

IMPACT OF LOW COOLING RATES AND HIGH COOLING RATE (QUENCHING) ON WATER SUPERCOOLING

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ABSTRACT

Supercooling is a phenomenon when a liquid crystallizes below the liquid–solid equilibrium temperature (melting temperature). Phase change materials can undergo supercooling, impacting the efficiency of the thermal battery or the energy storing/retrieving process. Among different factors influencing the nucleation, cooling rate is one of the factors. Different results have been reported in literature about the impact of cooling rate, and no consensus is observed between them. Due to this non-conformity of the results, impact of cooling rate is studied in this research. The objective of the current study is to investigate the effect of lower cooling rates (0.083°C/min to 1°C/min) and very high cooling rate on the degree of supercooling. The high cooling rate is termed as quenching. In quenching experiments, distilled water samples were immersed in the bath containing anti freezing fluid already at lower temperatures (-25°C), causing very rapid cooling of the samples. By integrating the output of a heat transfer model with an external temperature measurement, the degree of supercooling is evaluated. The samples have a volume of 3 ml in the cylindrical tubes with a diameter of 10 mm. The supercooling obtained in quenching experiments is compared to the supercooling observed in experiments with cooling rate in the range of 0.083°C/min to 1°C/min. The results of the quenching experiments showed that the supercooling of water was smaller than the degree of supercooling at slower cooling rates (0.083°C/min to 1°C/min). The lower cooling rates in the range of 0.083°C/min to 1°C/min had no discernible effect on the degree of supercooling.

1 INTRODUCTION

Renewable energy is important to mitigate climate change. Currently, renewable energy-based technologies face many problems. Amongst them inefficiencies and the intermittent nature of the renewable energy sources are the main issues. Thermal energy storage (TES) can be a solution to the problems faced by renewable energy systems (Cabeza, 2021). Thermal energy storages (TES) are devices that can store both heat and cold. In many engineering applications, thermal energy storage devices are favored because they can bridge the gap between energy supply and demand. There are three forms of thermal energy storage: sensible heat storage (SHS), latent heat thermal energy storage (LHTES), and thermo-chemical energy storage. LHTES exhibits superior energy storage capacity in comparison to sensible heat storage and can store large amounts of energy in a narrow temperature range within the storage medium. Phase change materials (PCMs) are the materials used in LHTES (Mehling & Cabeza, 2008). PCMs may encounter various issues, such as supercooling. During a phase change from liquid to solid, PCMs are anticipated to solidify at the solid-liquid equilibrium temperature. However, in certain scenarios, the temperature of the PCM drops below this threshold without undergoing crystallization. The difference in temperature between the solid-liquid equilibrium temperature and the crystallization temperature is referred to as the supercooling degree (Sandnes & Rekstad, 2006). Figure 1 depicts this behavior. Crystallization in the PCM initiates upon the nucleation of solid nuclei. There are two types of nucleation processes: homogeneous and heterogeneous

nucleation (Safari et al., 2017). Homogeneous nucleation happens when a PCM in a liquid state is evenly chilled below its freezing point without any nucleation sites present in it, which may cause solidification. In this case solid nuclei arise independently of an existing solid object, unlike in heterogeneous nucleation. Heterogeneous nucleation refers to the process where the nucleation occurs on the surface when a cooling liquid contacts foreign substances like fins, heat exchangers, and other structures (Shamseddine et al., 2022).

1.1 Disadvantages of Supercooling

The supercooling can be beneficial and adverse depending upon the application. Two significant downsides are associated with the supercooling.

- The nucleation temperature can be out of the system's operating temperature range.
- Second, the system will consume extra energy. (Zahir et al., 2019).

Between these two limitations, the most undesirable one is the fact that the range of temperature in which a system operates at, might not encompass the freezing point of the supercooled PCM. A liquid that has undergone supercooling may become exceedingly unstable; even a minor disruption or the presence of a nucleation site may trigger quick solidification. The second challenge introduced into energy systems owing to supercooling is the increase in energy consumption as the liquid PCM requires an extra energy input to drop the temperature below its solid-liquid equilibrium temperature, which can be a major expense in energy in some circumstances (Zahir et al., 2019).

