



## D-mannitol for medium temperature thermal energy storage

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### ABSTRACT

D-mannitol is a sugar alcohol with a melting temperature of approximately 167 °C. It has been identified as a phase change material for storing heat at a temperature range of about 150–180 °C. The outcome of the published research on its applicability for this purpose is inconsistent and sometimes contradictory. We identified that there is a need for further study on the material to overcome such uncertainty. Thus we conducted a series of thermal measurements to quantify its properties and suitability for a phase change thermal storage. Our differential scanning calorimetric measurements showed that the material has a melting temperature of 167 °C and fusion heat of 297 kJ/kg. However, our accelerated thermal cycling test revealed that this value decreases logarithmically to 249 and 240 kJ/kg after 100 and 200 cycles respectively. The material shows a significant level of sub-cooling in DSC measurement with a solidification temperature of approximately 110–120 °C which can render the material unsuitable for thermal storage purposes. However, our secondary measurement with a large quantity of D-mannitol shows that such level of sub-cooling is unlikely to occur in a macro-scale sample. We built and tested a thermal storage system containing 3.8 kg of D-mannitol to investigate its performance in larger quantities and evaluate the heat transfer properties of the heat exchanger mechanism in the storage system. We show that the material releases significant heat when it is cooled down to 150 °C which means implies the occurrence of solidification at a temperature above 150 °C.

### 1. Introduction

Global industrial sector consumes about 80 EJ of heat for thermal processes per annum. This is mainly used at temperatures below 400 °C [1]. Recovering waste heat or implementing solar thermal energy can partially offset this, and thus reduce the carbon footprint of the industrial sector. Taibi et al. [2] suggest that solar thermal systems can supply 5.6 EJ per annum to the industrial process heat demand by 2050. By providing a buffer for smoothing the renewable heat supply and addressing the associated asynchronous production and consumption of heat, a thermal energy storage (TES) system can play a key role to successfully integrate solar thermal energy for industrial process heat applications capable of operating at higher temperatures [3–5].

A TES system for process heat should be compact (high energy per unit volume), low cost, thermally stable, and capable of providing high enough charging/discharging rates to meet the thermal process requirements. Sensible heat [6,7], phase change latent heat [8], and thermo-chemical [9,10] TES systems have been investigated by researchers for years. Among these, the latter two are capable of providing dense thermal storage within a narrow temperature bracket.

A phase change TES system uses the heat required for physical

phase transformation of a material, normally from solid to liquid, to store and vice versa to deliver heat. Due to the availability of a range of different materials with diverse melting temperatures, this approach has shown significant potential [11].

Sugar alcohols have been identified as potential phase change materials (PCMs) for a temperature range of 50–220 °C [12]. Many process heat applications fit in this temperature range and consequently can be fed by sugar alcohol TES systems. As another advantage, sugar alcohols are not corrosive. This is very important because PCM based TES systems generally need a sophisticated heat exchanger to extract heat from the PCM. Corrosive PCMs can significantly reduce the life of the heat exchanger.

Coupling solar thermal collectors capable of delivering heat at temperatures of 150–200 °C with a thermal storage capable of storing heat at this range can be very beneficial. D-Mannitol (DM) is one of the sugar alcohols with a melting temperature of 167 °C. Although it is an artificial sweetener mainly used for pharmaceutical applications, researchers have considered its applicability for thermal storage applications because of its high heat of fusion (328.8 kJ/kg) [13,12], high density (1520 kg/m<sup>3</sup>) [13], safety due to it being non-toxic [12], and its relatively low cost (2.40 USD/kg). This has major advantages over

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pressurized water for that would require expensive and possibly dangerous pressure vessels to remain liquid at around 180 °C and mineral oils which is expensive with a relatively low heat capacity. DM can potentially store heat densely at low cost [14], however, there is a lack of consistency among the data reported for its thermal properties in the literature.

Gil et al. [15,16] conducted a series of experiments with varying initial and final temperatures and cooling rates of DM which resulted in a lower melting enthalpy, 263 kJ/kg rather than 328.8 kJ/kg. They concluded that this could be due to the inaccuracy of the equipment. They also emphasized the importance of accurate temperature and heat transfer rate control because of the polymorphic characteristics of DM. They tested the performance of a TES system with 160 kg of DM which underperformed in terms of thermal capacity storing/releasing about 200 kJ/kg and 100 kJ/kg of heat.

Barreneche et al. [17] studied the effect of DM polymorphism and sub-cooling on its thermal storage capacity. They observed varying DSC peaks due to the polymorphism of DM. Initially without any cycling, only 1 peak with 247 kJ/kg fusion heat was present at 167 °C which corresponds to  $\beta$  phase, however after the second cycle, an additional peak corresponding to the metastable  $\gamma$  phase with an enthalpy of lower than 238 kJ/kg emerged. The authors concluded that the polymorphism would not affect the performance of DM if the working temperatures are within 135–175 °C. But we suggest that the significant sub-cooling reported in their paper can be a major issue.

In simple terms, in the presence of sub-cooling, the material solidifies at a temperature lower than its melting point. A TES system made of such a PCM will downgrade the thermal energy produced by e.g. solar collectors. Such TES system needs to be charged at high temperature and releases heat at a lower temperature. However, the presence of sub-cooling for DM is worth to investigate further. This will be discussed in this paper.

