



Processing dates: received on 2024-07-15, reviewed on 2025-03-26,
accepted on 2025-03-27 and online availability on 2025-04-30

Development of a Sodium Chloride (NaCl) Eutectic Phase Change Material for Fish Cold Storage Applications

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Abstract

Sustainable marine resource management is essential, as the sea provides a vital source of nutrition for humans. Fish and shellfish are highly perishable, making effective low-temperature storage systems crucial for preserving their quality. This research developed a cold storage system utilizing Phase Change Material (PCM) for low-temperature applications. While water is commonly used as a PCM due to its excellent thermal properties, it is unsuitable for low-temperature applications because of its 0 °C freezing point. To overcome this, Sodium Chloride (NaCl) was introduced as a solute to lower the freezing point, resulting in eutectic salt solutions with 3%, 6%, and 9% weight concentrations. Thermal characterization revealed that the 9% wt. NaCl solution exhibited the most favourable properties, achieving a freezing point of -5.8 °C while maintaining a latent heat of fusion comparable to water. Experimental validation demonstrated that 5 kg of fish at approximately 0 °C for 24.3 hours, with minimal temperature fluctuation. Organoleptic evaluation yielded a score of 8 at a 95% confidence level, indicating excellent preservation quality. These findings highlight the potential of NaCl eutectic solutions as cost-effective, sustainable alternatives to conventional refrigeration methods for seafood storage.

Keywords:

Sustainable marine resource, eutectic aqueous, sodium chloride, phase change materials, cold storage.

1 Introduction

Indonesia is the second-largest fishery-producing country after China, producing 24 million tons of fish per year. However, the exported fishery products are still very small, only about 4% of Indonesian fishery production per year. This has resulted in Indonesia being in the 15th position globally in the export of fishery products. Some of the problems are caused by the fact that most capture fisheries, aquaculture, fishery product processing, and trade in fishery products are traditionally carried out on a small and micro scale. Even the fish containers used are usually reed, bamboo, grass, or bamboo. This results in a decrease in the quality of the fish, so it is not suitable for export.

A decrease in and quality of post-harvest fish occurs due to autolytic damage and the growth of microorganisms. One way to inhibit the decline in quality in fish is to store them at low temperatures because every 8 °C decrease in temperature can inhibit the rate of metabolic reactions in fish. Low-temperature storage in

fish is usually used as a cooling medium in the form of water. Water is one of the most commonly used Phase Change Material (PCM) types because it has good thermal properties. However, water is not suitable for low-temperature applications because it has a freezing point of 0 °C. Anam's research shows that 1.2 kg of ice can only maintain the temperature of fish at a temperature of 5.7 °C [1]. While for cold storage, the ideal temperature for fish is stored at 0 °C [2]. Fish storage temperatures above 40 °C can cause food poisoning due to the formation of histamine. Therefore, to maintain the temperature of the fish at around 0 °C, a cooling medium with a freezing point well below 0 °C is needed.

Given that water is one of the best PCMs for several reasons, such as low price, ease of obtaining, and good thermal properties, water is used as the basic material for the development of new PCMs that are more sustainable for fish storage applications [3], [4]. To suit the storage application temperature, solutes are added to the water to lower the solution's freezing point. Because, based on the colligative properties of the solution, increasing the percentage of solute in the solution can lower the freezing point of the solution. The solute that will be added to the water is in the form of salt to form a eutectic solution of brine. The most studied salts to form a brine eutectic solution are Sodium Chloride (NaCl), Potassium Chloride (KCl), and Sodium Nitrate (NaNO₃). Others have also analyzed that the use of NaCl is better than KCl and NaNO₃ in terms of thermal properties, chemical properties, and economic properties [5].

This study focuses on the thermal properties, such as the appropriate amount of cooling media needed to produce the desired cooling temperature, and organoleptic properties through the investigation of freezing point, degree of supercooling, and latent heat. The addition of the percentage of NaCl into the water to form an eutectic NaCl solution to obtain a new PCM which has good thermal properties like water, but has a freezing point far below 0 °C, making it suitable for fresh fish cold storage applications.