1.2 Factors Affecting Supercooling

It is crucial to reduce supercooling in situations where it is unwanted. It is necessary to investigate the elements that influence the supercooling. The following factors have the greatest impact on supercooling: (i) Volume, (ii) Surface roughness of the container, (iii) Thermal history, (iv) Purity of PCM, and (v) Cooling rate. This study just examined the influence of the cooling rate. Table 1 displays the literature review regarding the effect of the cooling rate on the degree of supercooling.

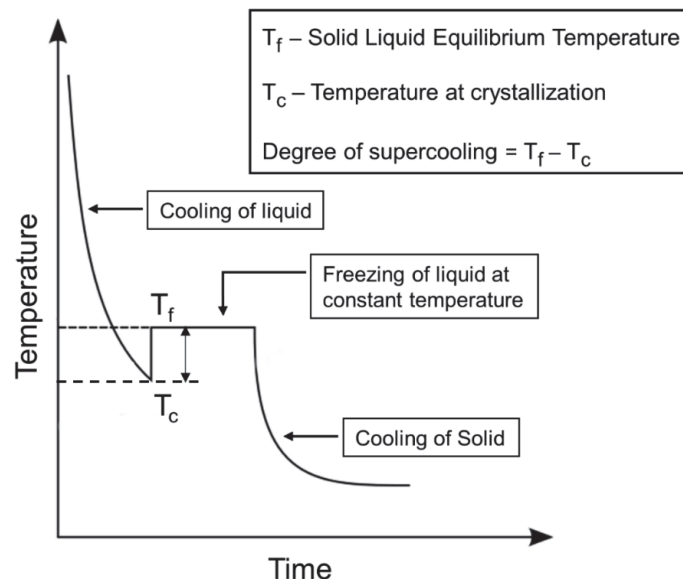


Figure. 1: Temperature evolution within the phase change material during a cooling process.

Table 1: Comparison of literature review presenting the impact of cooling rate on the degree of supercooling.

Ref	Def. of Cooling Rate	Cooling Rate (°C/min)	PCM	Volume of PCM (ml)	Number of Samples Analyzed	Thermocouple		Results
						Position (inside or outside the sample)	Position (inside or outside the sample)	
(Chen & Lee, 1998)	Ratio of the difference between the initial and crystallization temperature to time	0.5 – 3.2	Distilled water	780	24	Inside		At the lower coolant temperatures (high cooling rates), the degree of supercooling decreases.
(Taylor et al., 2016)	The slope ($\Delta T/\Delta t$) of the initial part of the curve before the onset of nucleation	20 - 80	hydrated CaCl ₂ based (PC25)	5.35	4	NTC Thermistors Inside		The degree of supercooling increased from 9 to 22 °C with increasing cooling rate.
(Solomon et al., 2013)	-	0.1 - 0.25	RT21 (Paraffin based)	3073	Few times for repeatability	Inside		The supercooling increases when the cooling rate increases.
(Yoon et al., 2001)	Ratio of the difference between the initial and crystallization temperature to time	1.2 - 18	Distilled water	35.34	-	Inside		The high cooling rates (10 – 18 °C/min) had a lower degree of supercooling (5 °C), while for the lower cooling rates, a higher degree of supercooling (7°C).
(Song et al., 2023)	Cooling rate applied on DSC	1 - 10	Decanoid acid	0.013	120	DSC(probe outside)		There was no impact of cooling rate on the degree of supercooling
(Song et al., 2023)	Cooling rate applied on DSC	1 - 10	MgCl ₂ 6H ₂ O	0.012	150	DSC(probe outside)		Higher values of degree of supercooling were observed for higher cooling rates
(Mossop, 1955)	Applied Cooling rate in dewar vessel.	0.05–0.5	Distilled water	5 × 10 ⁻⁵ to 12 × 10 ⁻³	36	Thermometer outside the sample		The difference between the degree of supercooling was less than 0.4°C, which is very insignificant

There is no unanimity in the literature review. Three distinct tendencies have been documented:, increasingly supercooling with cooling rates (Taylor et al., 2016; Solomon et al., 2013; Song et al., 2023), decreasing supercooling with increasing cooling rates (Chen & Lee, 1998; Yoon et al., 2001) and no impact of varying cooling rates (Mossop, 1955; Song et al., 2023). Therefore, it is very important to investigate this impact.

