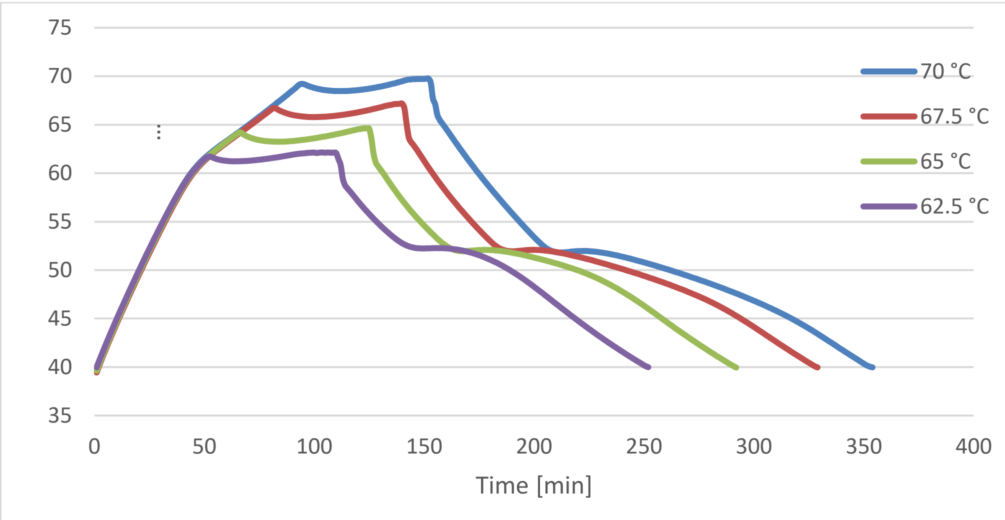

Figure 28 ATS58 PCM

Material ATS58 (Figure 28) showed the strongest thermal energy release from material and momentarily it warmed up the water halting the temperature drop. Material can also be seen absorbing the energy from water during the heating period. The effect can be seen reduced at lower temperature levels at 65 °C and 62.5 °C levels.

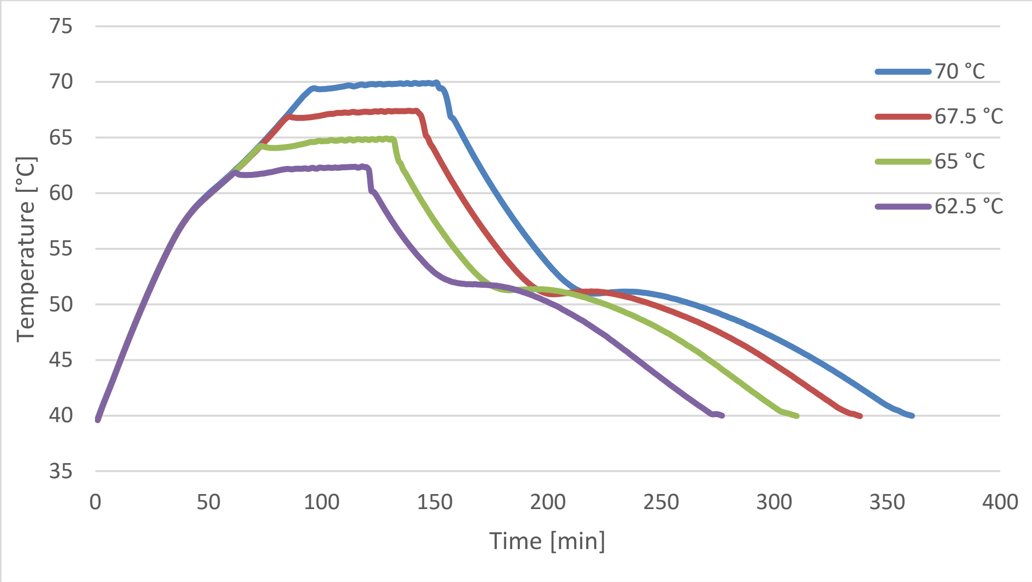

Figure 29 ClimSel C58

The second-best material with similar properties to ATS58 was Swedish C58 (Figure 29). In the same manner, C58 releases thermal energy strongly creating a visually easy-to-see effect that lengthens the cooling time. This material's ability to absorb and release thermal energy is slightly better than ATS58 at lower temperature levels. ATP60 material (Figure 30) showed the least thermal energy capacity but it still showed increased thermal capacity. GAIA HS58 material leaked capsules causing the test to fail. The system had to be cleaned and refilled.

