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Ankit Kumar Deshmukh
Ph.D., Dairy Engineering Division, ICAR- National Dairy Research Institute, Karnal, Haryana, India

PS Minz
Senior Scientist, Dairy Engineering Division, ICAR-National Dairy Research Institute, Karnal, Haryana, India

Rohit HK
Ph.D., Dairy Engineering Division, ICAR- National Dairy Research Institute, Karnal, Haryana, India

Sumit Mehta
Ph.D., Dairy Engineering Division, ICAR- National Dairy Research Institute, Karnal, Haryana, India

Anubhuti Dwivedi
M. Tech, Food Science Technology and Nutrition, Sambalpur University, Odisha, India

Corresponding Author:
Ankit Kumar Deshmukh
Ph.D., Dairy Engineering Division, ICAR- National Dairy Research Institute, Karnal, Haryana, India

Thermophysical Characterization and Response Surface Modeling of Aqueous Glycerol-NaCl Phase Change Materials

Ankit Kumar Deshmukh, PS Minz, Rohit HK, Sumit Mehta and Anubhuti Dwivedi

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Abstract
Developing effective low-temperature thermal storage requires precise control over phase change material (PCM) properties. This study engineered a multi-component PCM system by blending glycerol, sodium chloride (NaCl), and water, evaluating them via a Design of Experiments (DOE) strategy built on Response Surface Methodology (RSM). Solute concentrations for both glycerol and NaCl ranged from 0-5%. Batches were prepared using ultrasonic agitation to determine how these variables dictate freezing thresholds, viscosity variations, specific heat, latent heat capacity, and thermal conductivity. Our laboratory data prove that salt content controls the freezing threshold. Adding 5% NaCl depressed the freezing point to -5.85°C because ions physically disrupt the water crystallization lattice, which also triggered a distinct supercooling pocket down to -6.5 °C. Meanwhile, glycerol drives internal fluid friction and thermal transport. It pushes viscosity along a parabolic curve to a 3.86 cP peak via hydrogen bonding networks and forces a linear decline in thermal conductivity to 0.5976 W/mK. Combined, these solutes lowered specific heat to 3.926 J/g°C and latent heat to 300.6 J/g. Strong model accuracy (R^2 = 0.9642) provides a highly dependable optimization framework for cold-chain logistics engineering.

Keywords: Phase Change Material (PCM), Response Surface Methodology (RSM), Low-Temperature Thermal Storage, Glycerol–Sodium Chloride Mixture, Cold-Chain Logistics

Introduction
Thermal energy storage (TES) systems, cold-chain preservation, and specialized cryo-storage rely heavily on phase change materials (PCMs) capable of capturing and releasing substantial latent heat loads during phase transitions (Yang *et al*, 2024) [15]. Aqueous PCMs are frequently selected for subzero applications due to their exceptional latent heat capacities and minimal environmental impact. Even so, the rigid, fixed freezing point of unrefined water introduces engineering roadblocks when an application demands extended liquid-phase stability below 0ºC or customized, gradual crystallization pathways (Marques *et al*., 2025) [12]. To counter these limitations, chemical formulation adjustments using multi-solute solutions—particularly mixtures combining organic non-electrolytes with mineral salts—have become a vital pathway for tailoring thermophysical properties. This investigation documents the selection, multi-variable formulation, and algorithmic optimization of a ternary glycerol-salt-water PCM setup engineered for versatile solid- and liquid-phase applications. Within this matrix, glycerol acts as an organic, non-electrolyte cryoprotectant that modifies the foundational hydrogen-bonding network of the water base. Simultaneously, sodium chloride (NaCl) functions as an ionic electrolyte that depresses water activity and triggers significant colligative freezing point depression as suggested by Ouaouja *et al*. (2025) [13]. This localized pull lowers the chemical potential of the solvent and reduces water activity. To initiate freezing, the system must exert excess energy to separate water molecules from these ionic hydration forces. This behavior lowers the equilibrium freezing temperature and often creates a distinct supercooling window (Lamas *et al*., 2022) [10]. While the standalone physical impacts of these individual chemical agents are well-established, their concurrent, non-linear interactions across viscosity, thermal conductivity, specific heat, and latent heat characteristics necessitate an integrated multi-variable optimization approach.