1.3 Research Objectives

Researchers have attempted to investigate the supercooling and its relationship with different parameters. Experiments have been conducted on a small scale in the laboratory, and the impact of certain factors, like the cooling rate, remains uncertain. The supercooling's volume dependence renders the laboratory scale results inapplicable to bigger volumes and installations in the real systems. To forecast the supercooling of actual systems, a laboratory-scale analysis of the numerous elements influencing supercooling will be conducted. Our initiative aims to utilize laboratory scale data and extrapolate it using statistical modeling. The outcome of this research aligns with a goal of the PCM STORE project to enhance understanding of phase change materials utilized in practical applications and to create thermal energy storage devices for cold storage purposes.

This article discusses the findings of the initial phase of the study, which focuses on analyzing how supercooling is affected by various variables at a laboratory size. The objective of this study is to find the effect of low cooling rates and very high cooling rate (quenching) on the supercooling of distilled water. Along with the investigation of the impact of cooling rate, quenching is the novelty of this study.

2 MATERIALS AND METHODOLOGY

2.1 Experimental Setup

Distilled water serves as the phase change material (PCM). Water samples are collected in the polypropylene test tubes. The tubes have an inner diameter of 10mm and a thickness of 1mm. Each tube contains 3ml of water. Two distinct setups are depicted in Figure 2. The initial setup includes a single thermocouple positioned outside the sample at 1.5ml. The second setup has two thermocouples positioned at 1.5ml, one inside the water and one outside on the exterior wall. Thermocouple probes made of wire type are positioned outside and secured near the tube wall using a zip-tie. Rigid-probe thermocouples are utilized for sensing internal temperatures. The rigid probes ensure precise positioning. All probes are of the T type. It is widely accepted that foreign surfaces can affect the PCM supercooling (Zhao et al., 2020). The actual degree of supercooling can be altered through thermocouple probes inserted into the PCMs. This is validated by comparing results where temperature is measured inside the sample and outside the sample (see section 3.1). Therefore, probes are used outside the sample to measure its temperature.

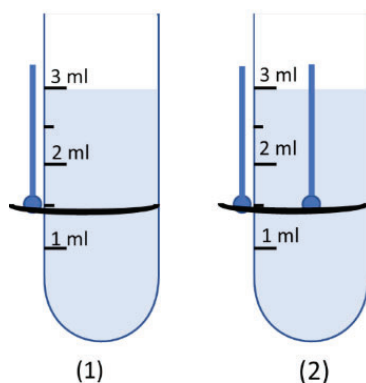


Figure 2: Different configurations: (1) Only one thermocouple probe outside the sample (2) Two probes, one inside and one outside the sample tube

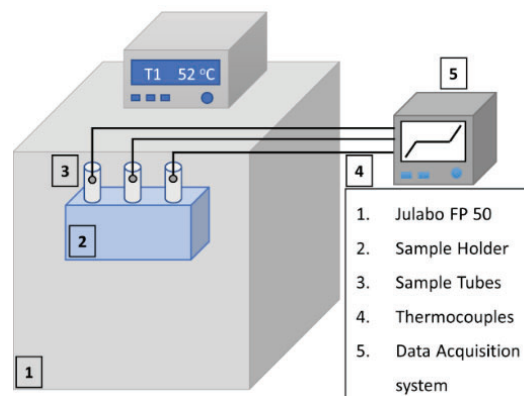


Figure 3: Experimental setup

Table 2: PCM samples set up in a variety of different settings for the experiments

Configuration	Location of Thermocouple	Purpose
1	Outside	To find actual supercooling
2	Inside and outside	To validate numerical model and find the impact of the probe inside the tube.

Configuration 1 is utilized to determine precisely the degree of supercooling without any external item in the sample, but it incorporates the temperature gradient along the tube wall. Thus, a numerical model is utilized to determine the temperature within the sample. The numerical model is validated by Configuration 2, which records the temperature difference between the PCM's center and the tube's exterior wall. This information is presented in Table 2. Figure 3 displays the experimental arrangement. A Julabo FP 50 refrigerated heating circulator with a bath is used to regulate the temperature of the heat transfer fluid (HTF) it contains. The water sample tubes are placed into a test tube holder that is submerged completely in the bath. The thermocouples are positioned at various positions in the tubes based on the combinations outlined in table 2. The KEYSIGHT DAQ970A data acquisition system is utilized to measure and record the temperature of the samples throughout time. The accuracy of thermocouples is $\pm 0.1^\circ\text{C}$. The HTF employed in our investigation is an aqueous solution containing 50% water and 50% monoethylene glycol (Neutragel®).