Overall, all materials showed a positive change in the heating and cooling graphs when compared to only water meaning that there is some increased thermal capacity present.

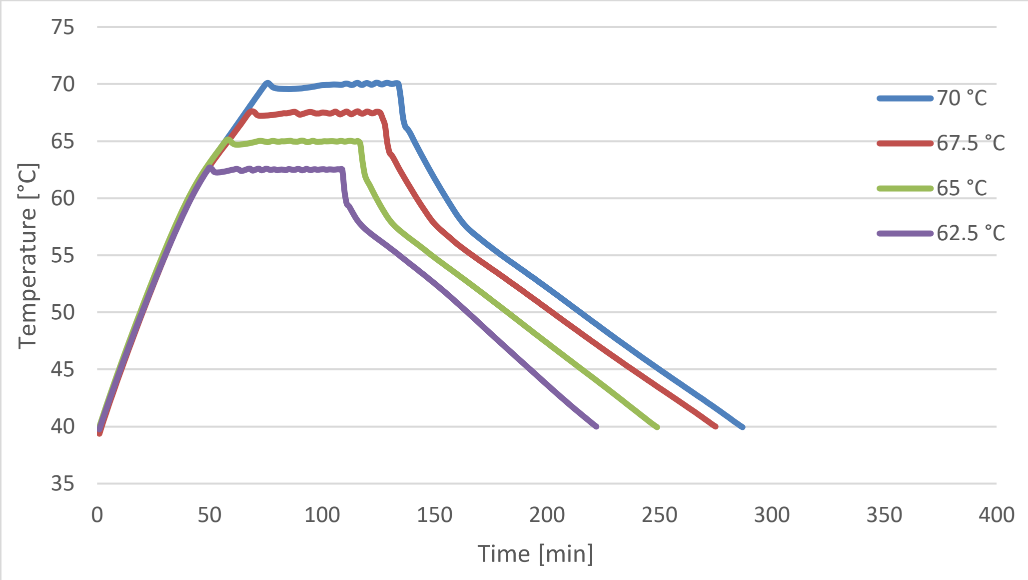

Figure 30 ATP60 PCM

7.3 Increasing the thermal storage capacity of PCMs

From the control test that had only water, the power of cooling was calculated since the properties of water are known. Once the material is added to the system it can be assumed that cooling power stays the same. Thus, by using the calculated cooling power, thermal energy released from the material can be calculated. Previously, theoretical calculations were done to give an idea of the amount of energy that can be expected from the materials. Comparisons of these energy values are imperative in analyzing the viability and effectiveness of the materials.

Energy content was calculated using the cooling side of the cycles as the measurement data was consistent. A water test was used to calculate the power of the cooler since water properties are known and test data was well collected.

For the water, energy was calculated theoretically, and then using the measured temperature data with time it was confirmed that energy contents are almost identical with some deviation as seen in Table 7, meaning that energy contents are valid.

Table 7 Comparison of calculated energy in water with temperature differences (down to 40 °C) and theoretical energy

Temperature	70 °C	67.5 °C	65 °C	62.5 °C
Average energy from test [kJ]	3752.7	3434.4	3125.9	2821.6
Theoretical [kJ]	3745.5	3433.4	3121.2	2809.1
Average deviation	3.6	0.5	2.4	6.2

From the water test, calculating the power of the cooling system was straightforward. With Eq. 2 energy can be calculated since the mass and temperature difference of the system is known and to convert energy into power it is divided by 3600 to change time to an hour. The resulting unit of [kWh] is divided by the measured time in hours it took for the water to cool down to the setpoint of 40 °C. Calculated from data, the cooling system's power is about an average of 0.48 kW through four different temperature levels. Power differentiates a little bit between temperature levels and this cooling power is assumed to stay the same during testing of the PCMs since flow speed and HTF are the same. Next, new energy contents could be calculated for the materials and gathered into Table 8.

