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TECHNICAL PAPER

Assessment of Solidification Parameters of Salts and Metals for Thermal Energy Storage Applications Using IHCP-Energy Balance Combined Technique

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Abstract Solar energy storage technologies have proved to be promising in terms of providing uninterrupted power supply. The phase change materials (PCMs) with their higher heat storage capacity are more efficient than sensible heat storage materials. In this study, a new method for thermal analysis of PCM salts was proposed. The method was based on the estimation of heat flux at the mold–salt interface using solution to inverse heat conduction problem and characterizing the salt using a simplified energy balance method. It was advantageous over other computer-aided cooling curve analysis methods as it eliminated the use of curve fitting approach involved in baseline calculations. KNO_3 and $NaNO_3$ salts were used to validate this method. The solidification parameters like cooling rate, liquidus and solidus temperatures, solidification time and latent heat were assessed. The results of the analysis were in agreement with the data reported in the literature.

Keywords Energy storage · Thermal analysis · Solidification · Inverse technique · Energy balance

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1 Introduction

The world is facing an environmental and energy crisis due to increased population and industrialization. This can be taken care by utilizing the renewable energy resources available, solar energy being one of them. The only limitation associated with this resource is its instability and intermittent nature which creates a misbalance between the energy supply and demand. In order to overcome this limitation, thermal energy storage technologies (TES) have become more attractive among the researchers. In TES systems, latent heat thermal energy storage systems (LHTES) using phase change materials have gained more recognition as it has higher energy storage density as compared to the sensible heat thermal energy storage systems (SHTES) and that too at a constant temperature with phase transformations. For successful development of LHTES materials, an accurate index for the solidification parameters of the phase change materials (PCM) is needed [1]. The methods that are being used for this characterization are differential scanning calorimetry (DSC) technique; thermal history (T-history) method and computer-aided cooling curve analysis methods (CACCA). The limitations associated with DSC are its small sample size and heating and cooling rates driven results which makes it inappropriate to characterize inhomogeneous materials [2]. T-history method overcomes these limitations, but the problem of selecting the appropriate reference materials for high-temperature application limits its usage only for low-temperature materials [3].CACCA techniques have proved suitable as compared to the above discussed techniques and can be used for materials of all temperature ranges [4]. The only drawback is the method of baseline calculations using fitting techniques which affects the results obtained. In the present work, a technique which combines inverse heat

conduction problem (IHCP) technique with a simple energy balance equation has been utilized to calculate solidification parameters.

2 Materials and Methods

The salt sample was melted in a graphite crucible (70 m × 70 mm) with a thickness of 11.67 mm, thermally insulated from the top and bottom. The mass of KNO_3 sample was 0.183 kg, and the mass of $NaNO_3$ sample was 0.138 kg. Three thermocouples were used, T1 at the Centre, T2 at 2 mm from the outer surface of the mold and another T3 at 2 mm from the inner surface of the mold. Calibrated 1 mm K-type Inconel sheath thermocouple was used for characterizing salts. A scanning frequency of 2 Hz was used for temperature data acquisition. NI USB 9213 data acquisition system was used to process the data. The experimental setup used in this process is shown in Fig. 1a.

Inverse heat conduction problem simulation software (TmmFe Inverse Solver, Thermet solutions, Pvt. Ltd., Bangalore) was used to estimate the spatially dependent salt/mold interfacial heat flux transients. The inputs to the solver were the thermal history of the mold near to the inner surface and the thermophysical properties of the mold. The insulating boundary conditions with heat flux (q) = 0 were applied at the top and bottom. A convective boundary condition with heat transfer coefficient (h) of 10 W/m^{2}K was applied at the outer surface of the mold with ambient temperature (T_0) taken as 30 °C. A four-node quadrilateral element was used to represent a cylindrical axisymmetric model of the mold thickness (L). The model was made with a mesh size of 70 × 70 with 4900 elements. The inner surface of the mold was taken as the unknown heat flux boundary where the heat flux was calculated as shown in Fig. 1b. A convergence limit of 1E − 6 was used during the simulation. Using the post-processing feature, an estimated temperature data was obtained at T2 which was near to the outer surface of the mold. These data were compared with the measured thermocouple temperature data to confirm the accuracy of the model and the estimated heat flux. Using the heat flux (q) obtained, the total heat released into the mold was calculated by multiplying it by the area of heat exchange (A). Using the energy balance equation at the inner surface of the mold, the rate of heat released at every time step during the solidification range (Q_{inst}) could be calculated using Eq. 1. The integration of this Q_{inst} during solidification divided by the weight of the sample produced the measured value of the latent heat of solidification per unit weight (H) as represented in Eq. 2.

$$Q_{\text{inst}} = q \cdot A - m \cdot c_p \cdot \frac{\mathrm{d}T}{\mathrm{d}t} \tag{1}$$

$$H = \frac{Q_{\text{total}}}{m}, \quad Q_{\text{total}} = \int_{\text{tss}}^{\text{tes}} Q_{\text{inst}} \tag{2}$$

where m = mass of the sample (kg), A = area of heat exchange (m^2), C_p = average specific heat of the sample (kJ/kgK), dT/dt = cooling rate of the sample (°C/s), and H = latent heat (kJ/kg).