2.2 Methodology for Quenching Experiments

The temperature of the cooling bath containing the HTF was set at -25°C . The rack with the distilled water samples was suddenly immersed in the cooling bath. The temperature of the samples was recorded with the outside probe. The temperature of the sample just before the crystallization is the required measurement to further determine supercooling. The inside wall temperature of the tube was determined by the result of a heat transfer model (see section 2.4). As shown in Figure 1, the degree of supercooling was determined by calculating the difference between the temperature at which crystallization occurred and the solid-liquid equilibrium temperature. In each experiment, there were a total of 27 sample tubes. In order to analyze the value of the degree of supercooling, the experiment was carried out a total of twelve times, resulting in a statistical average of three hundred twenty-four results.

2.3 Methodology for Slower Cooling Rate Experiments

The cooling bath with the heat transfer fluid (HTF) and the rack with the distilled water samples underwent a temperature cycle. The samples' temperature was recorded, and the degree of supercooling was determined by calculating the difference between the crystallization temperature and the solid-liquid equilibrium temperature, as detailed in Figure 1. Each experiment contained 16 sample tubes. The experiment was conducted 19 times to get the statistical average value of the degree of supercooling for each configuration at each cooling rate examined. Figure 4 displays the schematic of the applied cycle. Figure 4 also depicts the cooling rate.

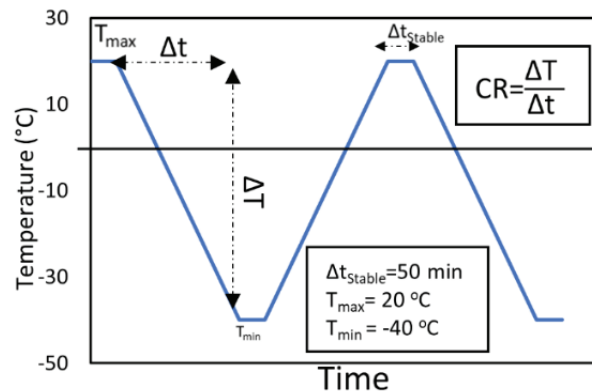


Figure 4: Temperature cycle applied on the bath and explanation of the cooling rate.

Combining the accuracy of the thermocouple itself with the standard error of the statistical data allows for the calculation of the error bounds for the outcomes of the experiment. The temperature within the tube for configuration 1 was determined using a numerical model (see section 2.4). Maximum error in validation is also coupled with the error for the numerical findings.

2.4 Numerical Modelling

The aim of the simulation was to determine the temperature within the tube right before the onset of crystallization, based on the experimentally determined temperature of the bath. For this reason, modeling the liquid-solid phase transition is not required. The sample's geometry was designed to be 2D axisymmetric. Figure 5 displays the geometry. The sample tube has identical dimensions to the tubes utilized in the experiments.

Natural convection is considered inside the water:

$$\frac{\partial \rho}{\partial t} + \nabla \cdot (\rho \mathbf{u}) = 0 \quad (1)$$

$$\rho \frac{\partial \vec{u}}{\partial t} + \rho(\vec{u} \cdot \nabla)\vec{u} = \nabla \cdot [-p\mathbf{I} + \mathbf{K}] + \rho \vec{g} \quad (2)$$

$$\mathbf{K} = \mu(\nabla \vec{u} + (\nabla \vec{u})^T) \quad (3)$$

$$\rho C_p \frac{\partial T}{\partial t} + \rho C_p \vec{u} \cdot \nabla T = \nabla \cdot (k \nabla T) \quad (4)$$

Conduction is considered in the tube wall:

$$\rho C_p \frac{\partial T}{\partial t} = \nabla \cdot (k \nabla T) \quad (5)$$

Natural convection is considered inside the bath in HTF.

$$\frac{\partial \rho}{\partial t} + \nabla \cdot (\rho \mathbf{u}) = 0 \quad (6)$$

$$\rho \frac{\partial \vec{u}}{\partial t} + \rho(\vec{u} \cdot \nabla)\vec{u} = \nabla \cdot [-p\mathbf{I} + \mathbf{K}] + \rho \vec{g} \quad (7)$$

$$\mathbf{K} = \mu(\nabla \vec{u} + (\nabla \vec{u})^T) \quad (8)$$

$$\rho C_p \frac{\partial T}{\partial t} + \rho C_p \vec{u} \cdot \nabla T = \nabla \cdot (k \nabla T) \quad (9)$$