2 Method

2.1 Materials

To reduce and maintain the temperature of the fish at 0 °C, the PCM developed must have a freezing point well below 0 °C to minimize the heat transfer area when cooling the fish. However, given that the PCM must be frozen during the charging process, the freezing point of the PCM should not be too low, and the high degree of supercooling will require a cooler unit that is more expensive and operates at lower efficiency. Li et al stated that a solution with 100% water would freeze at 0 °C [5], [6]. When more salt is added, the freezing point of the salt solution will decrease until it reaches the eutectic point. The eutectic point for NaCl solution occurs at a concentration of 23.35% wt. NaCl and a temperature of -21.5 °C [6]. Based on this, three different concentrations of 3%, 6%, and 9% wt. NaCl salt solutions were selected as PCM candidates. To facilitate handling and use regularly, PCM will be placed in a polycarbonate container measuring 22 x 13 x 2 cm.

In fish storage, the cooling medium absorbs heat from the environment. To handle this, a fish container with suitable insulating material is needed. Fish containers are generally available in various materials, from traditional ones with low prices but poor insulation, such as bamboo, wood, and reeds. Modern materials with high prices and good insulation, such as HDPE and aluminum.

Today, expanded polystyrene or Styrofoam, as an alternative material for fish storage containers, has been used in the Philippines [2]. Yusuf et al stated that Styrofoam material has good insulation compared to wood and fiber. Considering that Indonesian fishermen are small and micro-scale businesses, but require fish storage containers with good insulation, Styrofoam material is chosen as a fish storage container [7].

Fish is a perishable material. This is due to the decay process. The spoilage process in fish is influenced by temperature, physical damage, and intrinsic factors. Based on the intrinsic factor, fish with round, small shapes, fatty fish, and thin skin have a high decay rate.

Catfish have a round, small intrinsic factor, lean fish, and thin skin to have a high rate of decay.

2.2 Method

The characteristics of PCM have been proposed by T-History [7], as presented in Fig. 1. The initial step taken was PCM sample preparation, which involved dissolving NaCl powder into the water with concentrations of 0%, 3%, 6%, and 9% wt. NaCl. Next, stir the sample using a magnetic stirrer at a speed of 300 rpm at a temperature of 15 °C for 10 minutes to form a homogeneous solution. Put the PCM sample into an identical 20 ml test tube. Temperature measurements and recordings were carried out using a type K thermocouple with an accuracy of ± 0.5 °C. The thermocouple is placed in the PCM and the refrigerator. Based on the PCM temperature data, the average cooling rate is calculated. The cooling rate is calculated as a cross-sectional area using Eq. (1) [8]:

$$Q_{PCM} = (\Delta X_s)mC_{ps}dT + (\Delta X_{st})mL + (\Delta X_l)mC_{pl}dT \quad (1)$$

Where Q is the total energy, ΔX is the percentage of mass with subscript notation s for solid phase, st for phase change, and l for the liquid phase (%), dT is the temperature difference (K), and Cp is the specific heat ($\text{kJ}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$).

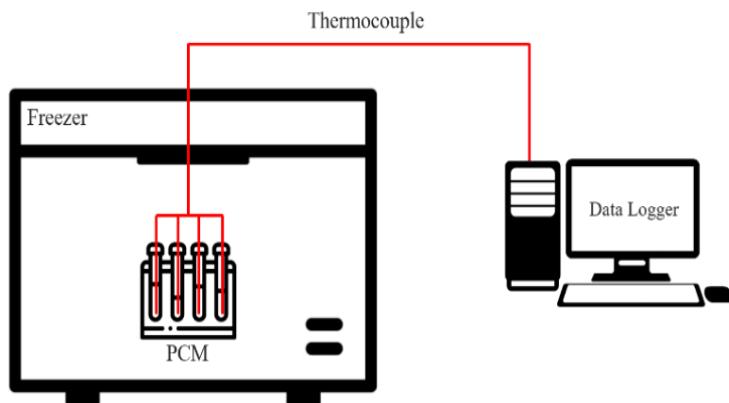


Fig. 1. T-History Schematic

Initially, the PCM was placed at a room temperature of 30 °C so that it was in the liquid phase, then put into the refrigerator at a temperature of -22 °C below the PCM's freezing point. As a result of the temperature difference, the PCM temperature drops, resulting in a freezing process. The following freezing process is achieved after the PCM undergoes supercooling and the temperature drops to close to the refrigerator temperature. Measurement and recording of temperature during the experiment were carried out using a temperature recorder brand named Lutron BMT-4208SD, with a time interval of 5 seconds.