Table 8 Calculated energy content of materials from tests in different temperatures and energy increase in percentage compared to water.

	Materials and energy content [kJ]		
Temperature	ATS58	C58	ATP60
70 °C	5975.0	6080.4	4525.6
67.5 °C	5528.8	5482.0	4358.7
65 °C	4781.8	4840.8	3785.6
62.5 °C	3901.6	4262.3	3083.1

	Increase of energy %		
Temperature	ATS58	C58	ATP60
70 °C	37 %	38 %	17 %
67.5 °C	38 %	37 %	21 %
65 °C	35 %	36 %	18 %
62.5 °C	28 %	34 %	9 %

These energies can be compared to water and to the theoretical energy content that was calculated earlier in Table 2 and Table 4. Figure 33 has cooling temperature curves for all three materials along with water base test.

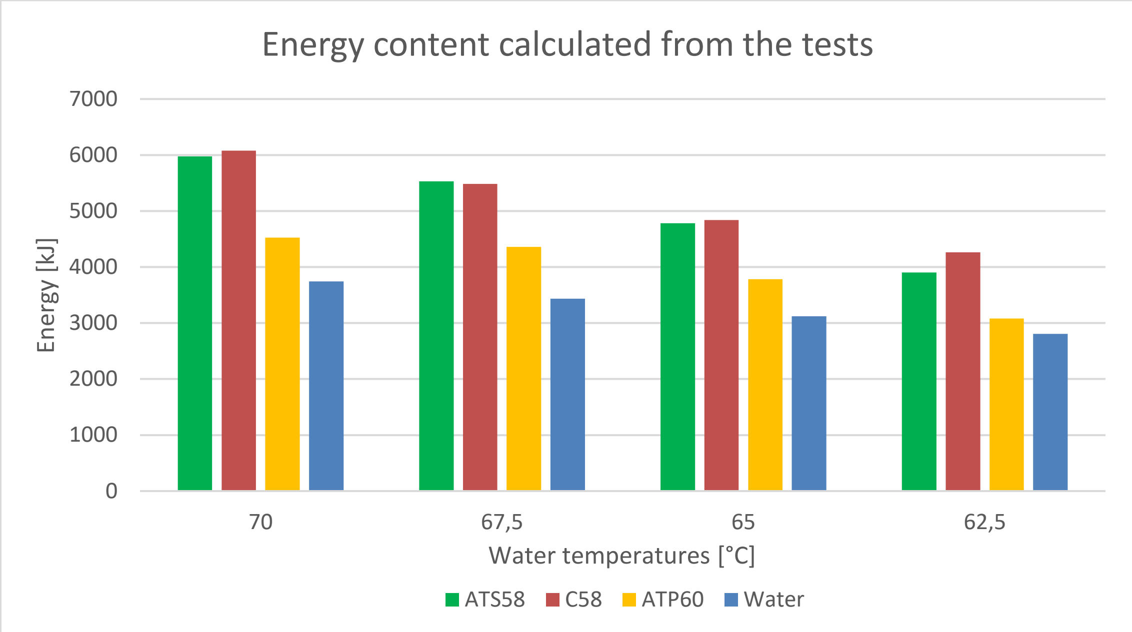

Figure 31 Calculated energy contents from test data using the cooling power.

Closer inspection of ATS58 material is done in the discussion part where its functionality is analyzed, and possible errors are mentioned and thought of. Figure 32 has comparison data regarding ATS58 enhanced storage versus water storage. Finally, from 70 °C tests, all cooling temperature curves are plotted next to each other in Figure 33 to show the differences between materials.