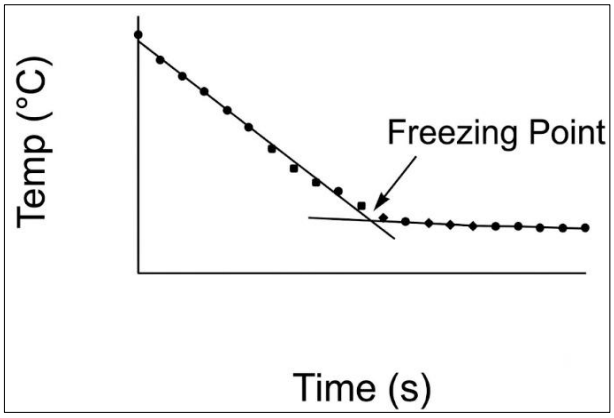
To address this challenge, a Design of Experiments (DOE) strategy utilizing Response Surface Methodology (RSM) was implemented. This setup maps the linear and quadratic dependencies that surface when manipulating raw concentrations of glycerol and NaCl. The resulting thermophysical parameters were validated using standard empirical instrumentation, such as Brookfield rotational viscometer configurations, alongside verified predictive equations like the weighted mass-average approach for heat capacity and the Bruggeman iterative model for effective thermal conductivity. Ultimately, this modeling effort charts the inherent trade-offs between bulk thermal storage capacity and fluid transport behavior, establishing a reliable toolkit for customizing subzero PCM configurations.

Material methodology

To evaluate the Thermophysical Characterization and Response Surface Modeling of Aqueous Glycerol-NaCl Phase Change Materials, a series of multi-component aqueous mixtures were developed and analyzed. A Design of Experiments (DOE) framework was executed via Design-Expert 13 software, leveraging Response Surface Methodology (RSM) to map out how changing the glycerol concentration (A) and sodium chloride (NaCl) content (B) individually and jointly alters the system. Each specific formulation was mixed in a 100 mL batch and processed with an ultrasonic stirrer to guarantee a completely uniform distribution of the solutes throughout the water matrix. Following homogenization, these samples were immediately routed to the thermal and rheological testing protocols outlined below.

Freezing Point Determination (Cooling Curve Analysis)

The phase change temperature of each aqueous blend was determined by evaluating its characteristic thermal history during solidification. For each test, a 100 mL sample was transferred into a glass container equipped with a calibrated T-type thermocouple positioned securely at its geometric center. This entire assembly was then exposed to a low-temperature cooling bath set at -18°C. A digital data logger captured the transient temperature reduction continuously at 5-second intervals.



Plotting these data points over time provided a clear visual map of the solidification progress. The freezing point was pinpointed by locating the distinct thermal plateau on the cooling curve (as shown in image below). This flat region represents the phase equilibrium zone where the sensible cooling of the liquid temporarily stops because latent heat is being released as ice crystals actively nucleate and grow. To confirm the accuracy of this plateau identification, all experimental runs were performed in triplicate.

Viscosity

The viscosity of the glycerol-salt-water mixture was measured using a Brookfield Viscometer with an SP1 spindle at 100 RPM. A 100 ml sample was maintained at 20°C using a temperature-controlled bath. The viscometer was calibrated, and the spindle was immersed to the recommended depth. After stabilizing, viscosity readings were recorded and repeated three times for accuracy. The average viscosity was calculated and compared with literature values to validate the measurement, ensuring reliable characterization of the mixture (Grazi *et al*. 2025) [7]

Specific heat and latent heat

Jochem and Körber (1987) [9] proposed a method to estimate the specific heat capacity and latent heat of glycerol-salt-water mixtures using a weighted average approach. In this method, the overall specific heat capacity (Cp) is calculated by summing the individual contributions of each component, weighted by their mass fractions, using the equation:

$$C_{p,\text{mixture}} = \left(\frac{m_{\text{Gly}}}{m_{\text{total}}}\right) C_{p,\text{Gly}} + \left(\frac{m_{\text{Salt}}}{m_{\text{total}}}\right) C_{p,\text{Salt}} + \left(\frac{m_{\text{Water}}}{m_{\text{total}}}\right) C_{p,\text{Water}}$$