Cold storage testing is carried out to cool the temperature of 5 kg of fish so that the cooling process can be achieved at the desired temperature optimally. The calculation of the cooling load needs to be carried out to determine the amount of cooling media needed. Calculation of the cooling load is done by the Cooling Load Temperature Difference (CLTD) method. The selection of PCM as a cold storage cooling medium was carried out. Before the cold storage test based on the thermal characteristics of the four PCM samples. The best PCM as a cooling medium for cold storage of fresh fish is selected and put into an icepack container according to need. Then the PCM is put into the refrigerator for the filling process. The charging process is complete when the PCM has frozen completely.

A box made of expanded polystyrene measuring 30x21x25 cm that can store 5 kg of fish is used as cold storage. After the PCM freezes completely, the PCM is inserted and placed on each cold storage wall, as shown in Fig. 2. Furthermore, 5 kg of fish is added to meet the cold storage. The testing process is carried out for at least 24 hours. For temperature measurement and recording during the

cold storage testing process, type K thermocouples are placed on the PCM, fish, and environment. The thermocouple is connected to a thermometer recorder for temperature recording at 1-minute intervals.

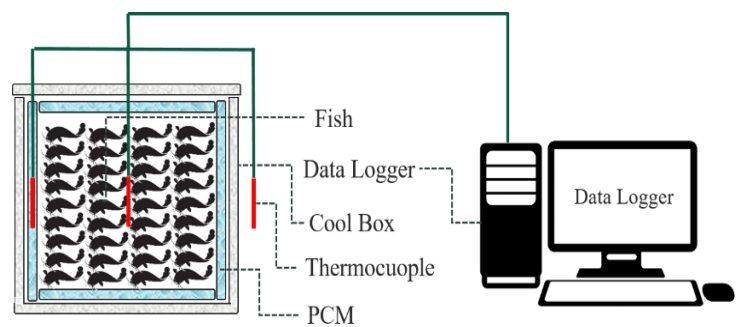
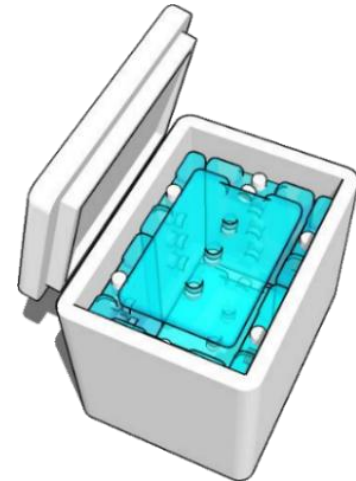


Fig. 2. Cold Storage test scheme

Organoleptic sensory testing may assess product quality using the human senses as the primary tool. Assessment includes appearance, smell, taste, consistency/texture, and several other factors. Due to the subjective nature of the test, a standard is needed in conducting organoleptic sensory assessments. The standard used in this organoleptic test is based on the Indonesian National Standard of SNI 01-2345-2006 [9]. The tests were then carried out on stored fish samples divided by six panelists for organoleptic tests. The results were then filled in an assessment sheet that contained the fish characteristics. The data obtained from the assessment sheet is then tabulated, and the quality value is determined at the 95% confidence level.

3 Results and discussion

The thermal analysis results are presented in Fig. 3 and Table 1. Based on Fig. 3, the cooling process of four types of PCM goes through the same four processes as also presented by others somewhere else [9]:

Sensible heat thermal energy storage process, from the initial temperature (T_o) to the formation of the nucleus (TN), in this process, the PCM is still in the liquid phase even though the temperature drops far below the freezing point. The difference between the freezing point and the initial point for the formation of the nucleus is called the degree of supercooling.

The ice crystal formation process, from the formation of the nucleus (TN) to the formation of an ice layer on the outside, so that sensible heat is still trapped inside, which then spreads over the entire surface until the temperature rises in the PCM until it ends at equilibrium (T_f). Latent heat thermal storage process, a phase change process to form a solid phase completely, and the Ice sensible heat thermal storage process until the temperature is close to the cooling temperature