3 Results

The CACCA analysis for KNO_3 and $NaNO_3$ was carried out using the IHCP-energy balance method to obtain the solidification parameters. The solidification path of both salts and the thermal history of the mold were obtained. These are shown in Fig. 2a, b where T1 represents the center thermocouple data, T2 and T3 represent the mold temperature near to the outer surface and the inner surface, respectively.

The cooling rate curves were superimposed on the cooling curves for KNO_3 and $NaNO_3$ in Figs. 3a, b, respectively. The liquidus and solidus points have been marked as E1 and E2, respectively, on the cooling rate curves of the salts.

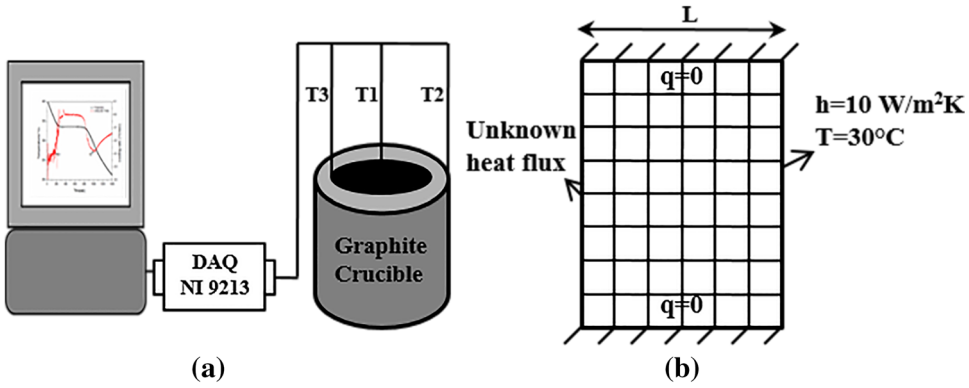

Fig. 1 **a** Schematic sketch of the IHCP–energy balance experimental setup. **b** Schematic sketch of the IHCP model used for unknown heat flux estimation

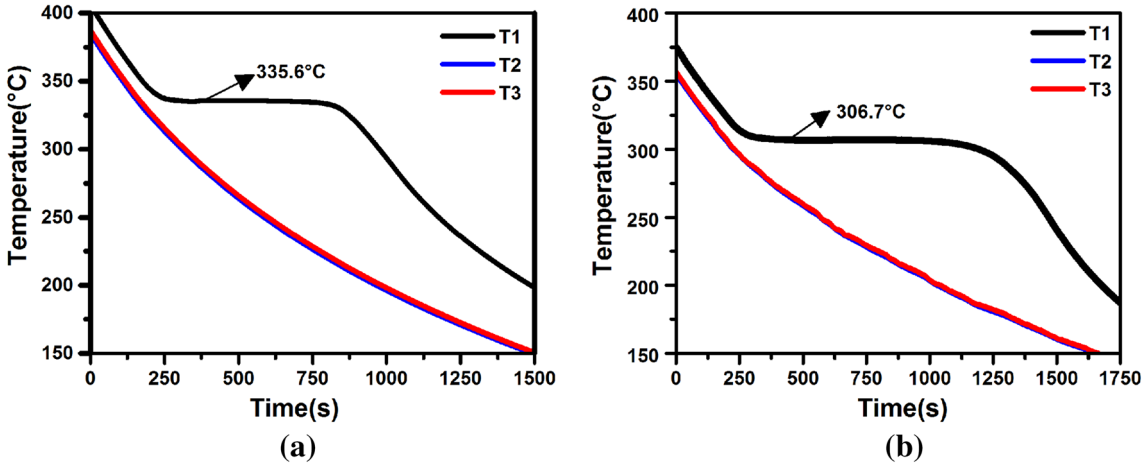

Fig. 2 **a** Thermal history during cooling of the salt KNO_3 and the mold. **b** Thermal history during cooling of the salt $NaNO_3$ and the mold