The other concern is the stability of DM under thermal cycling and long term exposure to heat. Solé et al. [12] studied this and concluded that when the sample is exposed to air, the phase change enthalpy reduces from around 240 kJ/kg to 150 kJ/kg after 20 cycles and reduces to 100 kJ/kg after 50 cycles which represents a reduction of 30% and 50% in enthalpy, respectively. A large drop in enthalpy is detrimental to the performance of the storage unit. They repeated the experiment in the absence of oxygen and reported no changes in the samples phase change enthalpy. They declared that the degradation was due to oxidation.

Rodríguez-García et al. [18] recently reported that DM degrades even in the absence of oxygen due to non-oxidation browning. They conducted thermal cycling and steady heat exposure tests using a custom-made device and observed mass loss and colour change. They concluded that removing oxygen from DM TES system will not prevent degradation and hence this material is not suitable for storing heat efficiently.

Such level of discrepancy implies that there is a need for further examination of DM stability under thermal cycling and heat exposure. We aim to do a series of accurate experiments with DM to help draw more accurate conclusions regarding its applicability to thermal storage. In this paper, we investigate its thermal stability under thermal cycling using DSC measurement of micro-scale samples up to 200 cycles, and try to quantify its degradation. We also show that the significant sub-cooling occurs in micro-scale measurements but there are indications showing that it doesn't happen in macro-scale quantities of DM. Then we examine the performance of a macro-scale TES system made of DM and a heat exchanger to examine the thermal behaviour of such storage cell in real world conditions.

## 2. Method

We started examining DM with micro scale samples using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC).

In TGA approach, we measure the mass loss of DM as a function of its temperature. We used a 2-mg sample at a heating rate of 10 °C/min with a temperature range of 25–850 °C in a PerkinElmer's Pyris 1 TGA. The sample weight measurement accuracy of the device was 2  $\mu$ g. We carried out this measurement under nitrogen flow to avoid any unknown effects of oxygen.

We performed the DSC analysis using a Perkin-Elmer differential scanning calorimeter (Pyris 1) exposing a DM sample and a reference (air) to a designated heating rate for a temperature range from 25 to 180 °C. The upper bound of the temperature bracket was limited to 180 °C based on the TGA results to ensure that the sample doesn't degrade in DSC tests. We used 25-mg aluminium sample pans and 22-mg aluminium lids to house DM powder.

The sample went through thermal cycling at accelerated rate of 50 °C/min. This high rate of heating was used to save time over more than 100 cycles and we didn't rely on the specific heat readings at this rate of heating. To measure the specific heat of the sample, we changed the heating rate to 10 °C/min every 20th cycle and scanned its specific heat. The time taken for each cycle at 10 °C/min and 50 °C/min was 37 and 8 min respectively. In order to make sure that the samples reach the extreme temperature of 180 °C at each cycle, we held the sample at that temperature for 2 min before cooling it down.

We analysed five 2-mg samples of DM in DSC thermal cycling. We used a weight balance accurate to 1  $\mu$ g to weigh the five samples. A total of 100 cycles were completed on all samples, with the exception of sample 4 which went through an additional 100 total cycles.

We altered the method to observe the effect, if any, the heating rate could have on the sample specific heat readings. We conducted a heating/cooling cycle at a rate of 2 °C/min on sample 5 and compared its result with the results obtained at 10 °C/min heating rate. We weighed the samples again after thermal cycling to see if there was any mass loss of the sample due to any possible leakage from the sealed containers.

DSC samples were micro scale test specimens in the range of a few mg. Following the completion of the DSC, we investigated the sub-cooling effect in macro-scale sample of DM using an industrial oven, a beaker and a 20-g sample of DM. This experiment was undertaken by heating up the sample to 175 °C as this would ensure that the entire sample was melted. Then we placed the molten DM outside the oven to cool down. We used a thermocouple to measure the temperature and visually inspected the solidification process.

To measure the performance of a DM TES system, we designed and built a full scale TES cell. Fig. 1 shows a sketch of the thermal storage cell. It is made of an aluminium finned stainless steel tube encased within a mild steel pipe. The finned length of the tube is 3000 mm which is entirely encased by the pipe. The tube OD is 1 in. with 16 mm high aluminium fins attached to it. The fin pitch is 2 mm. The OD of the pipe is 72 mm. The diameter of the fins is slightly smaller than the internal diameter of the pipe. Hence there is a gap between the rim of the aluminium fins and the steel pipe. DM fills this gap and the space between the fins. Two steel caps seal the cell and support the finned tube at the top and bottom allowing the ends of the finned tube to protrude through a hole for manifolding purposes.

The fin structure creates a large contact area between DM and the tube that contains the heat exchanging fluid, therminol 66. This storage cell can accommodate up to 12 kg of DM which in theory is capable of storing about 1 kWh of heat at 150–180 °C. However, in this experiment, we filled it with 3.8 kg of DM. Molten DM can travel along the fins adjacent to the central hot tube to accommodate for thermal expansion and prevent the system from excessive pressurisation.

The whole storage cell is thermally insulated with 25-mm glass wool to reduce its heat loss. Two RTD sensors measure the inlet and outlet temperatures of therminol 66 into and out of the TES cell. The thermal properties of therminol 66 were extracted from the manufacturer's data sheet.

We connected the TES cell to the high temperature thermal testing

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