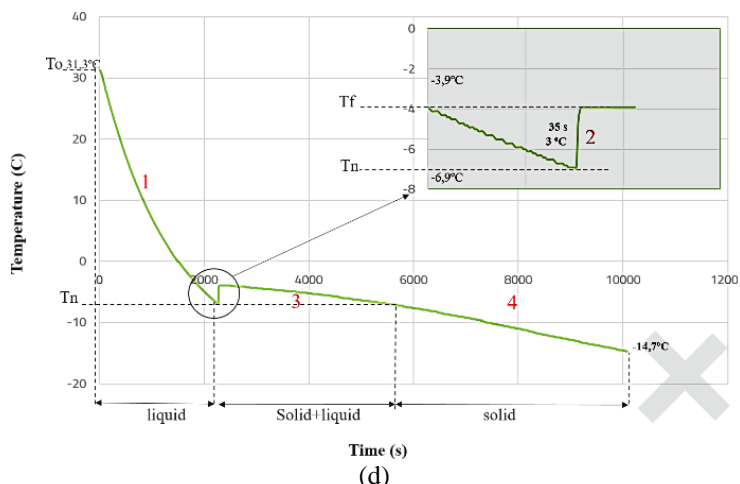
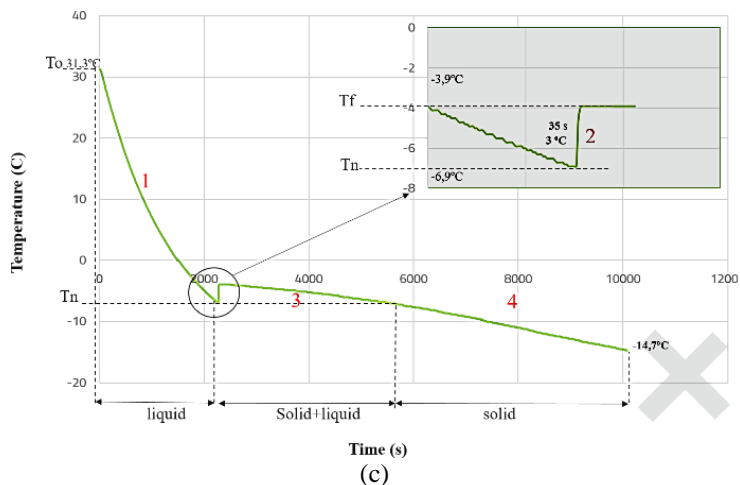
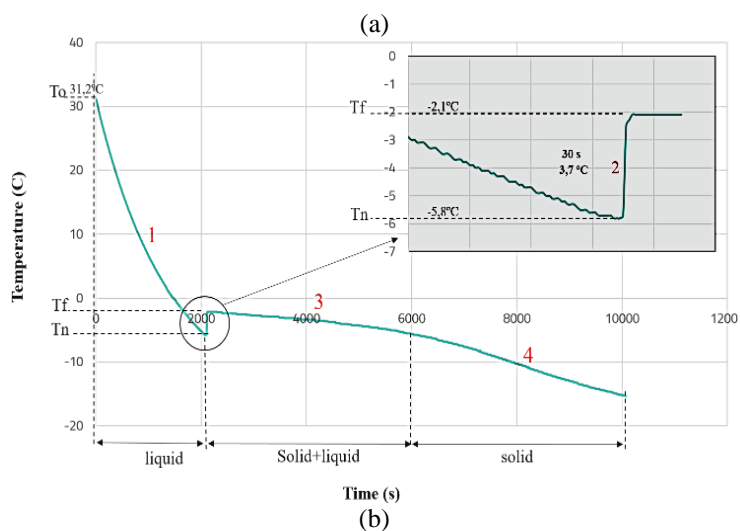
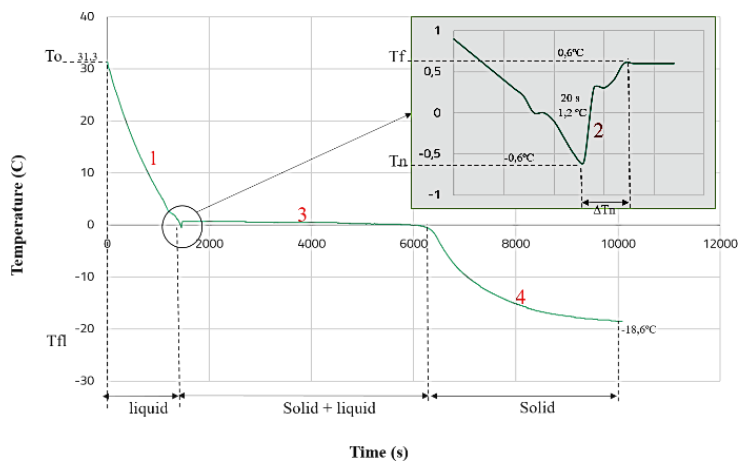