Similarly, the latent heat (L_f) of the mixture can be estimated using the same weighted average principle:

$$L_{f,\text{mixture}} = \left(\frac{m_{\text{Gly}}}{m_{\text{total}}}\right) L_{f,\text{Gly}} + \left(\frac{m_{\text{Salt}}}{m_{\text{total}}}\right) L_{f,\text{Salt}} + \left(\frac{m_{\text{Water}}}{m_{\text{total}}}\right) L_{f,\text{Water}}$$

This approach is justified by the extensive nature of specific heat and latent heat, where the total thermal capacity of a mixture is the sum of its individual components. Their work demonstrated that, for ternary systems like these, the predicted values align closely with experimental observations for glycerol-salt aqueous solutions, confirming the reliability of the weighted average method for thermal analysis.

Thermal conductivity

The effective thermal conductivity (λeff) of glycerol-salt-water mixtures was estimated using the Bruggeman model, as suggested by Anh Thu Phan *et al*. (2022) [14]. The model accounts for the volume fractions and thermal conductivities of individual components, expressed as:

$$f_{\text{Gly}} \frac{\lambda_{\text{Gly}} - \lambda_{\text{eff}}}{\lambda_{\text{Gly}} + 2\lambda_{\text{eff}}} + f_{\text{Salt}} \frac{\lambda_{\text{Salt}} - \lambda_{\text{eff}}}{\lambda_{\text{Salt}} + 2\lambda_{\text{eff}}} + f_{\text{Water}} \frac{\lambda_{\text{Water}} - \lambda_{\text{eff}}}{\lambda_{\text{Water}} + 2\lambda_{\text{eff}}} = 0$$

Where f_{Gly}, f_{Salt} f_{Water} are the volume fractions of glycerol, salt, and water, respectively, and λ_{Gly}, λ_{Salt} and λ_{Water} are their respective thermal conductivities. The equation was solved iteratively to determine λ_{eff}. This method is justified as it effectively accounts for phase distribution and has been validated against experimental data for multicomponent salt-based PCMs, ensuring reliable thermal transport predictions.

Result and Discussion

Temperature profile analysis

The temperature profile curves demonstrate (Fig 1) the cooling behavior of different glycerol and glycerol-salt solutions, highlighting the influence of solute concentration

on freezing characteristics. The recorded freezing points for the tested solutions show a clear trend: increasing glycerol concentration lowers the freezing point, and the addition of salt further enhances this effect. For instance, a 2.5% glycerol solution (G2.5S0) froze at -1.9°C, whereas adding 2.5% salt (G2.5S2.5) further reduced the freezing point to -4.3°C. Similarly, a 5% glycerol solution (G5S0) froze at -3.8°C, while the combination of 5% glycerol and 5% salt (G5S5) exhibited a freezing point of -5.85°C. The temperature curves also indicate supercooling, particularly in salt-containing solutions, where crystallization initiates at temperatures lower than the equilibrium freezing point. For example, G5S5 experienced supercooling down to -6.5°C before freezing. Additionally, the cooling rate after freezing was noticeably slower in solutions with higher solute concentrations, suggesting a delayed phase transition due to solute interference in ice crystallization.

The observed freezing point depression in glycerol and glycerol-salt solutions is primarily due to the colligative properties of solutes, which reduce the chemical potential of water and hinder ice nucleation. Glycerol, a non-electrolyte, disrupts hydrogen bonding among water molecules, preventing the formation of an ice lattice at higher temperatures (Bernacka *et al.* 2021) [2]. The addition of salt amplifies this effect because it ionizes into Na^+ and Cl^- ions, which create strong hydration shells around water molecules, further obstructing the freezing process (Li *et al.* 2021) [11]. This explains, salt-containing solutions exhibit lower freezing points and greater supercooling effects compared to pure glycerol solutions. Moreover, the gradual temperature decline post-freezing suggests a more dispersed crystallization process, which could be advantageous in applications such as thermal energy storage and cryopreservation, where controlled freezing is crucial. The results confirm that glycerol-salt mixtures provide greater freezing suppression and improved thermal stability, making them suitable for environments requiring extended subzero liquid-phase stability.