The specific heat capacity, thermal conductivity, dynamic viscosity, and density of the supercooled water were obtained from the data in the published literature. (Biddle et al., 2013), (Speedy, 1987) and (Dehaoui et al., 2015). While for the glycol-water (50% composition), properties were taken from *Monoethylene Glycol MEG Technical Product Brochure*, (2013). Figure 5 shows the boundary conditions. The initial conditions in case of quenching and slower cooling rate (0.083 – 1 °C/min) are presented in Table 3. The model equations were solved using a commercial software COMSOL Multiphysics 6.1, which is based on mathematical modeling technique Finite Element Method (FEM). A time-dependent study was adopted. For time-dependent convergence, error tolerance. was set at 0.001. The output time step in time-dependent study was set to 1 minute.

Table 3: Initial conditions for the numerical model in case of slower cooling rates and quenching

	Quenching		Cooling Rates (0.083 – 1 °C/min)	
	Tube	Bath	Tube	Bath
Temperature	T_i	–25°C	20°C	T_{cycle}
Velocity	0 m s ⁻¹	0 m s ⁻¹	0 m s ⁻¹	0 m s ⁻¹

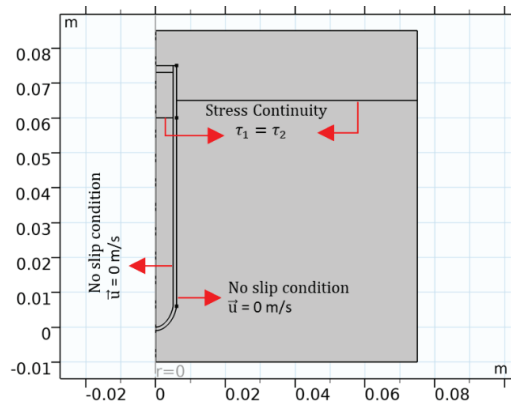


Figure 5: 2D axis symmetric geometry and boundary conditions

2.5 Validation and Mesh Dependence

Probes positioned on the tube's outside wall and in the middle of the sample were used to conduct the validation tests. The center probe results were compared with the numerical data obtained at the same point. The temperatures prior to crystallization are utilized for the model validation since the numerical model is unable to forecast the nucleation point. Figure 6 displays the experimental and numerical change of temperature over time for a specific cooling rate. Figure 7 illustrates the discrepancy between compares the experimental data and the computational model regarding the temperature at the center of the phase change material (PCM) right before crystallization. The variation is under 3% for all the examined cooling rates (0.083 to 1 °C/min). A mesh refinement study was conducted and increasing the number of domain elements from 700 to 2400 did not yield any substantial impact on the outcomes. As a result, the model is validated and suitable for forecasting PCM temperatures inside the tube. This validated model for lower cooling rates was extrapolated to predict the temperatures inside the PCM for quenching experiments. The temperature difference between the exterior wall and center of PCM, measured experimentally for a quenching experiment is depicted in Figure 8. This temperature difference is very close to the results obtained by the extrapolated numerical model. The goal of this quenching experiment with one probe inside and one probe outside the tube (configuration 2) was to evaluate if the extrapolation of the model to quenching experiments was relevant. This is shown in Figure 8.

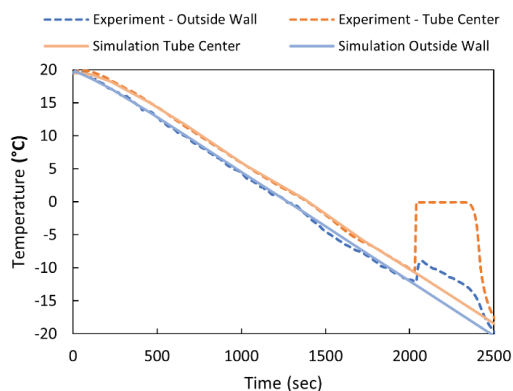


Figure 6: Temperature evolution in the center and outside wall, both numerically and experimentally