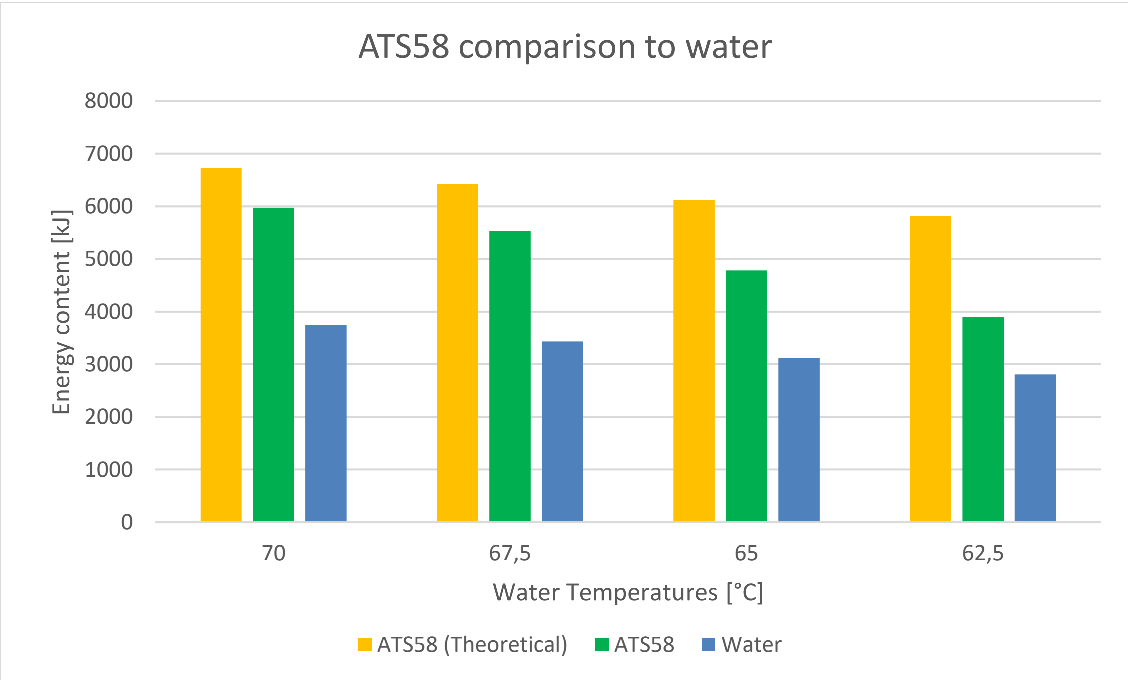

Figure 32 ATS58 material comparison

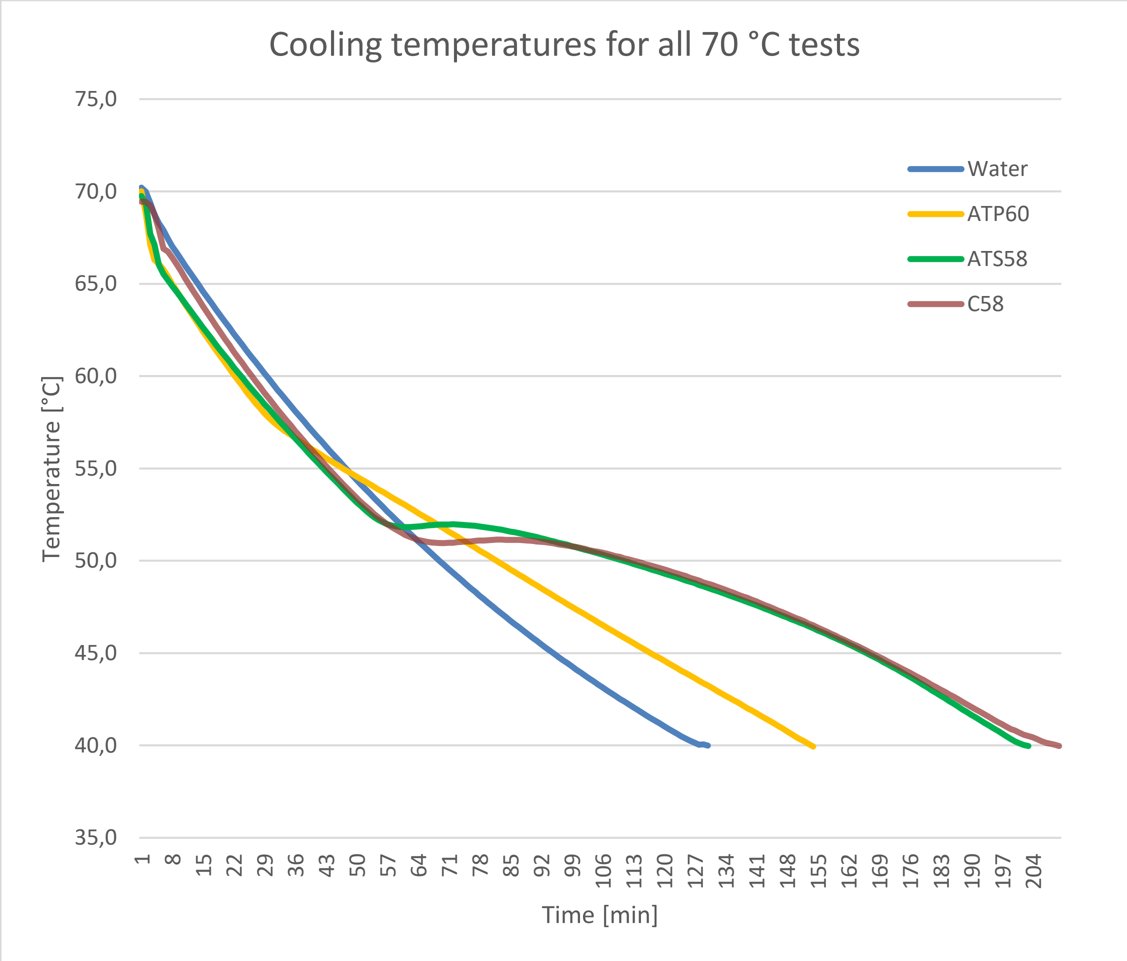

Figure 33 Averaged temperatures during the cooling period for all 70 °C tests from 70 to 40 °C.

8 DISCUSSION

Experiments went well and readable data was successfully extracted excluding the GAIA HS PCM58 test. Companies were cooperative with selling test materials but building a test environment had its challenges.

8.1 Testing environment and data gathering

Creating the test environment started with a simple vision of creating a model of hot water storage on a smaller scale. This however did not mean a dynamically scaled-down model of thermal storage, more like a small-scale tank where materials could be tested. Engine cooling system parts that were chosen were easy to acquire, provided guaranteed functionality with hot water, and were controllable using the Z-wave sockets. Flow sensors were installed but they provided only an approximation of ongoing flow rates. Since the temperature was being measured as a function of time and volumes were known the flow rate was not needed for the calculations. During the tests, some temperature sensors broke causing errors in the initial tests. Fortunately, sensors that malfunctioned did that at the start of testing meaning that after replacing broken ones, the remaining sensors stayed operational throughout the test cycles. At the start of cooling cycles, there is a drop in temperature due to cold water that is in the cooling pipe starts flowing to the water tank causing a drop in temperature. This drop has been taken off from cooling power calculations and it does not affect the PCMs. To remove this temperature decrease would have been possible by circulating cold side water at the same time as heating pumps to even the temperatures in hoses throughout the test.

When calculating the power of the cooler and increase of energy content, heat losses were not taken into account. This is due to fact that the latent heat phenomenon that is being inspected is located in the water tank and is not greatly affected by the heat losses in the system. Additionally, heat losses are included in the cooler’s power calculation since it was calculated with the change of temperature in function of time. Heat losses could have been calculated by using the dimensions of the tank and the pipes, but this was not considered to be the most important aspect of the study. Heat losses are more significant in higher temperatures and with longer storage times. The main water tank was isolated with foamed plastic which was the most crucial part of the isolation. Rubber water hoses were not isolated. Since the tank that holds the water was round cylinder type it caused some minor fitting problems with some testing materials due to some of the capsule's size. Tests were performed in 30-liter volume and especially Axiotherm's long plastic capsules were submerged 95 % of the whole surface. Choosing a larger volume with the same proportions could have been easier in terms of handling the capsules. Different volume sizes between capsules also caused some errors in test volumes since it was impossible to have the exact volume of 10 liter PCM with different capsules. These volume errors were small, and the scope of the tests can be neglected.