Fig. 3. T-History results (a) Water, (b) 3% wt NaCl, (c) 6% wt NaCl, and (d) 9% wt NaCl

Table. 1. Characteristics of PCM

(%) NaCl	T-History					Calculation	
	T ₀ (°C)	T _N (°C)	T _F (°C)	S (°C)	T _F (°C)	ρ(kg/m ³)	L(kJ/kg)
0 %	31,3	-0,6	0,6	1,2	0	1000	333
3 %	31,2	-5,8	-2,1	3,7	-1,8	1023	329,45
6 %	31,3	-6,9	-3,9	3	-3,7	1046	325,31
9 %	31,3	-8,9	-5,5	3,4	-5,8	1069	321,11

The composition of the 100% aqueous solution has a freezing point of 0.60°C, with the addition of wt.% NaCl to H₂O will lower the freezing point of the solution. The freezing point depression is caused by the colligative nature of water which states that the freezing point of water with the addition of ingredients will drop below the freezing point of water at 0°C, as in Fig. 4. This freezing point depression is known as freezing-point depression, as highlighted by other [10].

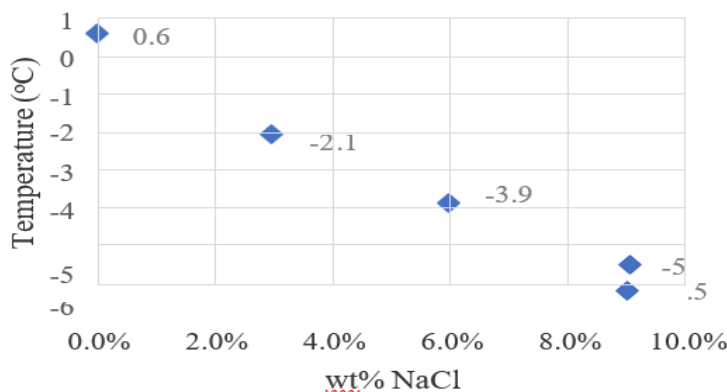


Fig. 4. Freezing point

H₂O underwent supercooling with a supercooling degree of 1.20°C, while the solutions of 3%, 6%, and 9% wt. NaCl were 3.7°C, 3°C, and 3.4°C. Adding wt.% NaCl into H₂O causes the degree of supercooling of the solution to be greater than the degree of supercooling of H₂O (Fig. 5). The increase in the degree of supercooling caused by the added NaCl salt affects the crystallization and agglomerate. NaCl ration processes cause problems in the phase change transition [11].

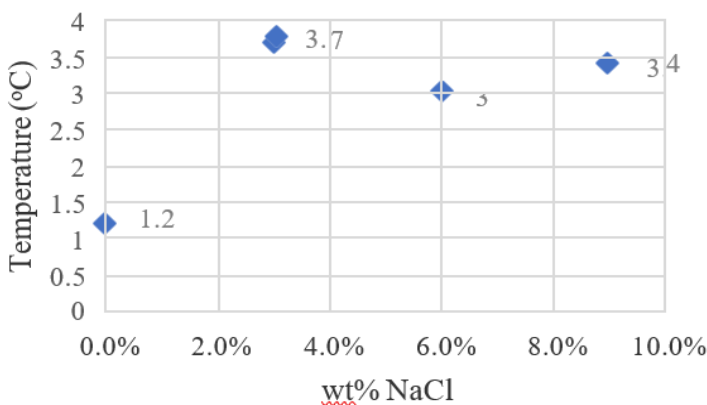


Fig. 5. Supercooling degrees

However, the 3% wt. NaCl solution had a higher degree of supercooling compared to the 6% and 9% wt. NaCl solutions. This is because the 3% solution has a lower initial temperature, resulting in slower freezing. This phenomenon is called the Mpemba effect, which states that a solution with a lower initial temperature freezes more slowly than a solution with a higher temperature [12]. This is also following the explanation given by Tan et al, that each difference in the initial temperature of the cooling solution will have a different cooling rate. This causes the solution with a lower initial temperature to have a higher supercooling [13]. The condition also presented by Wilson (2012) that the degree of supercooling is influenced by the volume and rate of cooling of the solution [14].

The latent heat of fusion of the eutectic wt.% NaCl solution is presented in Fig. 6. The addition of NaCl as a solute causes a decrease in the latent heat of fusion of the solution. The decrease in latent heat is proportional to the percentage of NaCl in the solution. The explanation given by Kumano et al, that the latent heat of fusion of aqueous solutions is directly proportional to the latent heat of water, the decrease in enthalpy due to freezing point depression, and the heat generated by variations in concentration due to the dilution effect in the solution [15], [16].