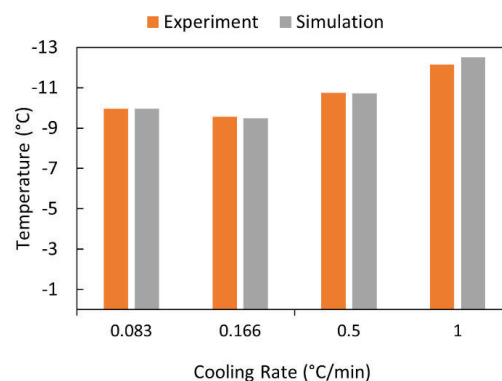


Figure 7: Difference in temperature between the computational and experimental data for various cooling rates at the center of samples prior to crystallization

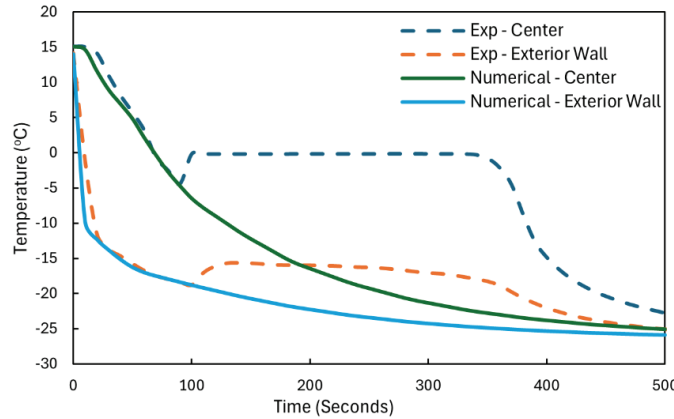


Figure 8: Temperature difference between the center and outside wall, both numerically and experimentally

3 RESULTS AND DISCUSSION

3.1 Impact of Thermocouple Probe

Several configurations are used to investigate the impact of the thermocouple probe on the supercooling. If the degree of supercooling measured by the probe inside the sample (configuration 2) is lower than the degree of supercooling measured by the probe outside the sample (configuration 1), we will conclude that measuring temperature inside the sample will alter the degree of supercooling. Figure 9 demonstrates that samples with single outside probes (configuration 1) have a larger degree of supercooling than samples with probes inside (configuration 2), for all cooling rates. This indicates that the 3 ml distilled water sample's supercooling degree is affected and decreased by the probes inside the sample tube.

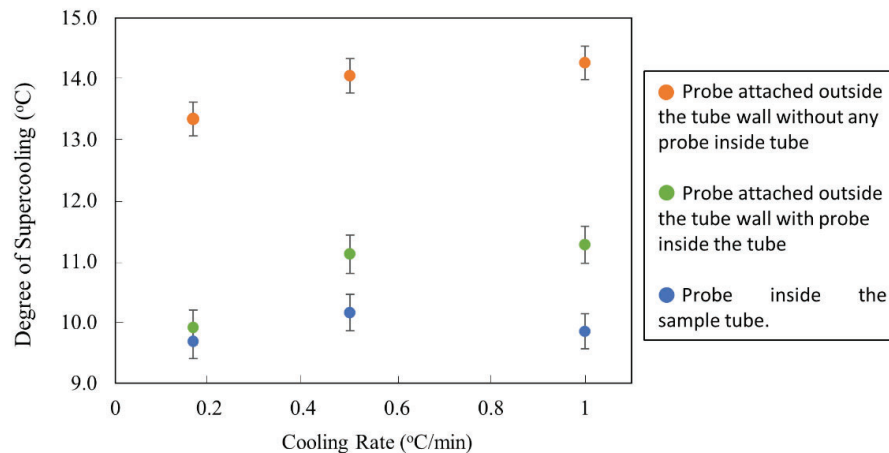


Figure 9: Comparison of the degree of supercooling measured with probe placed inside the sample and outside the sample.

3.2 Temperature Measured Outside the Sample Tube.

The degree of supercooling was measured by placing a thermocouple outside the tube containing the distilled water sample (configuration 1) for both cases (slower cooling rates and quenching). The studied

cooling rates were 0.083, 0.166, 0.5, 1 °C/min and quenching. Figure 10 shows the degree of supercooling with temperature measured outside the sample tube just before the crystallization.