8.2 The functionality of PCM compared to water-only

The reason for these tests was to see PCMs function as intended in increasing the energy content of hot water tanks using forced cooling with temperature measurements. This was a largely successful test as three materials showed this behavior with ATS58 and ClimSel C58 showing the most drastic phase change event in the temperature curve. This event occurred at a lower temperature than mentioned in the technical specifications of the products. This can be due to the relatively high flow rate in the system meaning that start of the phase change when it releases thermal energy is unnoticeable until the temperature difference between water and material increases enough. It would be feasible to experiment with different flow rates with accurate measuring instruments. Different flow rates were left out of the test scope since the number of testing cycles increases making the tests more time-consuming. Since all successfully tested materials showed properties of increasing thermal capacity of the system, it could be said that with lower flow rates the release of thermal energy from materials could be possibly be noticed more clearly. Another possible reason for the lower temperature solidification can be supercooling of the material. Supercooling phenomenon absorbs energy before the solidification process starts meaning that there can be "lag" before the material starts to warm the water around it. This would lead to a seemingly lower working temperature.

ATS58 material along with the ClimSel C58 showed the most promising results. When compared to using water alone as a thermal storage medium, ATS58 theoretically increased the thermal storage capacity of storage by 44 % to 52 % (Table 5). When tests were done and increased energy content was calculated using the cooling power, the resulting energy increase was 28 % to 37 % (Table 8 and Figure 32). After seeing the results with ATS58 as well as C58 it can be said that increase of energy was achieved in the test storage. Energy increase however did not reach the calculated theoretical amount. The most likely reasons for this are the high flow rate and cooling power compared to the heat conduction properties of the material. There is likely energy left in the PCM capsules after reaching the 40 °C target temperature. It can also be possible that materials do not melt properly during the one-hour upkeep time. The reason is probably in the middle of these two alternatives and could be inspected with alterations to the testing properties and sequences such as lower flow rates and longer heating upkeep time before discharging thermal energy.

Gaia HS PCM58 from Global-E-System company is left out of the results due to leakage of the materials. Gaia HS PCM58 material seemed to have the most promising capsulation at the beginning. This was due to the spherical shape of the capsules which could be the most effective shape from a flow point of view and stacking of the capsules would be easier. The material used for the capsules was high-density polyethylene (HDPE) plastic with a filling inlet closed with welded "cap". Unlike other materials that sank when placed in the water, Gaia spheres floated requiring a plex plate that held them underwater for the duration of the test. After capsules were through halfway mark, collected data in the cloud shown very little energy storing capacity and inspection revealed that contents of the spheres had leaked into the tank. This required cleaning of the tank and spheres and since the material was inorganic salt-based it caused a large amount of corrosion in the system. Later this required change of heaters to new ones due to extensive corrosion. This demonstrated one of the weak points of inorganic materials that are highly corrosive properties. If leakage happens in a larger scale system, it can cause large complications and costly maintenance. After the tests and during writing this thesis they launch a new encapsulation method for their materials using stainless steel. These capsules might be reliable, and they also provide better thermal conduction than plastic however their lifetime is unknown since most of the materials have thermal expansion properties meaning that metal can be subjected to stress in the long term leading to breaching of the capsules.

As the capsule failure with GAIA HS PCM58 showed that it is important that capsules are durable and robust. This is imperative regarding the usability of the materials since it does not matter how good the material functions if their capsules break in a short time causing possible expensive maintenance. Axiotherm's capsulation seemed most highly developed and meant for work inside thermal water storage. Climator's ClimSel functioned as intended during the tests, but the aluminum sheet package's lifetime is unknown.

Higher energy content in TES can in the future increase flexibility with peak-shifting properties of smart apartments or districts. When utilizing waste heat in buildings it is important to have the ability to store excess heat if it exists to make full use of energy later with e.g. heat pumps. Sometimes due to space limitations, it can be beneficial to use encapsulated materials to enhance energy capacity if their commercial production increases making them more affordable and reliable. As more control over the usage of energy is wanted, effective means to store thermal energy are needed to achieve effective methods to "peak shift" especially in colder climates.