The addition of the percentage of NaCl into the aqueous solution causes a decrease in the freezing point, which will result in a decrease in enthalpy. This is what causes a decrease in the latent heat of fusion in the NaCl solution, which is getting bigger with the addition of the percentage of NaCl into the aqueous solution, as presented in Fig. 6

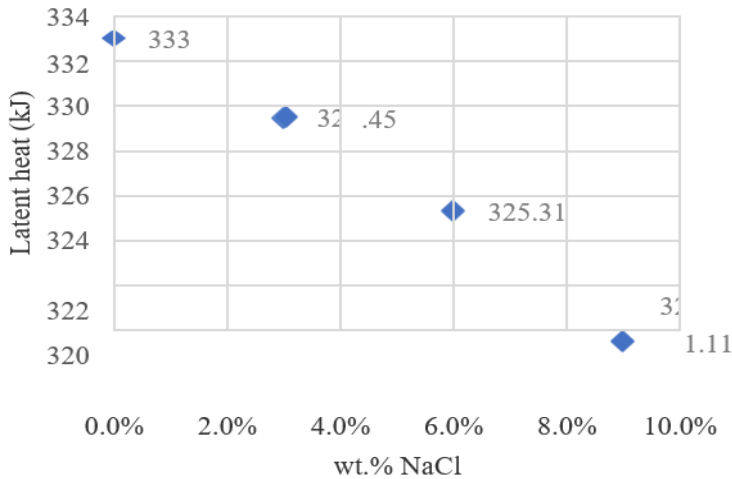


Fig. 6. Latent heat

3.1 Cold Storage Test Results

The cooling system must calculate the cooling load to determine the appropriate amount of cooling media needed to produce the desired cooling temperature. Cooling load is calculated using the Cooling Load Temperature Difference (CLTD) method from the ASHRAE Handbook Refrigeration [17]:

1. Product load

The product load is the heat load that comes from the product to be cooled, the product in the form of African catfish with a total weight of 5 kg, which must be lowered from 30 °C to 0 °C. Water content is the main constituent in fish, so the water content in fish significantly affects the thermophysical properties of the fish. Based on Rozi's research, fresh catfish have a water content of around 77.78±0.79% [16]. Temperature 0 °C is the freezing point of water. The initial freezing point of fish is slightly lower than the freezing point of pure water because of the dissolved substances in the fish. Based on the ASHRAE Handbook, the initial freezing point of fish is -2.2 °C [17]. Thus, fish stored at 0 °C will not freeze. Thus, the specific heat used is above the freezing point of 3.47 kJ/kg °C. Since fish do not freeze, the heat release consists only of sensible heat. The product load is 520kJ.

2. Transmission Load

The transmission load is the heat that enters the storage medium through the walls. The incoming heat is due to the temperature difference between the environment and the cold storage. According to BNP data for 2020, Indonesia's maximum temperature is in the range of 37-37.4 °C, which occurred in September. In calculating the transmission load, the highest ambient temperature is chosen so that the cooling system can maintain the room temperature when the ambient temperature is at its peak. This load is greatly influenced by the insulation layer used. This test uses a Styrofoam box as a storage medium. The Styrofoam used as an

insulation medium has a thermal conductivity of 0.03 kcal/hm² °C with a thickness of 2.0 cm. The absolute thermal conductivity value is 0,657 kcal/hm²°C. The surface area of the storage medium is 0,453m². Cold storage is used to maintain temperature for 24 hours. The total transmission load received for 24 hours is 1073,35 kJ.

3. Infiltration Load

The infiltration load caused by cold storage's opening and closing process depends on the time the door opens, which is expressed in the Decimal Portion. The number of cold storage doors is 1, with an opening and closing time of 3 seconds and the door opening for 5 minutes to enter fish. The process of opening and closing the door is only one time when putting fresh fish into cold storage operation time for 24 hours. The air density in cold storage at a temperature of 0 °C was 1.2922 kg/m³. Based on the Psychrometric Chart at 1atm, ambient temperature at 37.4 °C with an average humidity (RH) of 61%, the enthalpy value of 102 kJ/kg was obtained. The temperature in cold storage used the maximum temperature for fish storage, which was 0 °C at 90% relative humidity, so an enthalpy of 8.5 kJ/kg was obtained. If the airspeed is 15 mph or 6.71 m/s based on equation 2.9, then the magnitude of the infiltration load is 20,43 kJ.