Freezing point

The effect of glycerol and salt concentrations on the freezing point of multicomponent PCM solutions was analyzed using response surface methodology (RSM), as shown in Fig. 2(a). The freezing point of glycerol-salt-water solutions was significantly influenced by the concentration of salt and glycerol, as evident from the response surface analysis. Salt concentration (B) had the most dominant effect, with an F-value of 105.58 ($p < 0.0001$), showing a strong freezing point depression due to the ionic disruption of ice crystallization (Lamas, Vega, and Noya 2022) [10]. Increasing salt concentration led to a progressive decrease in freezing temperature, reaching a minimum of -5.85°C at 5% salt. Glycerol concentration (A) also contributed to freezing point depression, but its effect was weaker compared to salt ($F = 51.06$, $p = 0.0002$), as glycerol primarily reduces water activity and affects hydrogen bonding in water molecules (Guo *et al.* 2025) [8]. The interaction effect (AB) was found to be insignificant ($p = 0.2267$), indicating that the combined impact of salt and glycerol is mostly additive rather than synergistic. These findings suggest that for precise freezing point control in thermal energy storage applications, salt concentration should be the primary tuning factor, while glycerol can be used for minor adjustments. The quadratic model for freezing point prediction was found to be statistically significant ($p < 0.0001$), with an R^2 value of 0.9642, indicating a good fit. However, the predicted R^2 (0.6730) was lower than the adjusted R^2 (0.9386), suggesting some block effects or variability in experimental data. The final regression equation in coded terms is:

$$\text{Freezing Point} = \mathbf{0.3757}A^2 + \mathbf{1.21}B^2 - \mathbf{0.3025}AB - \mathbf{1.33}A - \mathbf{1.91}B - \mathbf{4.08}$$

This equation effectively predicts the freezing point within the tested range and highlights the dominant influence of salt concentration over glycerol.

Specific heat

The specific heat capacity of aqueous glycerol-NaCl solutions was analyzed by considering glycerol (A) and NaCl (B) concentrations as independent variables. The specific heat values ranged from 3.926 J/g°C to 4.18 J/g°C, with the maximum (4.18 J/g°C) observed for pure water (0% glycerol, 0% NaCl) and the minimum (3.926 J/g°C) at the highest solute concentrations (5% glycerol, 5% NaCl). This trend indicates a consistent reduction in specific heat with increasing solute content, as illustrated in the Response Surface Methodology (RSM) plot (Fig. 2(b)). This trend aligns with previously reported observations, where the incorporation of additional solutes into phase change systems led to a decrease in specific heat values, attributed to the reduced ability of the modified system to store thermal energy per unit mass (Cheng *et al.* 2023) [3]. ANOVA analysis confirmed the statistical significance of these effects. NaCl concentration had the most substantial impact (F-value = 722.15, $p < 0.0001$), while glycerol also played a significant role (F-value = 318.00, $p < 0.0001$). Their interaction (F-value = 18.12, $p = 0.0038$) was also significant, indicating a combined influence on specific heat. However, the quadratic terms were not statistically significant, suggesting a primarily linear relationship.

The RSM plot (Fig. 2(b)) shows a clear decline in specific heat with increasing solute concentrations, with NaCl exhibiting a stronger effect due to ion-solvent interactions. Glycerol also contributes to the reduction but to a lesser extent. The statistical analysis confirms the model's validity, emphasizing that specific heat decreases as glycerol and NaCl concentrations increase, with NaCl having a dominant effect. These findings are crucial for applications involving aqueous electrolyte-organic mixtures, where thermal properties significantly impact system performance.