8.3 Results in light of previous research

Similar research where different materials with the same working temperature ranges have not been found, but a lot of research regarding the PCMs has been done. In one research done by the previously mentioned University of Lleida (Gasia et al. 2018), PCMs were tested in compact latent heat storage with one material being from Rubitherm GmbH RT58 organic paraffin material which has a working temperature of 53 - 59 °C. This material is not produced by Rubitherm (Rubitherm 2021) anymore. From their test with the PCM charging and discharging temperature curves are comparable with our temperature curves (Figure 13, Figure 16, and Figure 14). However, in their study variable was flow rate instead of different temperatures and it can be seen how the discharge curve changes as the flow rate lower. The flow rate used in the test was around 12.5 l/min, which rounds to about 735 kg/h mass flow rate. As seen in Figure 13 test's mass flow rate is between 500 and 1500 kg/h showing a similar discharge curve as in tests with ATS58, ATP60, and C58. However, gained energy benefits are not estimated when compared to conventional SHTES working with water as a storage medium. Figure 16 also shows a discharging curve in tube-and-shell type testing equipment with similarity to our measured temperature curves. Kaygusuz K and Sari A (Kaygusuz and Sari 2005) in their research have a discharge curve of LHTES with almost no supercooling, which can be explained possibly due to a slow flow rate of 0.016 kg/s compared to the used ~0.208 kg/s flow rate. In Xu's research where macro-encapsulated materials were tested, the temperature curves from discharging energy from the vertical tank are in similar shape

(Figure 18). The testing method also involved some similarities, for example, the charging period was long to ensure complete melting of PCM.

8.4 Technological readiness level (of the tested PCM)

TRL (Figure 11) for PCMs can be considered to be in levels 6,7 and 8 depending on the viewpoint and manufacturer. For the performed test, a level can be thought to be in the area of 6 where technology is being demonstrated in a working environment. However, when reading more about the PCM products it can be seen that it is being used commercially meaning that TRL is in level 8 or even in level 9 where the system is proven to function where competitive manufacturing is present. Though technology is on the usable levels on the TRL chart it is unclear how functionality is kept for a longer time. However, looking into the manufacturers made it clear that interest in higher commercialization is existing due to changes and restrictions to reduce GHG in the world. The energy market is changing towards RES meaning that Power-To-Heat technologies, as well as other P2X methods, will be utilized more to consume excess production.

8.5 Uncertainty and errors

In everything that is done exists errors and uncertainties. Errors depend on many variables whenever they are caused by the environment, instruments, or human errors and can be near impossible to remove entirely. For validating tests and conclusions, it is important to acknowledge possible error sources. When working with higher temperatures it can be assumed that heat losses occurring can affect the test results and depending on the environment's ambient temperature speed of cooling changes. In the test equipment hoses were left uninsulated since its effect was estimated to be neglectable. Air conditioning in the test room kept the temperature at 21 °C which increased slightly during the cooling period. An increase in room temperature occurred similarly during every test meaning that repeatability remained. Changes in the room temperature affect the energy losses in the tank and repeating the test in another environment can produce different results. The largest variable for possible errors originates from temperature sensors that were utilized. Their error estimate is about ±0.5 °C at a temperature range of -10 to +85 °C, which was provided in the datasheet. The traceability of the calibration for the sensor is not clear but the functionality was confirmed with temperature measurement. To minimize error multiple sensors were used to measure the temperature of the water. To have accurate measurements, the sensors used must have an unbroken traceable calibration path that leads up to the SI unit. To confirm this, it would have been possible to contact the sensors manufacturers for more details regarding measurement calibrations. This was deemed unnecessary for the work but for future studies, it is important to have accurate sensors with SI calibrations.

Table 9 Quantifiable errors

Measurement errors in sensors - Room's ambient temperature shift	±0.5 °C
Thermal losses in the system.	-
Difference of the weight in PCM capsules.	1 – 60 g
Estimation of PCM capsule volume.	10 – 20 ml
Error in measured 30-liter water volume in the main tank.	100 – 300 ml ~

Table 10 Unquantifiable errors

Sensor locations in the tank.	Sensors were distributed evenly by hand, but their locations varied with new test materials loaded
On test cycles, a small amount of water evaporated despite the lid in place.	Evaporation of water was present, and it was monitored to prevent it from affecting the tests.
During the heating and upkeep period, the cooling side water line was uncirculated causing a small temperature drop every	Temperature drop did not affect the test since it happened well above the temperature range of PCMs functional

time the cooling phase started, which can be observed from the test figures	temperature. The continuous flow would have fixed this.
No certificate other than provided datasheet regarding temperature sensors SI-calibration traceability.	Sensors purchased new with factory calibration in place. Sensors were ready to use with the measurement error mentioned in the datasheet.