Based on the thermal characteristics of each PCM. PCM made of 9% wt NaCl was chosen as the cooling medium because it has the right application temperature for cold storage, high latent heat and density, and the degree of supercooling that can be handled well. The total cooling load that occurs in cold storage of fresh fish is the sum of product load, transmission load, infiltration load, and safety factor with a magnitude of 15%. Then the total cooling load is 1900.43kJ. To determine the number of PCM need to be used as a cooling medium [18]. It is assumed that the cooling medium absorbs the entire cooling load. The total heat absorbed is the total heat that the PCM cooling medium can absorb, consisting of sensible heat absorption and latent heat absorption. In applications for cold storage, PCM is put in a container (pack) with a capacity of 0.7 kg. The ability of 1 pack of PCM to absorb heat is 253.98 KJ/pack. Then the number of PCM needed is 8 packs.

After knowing the amount of cooling media needed, a testing process is carried out whose results are implemented into the graph in Fig. 7. The initial PCM temperature is -22°C, then the temperature rises drastically to -16.8°C in just 2 minutes. This drastic increase in temperature is due to the large infiltration load received by the PCM after leaving the freezer, and also the infiltration load when filling fish into cold storage. After the cold storage is closed, the PCM temperature decreases slowly because the absorbed heat load only comes from the product and transmission loads.

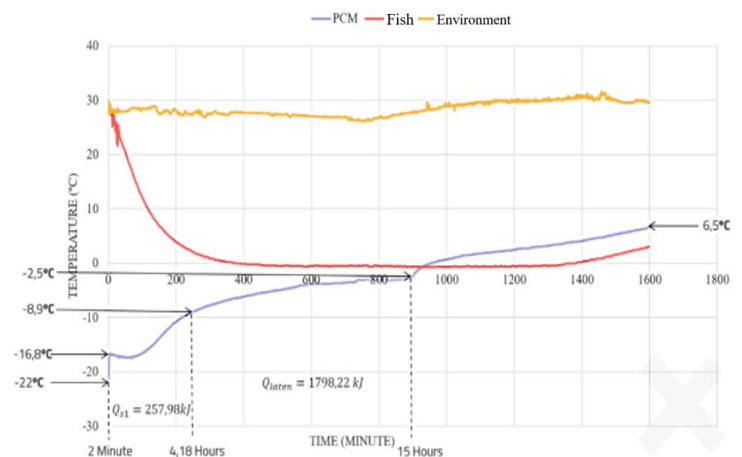


Fig. 7. Cold storage test results

An exponential line shows this high-temperature change on the graph. This happens because PCM absorbs sensible heat. The following process is the absorption of latent heat, which is indicated by a linear line and the temperature of - 8.9 °C to -5.5 °C. The temperature changes that occur are small. The absorbed heat is used

more to change the PCM phase from solid to liquid. After the PCM changes to a completely liquid phase, the only heat absorbed is sensible heat. This sensible heat absorption process in the liquid phase takes place from a temperature of $-5.5\text{ }^{\circ}\text{C}$ to a temperature of $6.5\text{ }^{\circ}\text{C}$ when the equilibrium temperature between PCM and fish is reached. This process occurs for 1146 minutes. When the PCM temperature and fish were both right at $-0.6\text{ }^{\circ}\text{C}$ at the same time, the PCM temperature continued to rise, but the fish temperature remained around $-0.6\text{ }^{\circ}\text{C}$ and even dropped to $-0.7\text{ }^{\circ}\text{C}$. This indicates a time lag that causes the temperature of the fish on the edge that is in contact with the PCM to be different from that of the fish in the center of the cold storage. In this case, the temperature of the fish measured in the test is the fish in the cold storage center. So that when the fish undergoes a cooling process, the fish on the edge will lower the temperature first and then the fish in the center.

The following analysis is aimed at fish. Based on Fig. 7, it can be seen that the initial temperature of the fish is at $28.9\text{ }^{\circ}\text{C}$. After the fish is put in cold storage, the temperature of the fish slowly decreases. However, the fish are still alive so they can make some movements. As a result, the temperature of the fish rises slightly and is then lowered again by the PCM. This continued for 35 minutes, with marked fluctuations in the temperature chart of the fish. Furthermore, the temperature of the fish decreased until it reached a temperature of $0.9\text{ }^{\circ}\text{C}$. This point is reached in 300 minutes or 5 hours with a temperature decrease rate of $5.48\text{ }^{\circ}\text{C}$.