Latent heat

The Response Surface Methodology (RSM) plot (Fig. 2(c)) illustrates a declining trend in latent heat with increasing concentrations of glycerol and NaCl. As solute concentration increases, the latent heat of fusion decreases due to the disruption of water's hydrogen bond network by both glycerol and salt molecules (Bashir *et al.* 2023) [1]. Among the two solutes, NaCl has a more pronounced effect, as observed in the steeper decline along the NaCl axis in the RSM plot. This trend is statistically confirmed by the ANOVA analysis, where NaCl concentration (Factor B) shows the strongest influence, followed by glycerol (Factor A). The interaction effect (AB) is also statistically significant, indicating that the combined presence of glycerol and salt enhances the reduction in latent heat. The quadratic terms, however, are not significant, suggesting a primarily linear dependency. Examining the experimental

values, the latent heat varied between 300.6 J/g and 334 J/g. The highest value (334 J/g) was recorded for pure water (0% glycerol, 0% NaCl), confirming that the absence of solutes allows maximum energy absorption during phase change. Conversely, the lowest value (300.6 J/g) was observed at the highest solute concentration (5% glycerol, 5% NaCl), demonstrating the cumulative impact of both glycerol and NaCl in reducing latent heat. This behavior is consistent with the known thermophysical effects of solute addition, where both glycerol's molecular structure and NaCl's ionic nature interfere with water's phase transition.

Viscosity

The Response Surface Methodology (RSM) plot (Fig. 2(d)) shows a parabolic trend in viscosity, increasing with glycerol and NaCl concentrations before slightly decreasing at higher levels. This trend arises from glycerol's hydrogen bonding, which enhances viscosity, and NaCl's ion-solvent interactions, which initially strengthen fluid resistance but weaken at higher concentrations. The ANOVA results confirm that glycerol ($F = 49.57$, $p = 0.0002$) has the strongest influence on viscosity, followed by NaCl ($F = 38.83$, $p = 0.0004$). The interaction term (AB) is also significant ($F = 6.58$, $p = 0.0373$), indicating a nonlinear effect. The quadratic term A^2 ($F = 10.05$, $p = 0.0157$) further supports the observed parabolic trend.

Viscosity values ranged from 1.02 to 3.86 cP, with the lowest (1.02) at 0% glycerol and NaCl and the highest (3.86 cP) at moderate solute concentrations (~3–4% glycerol and NaCl). Similar trend of increasing viscosity in glycerol system by addition of salt has been observed by study done by D'Agostino, Davis, and Abbott (2021) [5]. This suggests viscosity peaks at intermediate concentrations before slightly decreasing due to molecular crowding effects. Viscosity is primarily influenced by glycerol, with NaCl also contributing. The statistical analysis confirms a parabolic trend, where moderate solute concentrations maximize viscosity before a slight decline. These findings are useful for optimizing viscosity in industrial applications requiring controlled fluid dynamics.

Thermal conductivity

The thermal conductivity of aqueous glycerol-NaCl solutions exhibits a linear decreasing trend, as depicted in the Response Surface Methodology (RSM) plot (Fig. 2(e)). The highest thermal conductivity (0.606 W/m·K) was observed for pure water (0% glycerol, 0% NaCl), while the lowest (0.5976 W/m·K) occurred at 5% glycerol and 5% NaCl. This reduction is attributed to the disruption of the hydrogen bond network in water by glycerol and NaCl, which limits the transfer of thermal energy (Dashnau *et al.* 2006) [4]. Also The reduction in thermal conductivity observed with glycerol addition can be attributed to its naturally low thermal conductivity and the increased intermolecular resistance it introduces, which together hinder the efficient transfer of heat through the medium (Ge and Zheng 2020) [6]. The ANOVA results indicate that the model is statistically significant ($F = 129.97$, $p < 0.0001$). However, glycerol ($F = 259.15$, $p < 0.0001$) has a dominant influence, whereas NaCl's effect is negligible ($F = 0.7852$, $p = 0.3964$). This suggests that glycerol primarily governs the decline in thermal conductivity, while NaCl has an insignificant contribution. From the experimental values, the thermal conductivity decreases as glycerol concentration increases, with a slight additional reduction from NaCl. The linear nature of the model confirms that no significant interaction or quadratic effects influence thermal conductivity. These findings are important for applications where controlling heat transfer efficiency in aqueous systems is necessary, particularly in thermal management and energy storage solutions.