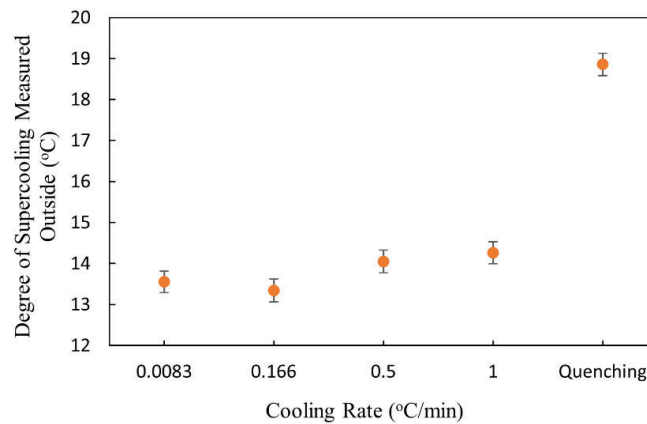


Figure 10: Degree of supercooling obtained by the temperature measurement outside the sample.

However, these results are obtained through a temperature measurement obtained with a thermocouple placed outside the tube and the temperature measured outside the sample does not represent the actual temperature of the PCM inside the sample. This is due to the temperature difference present across the tube wall. To accurately anticipate the right level of supercooling, it is essential to determine and then subtract the temperature difference from the externally recorded degree of supercooling.

3.3 Correcting Degree of Supercooling using Numerical Model

The degree of supercooling can be determined by calculating the temperature of the inner wall of the tube through a numerical model. The nucleation happens at the sample's lowest temperature point, which is the interface between the PCM and tube, hence this temperature is necessary rather than any other location in the sample. Figure 11 illustrates that the temperature gradient across the wall rises with an increase in the cooling rate. It confirms that the degree of supercooling measured by using the thermocouple probe outside the sample will not correspond to the actual nucleation temperature.

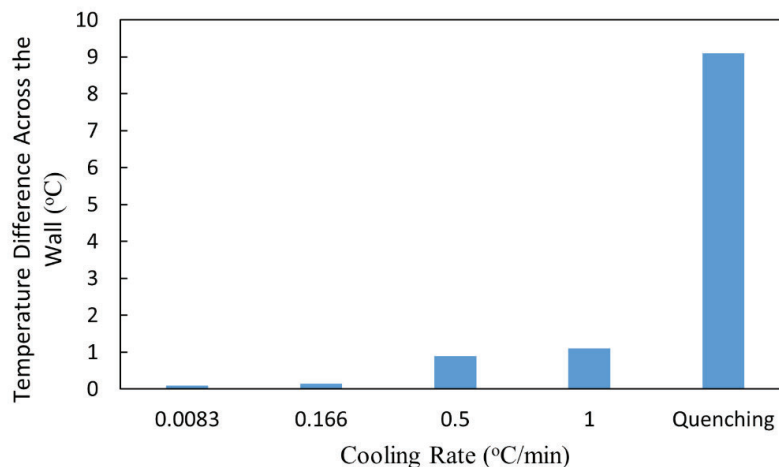


Figure 11: Temperature difference across the wall.

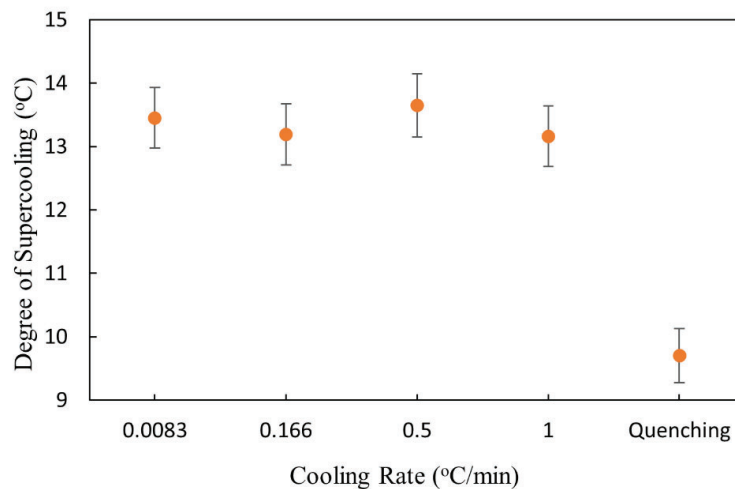


Figure 12: Degree of supercooling estimated at inner wall of tube.