9 CONCLUSION

The project was funded by the EU in part of the Horizon 2020 Making-City program. For this work, three main questions were looked into. Commercial availability, how viable they are in increasing thermal storage capacity, and how they perform in practical tests?

Commercial availability of PCMs today?

During the work, it became apparent that there are companies actively researching different utilities for phase change materials. Some of them focus more on construction and material technology while others have pharmaceutical and medical transportation applications as focus. The energy sector has its share of developments as well, showing that there is a possible growing demand for PCM enhanced technologies, especially with the need to lower CO_2 emissions.

Theoretical viability in increasing thermal energy capacity.

From theoretical calculations, an increase in thermal energy capacity is apparent with all materials. This however does not translate to the functionality of the material due to heat conduction properties and capsulation methods. In literature and research papers regarding the subject the largest challenge has been the poor heat conduction properties and in some cases the supercooling.

Performance during the tests.

Successful tests showed that materials are increasing the thermal energy capacity of the test storage with some materials showing good heat conduction despite high flow rates used. Energy contents and speed in which they released the energy differed. ATS58 and C58 material functioned similarly with some differences in energy contents as seen in Figure 28, Figure 29, and Table 8. With ATP60 which was an organic-based material, transfer of thermal energy was slower meaning that it only could slow down the cooling process. The rate of energy released however seemed to be stable throughout the cooling period. ATS58 and C58 materials were inorganic materials and their heat conduction rate was significantly better as mentioned in the theory, causing the temperature decrease to stop for a short period. GAIA HS PCM58 material was untested due to capsule breakage but they announced new stainless-steel capsules which remain untested. All the test materials functioned at a lower temperature than mentioned in the specs which can be an issue in some applications where the precise temperature is required. The reason for this drop was speculated in the discussion.

There were some limitations to the work regarding the test environment. It was kept simple for a purpose but there could have always been room for improvement. A larger tank with more accurate flow sensors accompanied by more precise pumps to achieve better dynamical scalability. This scalability would be important for the possible modeling of the system.

9.1 Summary

Overall work provided good experience and information regarding PCM technology today. Commercial availability, theoretical calculations, and its challenges as well as practical information through tests. It seemed that this technology has advanced a lot in 10 years to point commercialization. Practical tests showed that there is a potential for an increase in thermal energy capacity. This alone is not enough for a successful product since the price of this material, as well as its lifetime, will show if it will be adopted on a larger scale. Successful application of PCM in thermal storage could increase the building's overall capacity for flexible use of energy or peak shaving. Higher thermal capacity in buildings can open flexible energy market control on a wider scale to lower CO_2 emissions and improve renewable energy production efficiency by lowering the peak demands.

Out of many papers read, none seemed to have direct comparisons of different PCMs and their increase of energy content in water. This is a significant aspect when looking into the economic feasibility and energy benefits of the encapsulated PCM used in TES applications. The importance of flexibility in thermal energy was brought up in many publications, implying that research for effective thermal storage is still relevant. PCM technology is tested in the district of Kaukovainio to improve thermal energy utilization of the local market.

9.2 Future research needs

The task in the main project was to research the viability of the PCM materials to be used in the domestic TES systems. This and other research questions were answered and there is potential for PCMs to be used effectively in peak-load shifting purposes due to an increase in thermal energy capacity and thus lowering CO_2 emissions when used with smart energy systems. As it is now there is still an issue with scare temperature variety with material suppliers, the question of a lifetime coupled with the question of economic feasibility. The next steps possible for the study would be increasing the scale of the test equipment and testing PCMs with different flow rates coupled with dynamically scaled dimensions. This was not possible at this time due to time restrictions and they should be considered in future research. PCMs should also be made with bio-degradable materials and have a long lifetime. At the end of their use, their recyclability should be looked into to have an efficient lifecycle. These were left out of the study and with the possibility of more common use of PCMs, these are aspects that cannot be ignored. Additionally, low-temperature materials that function in a range of 15 – 40 °C should be studied more coupled with PED peak shifting capabilities.

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