Table 1: Design of experiment (Glycerol and salt) for liquid phase

	Factor 1	Factor 2	Response 1
Run	**A: Gly**	**B: Salt**	
	%	%	
1	30	3	
2	40	1	
3	30	3	
4	40	3	
5	20	3	
6	40	5	
7	30	3	
8	30	3	
9	20	5	
10	20	1	
11	30	1	
12	30	5	
13	30	3	

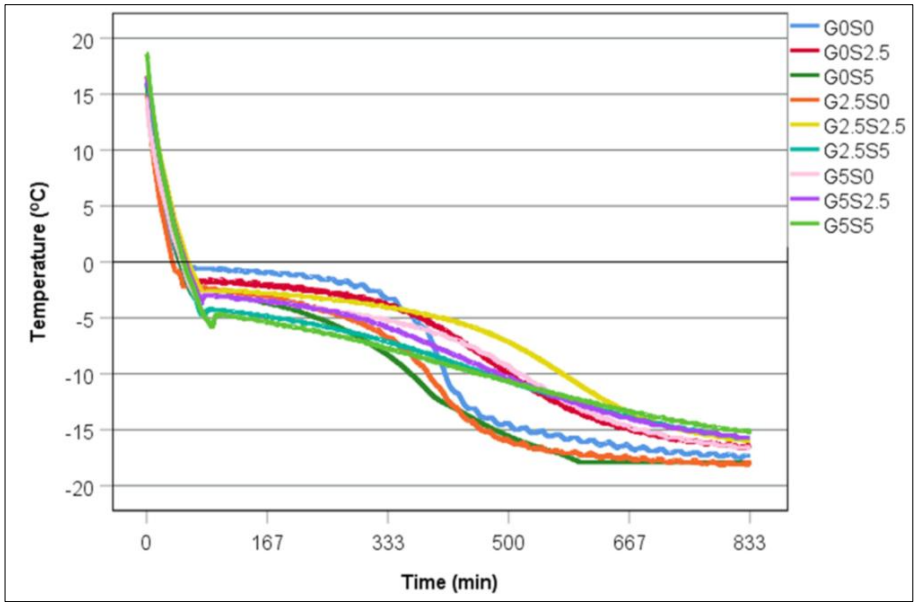


Fig 1: Temperature profile of Multicomponent PCM solution

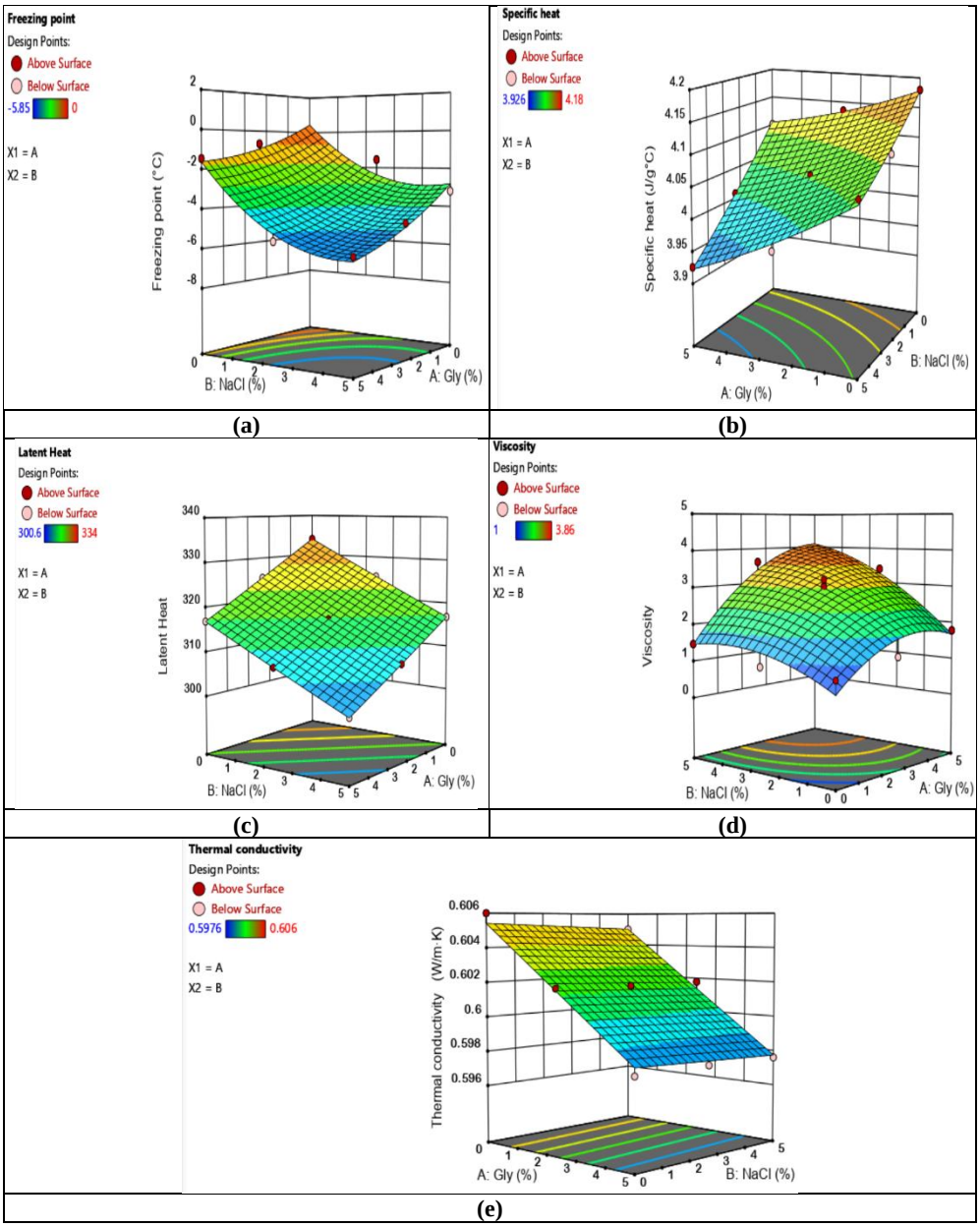


Fig 2: Effect of glycerol and salt concentration on (a) freezing point (b) specific heat (c) Latent heat (d) Viscosity (e) Thermal conductivity of multicomponent PCM solution

Conclusion

This research successfully evaluated, formulated, and mapped a multi-component glycerol-salt-water phase change material (PCM) using Response Surface Methodology (RSM). The resulting empirical models displayed strong statistical validity, defining the distinct physical mechanisms and structural shifts that glycerol and NaCl introduce to aqueous thermal media.

The key outcomes established from this work include:

- **Freezing Point Modulation:** Dissolved salt concentration functions as the dominant control variable for subzero liquid maintenance, driving the freezing point down to a minimum of -5.85°C at 5% NaCl due to intense ionic hydration interactions. This combined solute action halts rapid ice nucleation and induces steady supercooling down to -6.5 °C, delaying the overall phase transition.
- **Viscosity Variations:** Fluid dynamics and internal shear resistance are governed principally by the glycerol fraction. Viscosity displays a distinct parabolic curve, peaking at 3.86cP at intermediate loading levels (3-4%) due to physical molecular crowding and complex hydrogen-bonding configurations.
- **Thermal Transport Declines:** Effective thermal conductivity drops linearly to a minimum value of 0.5976W/m K. This shift is driven almost entirely by the low native thermal conductivity of pure glycerol and the corresponding increase in intermolecular thermal resistance, while the impact of NaCl remains statistically minor.
- **Energy Density Reductions:** Incorporating these solute combinations creates an unavoidable trade-off in overall thermal performance, reducing the specific heat capacity to 3.926 J/g°C and depressing the latent heat value to 300.6 J/g at peak solute concentrations (5% glycerol, 5% NaCl) because of the systematic disruption of the water lattice.

In conclusion, the derived regression models provide a dependable predictive framework for industrial scaling. By utilizing inorganic salt as a primary control for phase change temperatures and organic glycerol as a secondary tuning agent for viscosity and heat distribution, these multi-component solutions can be customized for cryopreservation, refrigeration logistics, and low-temperature thermal energy storage systems.

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