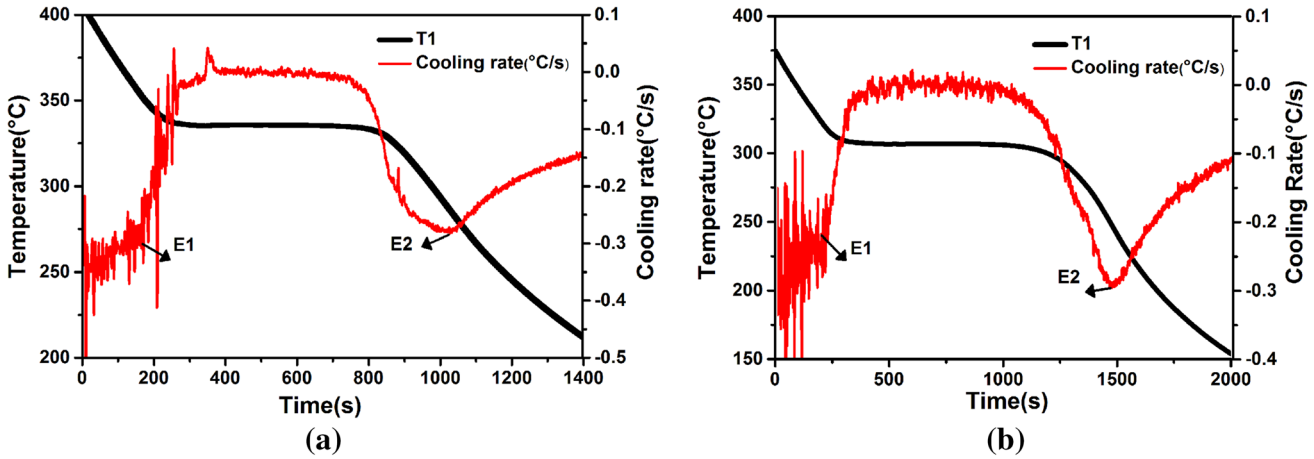

Fig. 3 **a** The cooling curve and the cooling rate curves for KNO_3. **b** The cooling curve and the cooling rate curves for $NaNO_3$

In the inverse analysis, the input given was the data file of the measured temperature data of T3 thermocouple. The heat flux at the inner surface was estimated for both the KNO_3 and $NaNO_3$ as shown in Fig. 4.

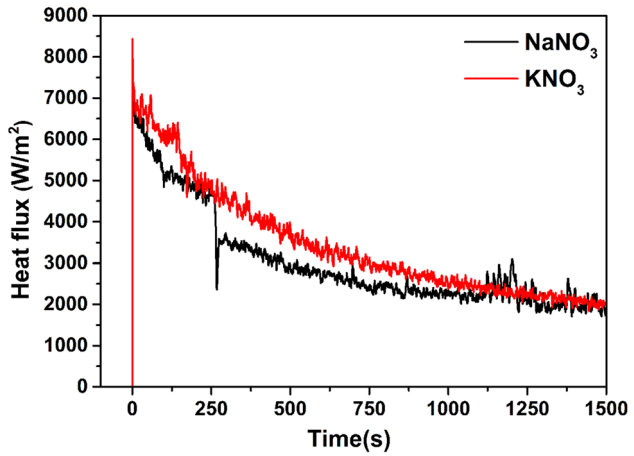

Fig. 4 Estimated heat flux transients for the salts

4 Discussion

In the case of KNO_3, as per the solidification path shown in Fig. 3(a), the solidification of the salt starts at around 173.5 s at a temperature of 350.57 °C. This point is referred to as liquidus point. The solidification ends at 1030.5 s at a temperature of 285.1 °C. This point reflects the complete solidification of the salt and is called solidus point. This salt melts at a nearly constant temperature of 335.6 °C reflecting the phase change temperature of the salt. KNO_3 takes around 857 s for complete solidification. The liquidus and solidus points are selected from the cooling rate curves rather than from the cooling curves. Similarly, in case of $NaNO_3$ as shown in Fig. 3b, the melting temperature from the cooling curve is measured to be 306.7 °C. The solidification of this salt starts at 211 s and at a temperature of 321.1 °C. This liquidus point is represented by a change in slope on the cooling rate curve. The solidus point of the salt is at 1468 s and at a

temperature of 250.2 °C.$NaNO_3$ has taken around 1257 s for complete solidification.

The average specific heat value used for calculations is 1.2 kJ/kg K for KNO_3 [5] and 1.11 kJ/kg K for $NaNO_3$ [6].The latent heat values for KNO_3 using this method during cooling have been calculated to be 112 kJ/kg which is comparable to the reported values of 91 and 110 kJ/kg [5].The phase change enthalpy for $NaNO_3$ has been calculated to be 161 kJ/kg and the reported values in the literature are 172 and 177 kJ/kg [6, 7]. It can be seen that the values of the enthalpy of solidification obtained using this method are in good agreement with the reported values in the literature. The error percentage values calculated using the measured and estimated temperature data for the T2 thermocouple near to the outer surface of the mold are $\pm$ 0.88% for KNO_3 and $\pm$ 0.42% for $NaNO_3$. These error values show the reliability of this method and the selection of parameters in the inverse analysis and the energy balance calculations.

5 Conclusion

The IHCP-Energy Balance technique offers a simple and inexpensive tool to study and assess the solidification parameters of thermal energy storage materials. The proposed technique does not suffer from the limitations due to fitting errors involved in baseline calculations. The applicability of the method can be further investigated with metals and other salts. Highly sensitive and well-calibrated thermocouples along with accurate data on the thermo physical properties of salts and mold will enhance the accuracy of the results obtained.

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