The temperature of the fish was kept constant at around $0\text{ }^{\circ}\text{C}$ for 1160 minutes, or about 19 hours and 20 minutes. After that, the temperature of the fish rose above $0.9\text{ }^{\circ}\text{C}$. The temperature change occurred quite quickly and ended at a temperature of $3.1\text{ }^{\circ}\text{C}$ for 138 minutes, or 2 hours and 18 minutes. The use of cold storage with cooling media in a solution of 9% wt NaCl can reduce and maintain fish at a temperature of $0\text{ }^{\circ}\text{C}$. Temperature for microbial growth is classified as thermophilic ($35\text{--}55\text{ }^{\circ}\text{C}$), mesophilic ($10\text{--}40\text{ }^{\circ}\text{C}$), and psychrophilic ($-5\text{--}15\text{ }^{\circ}\text{C}$) [19]. Based on this, the cooling process in fish will only inhibit the growth of thermophilic and mesophilic microbes so that the growth of psychrophilic bacteria can still develop. Psychrophilic bacteria can cause spoilage in fish, but this type of bacteria is not pathogenic, so it does not cause disease. So, the process of storing fish at a temperature of $0\text{ }^{\circ}\text{C}$ is done to inhibit pathogenic bacteria. In addition to inhibiting the growth of pathogenic bacteria, the cooling process also reduces the rate of enzymatic and microbiological changes.

3.2 Organoleptic Test Results

The number of fish that have been cooled is 58. With a random sampling technique using the Slovin formula to find samples, with a precision value of 90%, the number of test samples is 37 individuals. Furthermore, organoleptic tests were carried out on 37 samples taken randomly with an organoleptic rating scale for fresh fish referring to SNI 01-2346-2006.

As a result of the test, the organoleptic value interval for fresh is 8.25–8.45. According to SNI 01-2346-2006, the final organoleptic value of fresh fish is taken from the smallest value of 8.25 and rounded up to 8. So, based on the organoleptic assessment for fresh fish referring to SNI 01-2346-2006, with a 95% confidence level and an error rate of 5%, and an organoleptic value of 8. This value indicates that fish are included in fresh fish with good quality and quality as highlighted by others [20], and it fits the Indonesian Export Quality Certification [21].

4 Conclusions

The thermal properties of the tested solutions were characterized as follows: pure water exhibited a freezing point of $0.6\text{ }^{\circ}\text{C}$, a supercooling degree of $1.2\text{ }^{\circ}\text{C}$, a density of 1000 kg/m^3 , and a latent heat of fusion of 333 kJ/kg . The 3% wt. NaCl solution demonstrated a freezing point of $-1.8\text{ }^{\circ}\text{C}$, a supercooling degree of $3.7\text{ }^{\circ}\text{C}$, a density of 1023 kg/m^3 , and a latent heat of 329.45 kJ/kg . The 6% wt. NaCl solution recorded a freezing point of $-3.7\text{ }^{\circ}\text{C}$, a supercooling degree

of $3.0\text{ }^{\circ}\text{C}$, a density of 1046 kg/m^3 , and a latent heat of 325.31 kJ/kg . The 9% wt. NaCl solution, selected as the optimal PCM, exhibited a freezing point of $-5.8\text{ }^{\circ}\text{C}$, a supercooling degree of $3.4\text{ }^{\circ}\text{C}$, a density of 1069 kg/m^3 , and a latent heat of 321.11 kJ/kg . Increasing NaCl concentration systematically lowered the freezing point and latent heat while increasing the density and supercooling degree [22]. Application of the 9% wt. NaCl eutectic solution in a practical cold storage test successfully maintained fish at approximately $0\text{ }^{\circ}\text{C}$ for 24.3 hours, reducing the initial temperature from $28.4\text{ }^{\circ}\text{C}$ to a minimum of $-0.7\text{ }^{\circ}\text{C}$. Organoleptic assessment after 25 hours indicated a freshness score of 8 with 95% confidence, meeting the standards required for Export Quality Certification for Small and Medium Enterprises (SMEs) in Indonesia. Overall, the results confirm the feasibility of utilizing sodium chloride-based eutectic solutions as an effective, sustainable cold storage medium for the seafood industry.

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