The true degree of supercooling, determined computationally knowing the temperature difference across the wall, is displayed in Figure 12. Because of the temperature difference across the tube wall, the degree of supercooling as measured from outside the sample (Figure 10) is larger than the actual degree of supercooling. These results show that the degree of supercooling does not change by varying cooling rate in the range of lower cooling rates (0.083 to 1 °C/min) but the degree of supercooling decreases at very high cooling rate (quenching).

3.4 Comparison of the Results with the Literature

The two major results obtained in this study are:

- i. The cooling rate in the range of 0.083 to 1 °C/min has no impact on the supercooling.
- ii. The high cooling rate (quenching) decreases the degree of supercooling.

The first conclusion of this study was in agreement with the results of Mossop, 1955 and Song et al., 2023. Mossop, 1955 used the distilled water (5×10^{-5} ml). The author varied the cooling rate from 0.05–0.5 °C/min. The results showed that there was no significant impact of the cooling rate on the degree of supercooling. Song et al., 2023 used 0.013 ml of the decanoic acid. The authors varied the cooling rate from 1–10 °C/min. The results showed that there was no significant impact of the cooling rate on the degree of supercooling. The second conclusion of this study was in agreement with the results of the Yoon et al., 2001 and Chen & Lee, 1998. Yoon et al., 2001 used 35 ml of distilled water. They varied the cooling rate from 1.2–18 °C/min. The results showed that the high cooling rates (10 – 18 °C/min) had a lower degree of supercooling (5 °C), while for the lower cooling rates, a higher degree of supercooling (7°C). Chen & Lee, 1998 used 780 ml of distilled water. They varied the cooling rate from 0.5 – 3.2 °C/min. The results showed that at the lower coolant temperatures (high cooling rates), the degree of supercooling decreases. While other authors find the results, which are different than this stud. This may be due to differences in volumes of the PCM used, number of experiments performed to obtain results, and position of the thermocouple.

4 CONCLUSION

PCM Store is a project focused on developing thermal energy storage devices for cold storage facilities. To do this, understanding the relationship between supercooling and other parameters at the laboratory scale is essential. The supercooling of actual systems can be predicted by extrapolating this laboratory-scale data. This study examines how supercooling is affected by different lower cooling rates (0.083-1 °C/min) as well as a high cooling rate known as quenching. The temperature is measured using a probe placed outside the sample to avoid affecting nucleation. Initially, the temperature is measured outside the tube and then the temperature inside the tube is calculated using simulation to determine the real

degree of supercooling. The model was validated for lower cooling rates. The same model was extrapolated for quenching experiments. Though the results obtained by this extrapolated model for quenching experiments performed at -25°C are very close to first experimental results, it would be necessary, in the future, to validate the model for quenching experiments with more experiments at -25°C and mostly at lower temperatures (-30°C and -35°C). It was observed that the quenching (high cooling rate) has a significant impact on the degree of supercooling of distilled water. The degree of supercooling found in the quenching experiment was smaller compared to degree of supercooling determined at slower cooling rates (0.083, 0.16, 0.5, $1^{\circ}\text{C}/\text{min}$). Finally, no major impact of the cooling rate on the supercooling degree was found for cooling rates from $0.083^{\circ}\text{C}/\text{min}$ to $1^{\circ}\text{C}/\text{min}$.

NOMENCLATURE

<i>DSC</i>	Differential scanning calorimetry	
<i>LHTES</i>	Latent Heat Thermal Energy Storage	
<i>NTC</i>	Negative Temperature Coefficient	
<i>PCM</i>	Phase Change Material	
<i>TES</i>	Thermal Energy Storage	
C_p	Specific heat capacity	$(\text{J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1})$
D	Diameter	(m)
h	Heat transfer coefficient	$(\text{W}\cdot\text{m}^{-2}\cdot\text{K}^{-1})$
\vec{g}	gravitational acceleration vector	
I	Identity Tensor	
k	Thermal Conductivity	$(\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1})$
Pr	Prandtl Number	
P	Pressure	$(\text{N}\cdot\text{m}^{-2})$
Re	Reynolds Number	
t	Time	
T	Temperature	(K)
T_{ext}	External Temperature	(K)
\vec{u}	Velocity vector	$(\text{m}\cdot\text{s}^{-1})$
Q_{wall}	Heat Flux	$(\text{W}\cdot\text{m}^{-2})$
ρ	Density	$(\text{kg}\cdot\text{m}^{-3})$
μ	Dynamic Viscosity	$(\text{N}\cdot\text{s}\cdot\text{m}^{-2})$

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