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Thermal Conductivity Measurement of Erythritol, Xylitol and Their Blends for Phase Change Materials Design- a Methodological Study

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Abstract

This work presents and discusses a detailed thermal conductivity assessment of erythritol, xylitol and their blends: 25 mol% erythritol and 80 mol% erythritol using the Transient Plane Source (TPS) method with a Hot Disk Thermal Constants Analyzer TPS-2500S. Thereby the thermal conductivities of xylitol, 25 mol% erythritol, 80 mol% erythritol and erythritol were here found for respectively in the solid-state to be 0.373; 0.394; 0.535; and 0.589 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ and in the liquid-state to be 0.433; 0.402; 0.363; and 0.321 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$. These obtained results are comprehensively and critically analyzed as compared to available literature data on the same materials, in the phase change materials (PCMs) design context. This study clearly indicates that these thermal conductivity data in literature have considerable discrepancies between the literature sources and as compared to the data obtained in the present investigation. Primary reasons for these disparities are identified here as: the lack of sufficiently transparent and repeatable data and procedure reporting, and relevant standards in this context. To exemplify the significance of such transparent and repeatable data reporting in thermal conductivity evaluations in the PCM design context, here focused on the TPS method, a comprehensive measurement validation is discussed along various residual plots obtained for varying input parameters (i.e., the heating power and time). Clearly, the variations in the input parameters give rise to various thermal conductivity results, where choosing the most coherent result requires a sequence of efforts per material, because there are no universally valid conditions. Transparent and repeatable data and procedure reporting is the key to achieve comparable thermal conductivity results, which are essential for the correct design of thermal energy storage systems using PCMs.

Keywords: phase change materials, thermal energy storage, thermal conductivity, transient plane source method, erythritol, xylitol

Abbreviations

BE	Boundary Exceeded
dT	Temperature variations (in K)
Er	Erythritol
FC	Free convection

L	Liquid
LFA	Laser Flash Apparatus
LS	Laser Step
na	not available
NIST	National Institute of Standards and Technology
PCM	Phase change material
S	Solid
SS	Stainless steel
TCR	Temperature Coefficient of Resistivity
TES	Thermal Energy Storage
THS	Transient Hot Strip
THW	Transient Hot Wire
TPS	Transient Plane Source
USP	Uneven Signal Penetration
Xy	Xylitol

1 Introduction

Thermal Energy Storage (TES) can provide effective solutions for better energy management, improved energy efficiencies, climate change mitigation, renewables integration, and flexible operation. Surplus thermal energy, from e.g. power plants and industries,^{[1], [2], [3]} can be stored and used at different locations and time^{[2], [4], [5]} thus realizing better management. Improved efficiencies and CO₂ mitigation are achieved by storing surplus thermal energy to fulfill existing heating/cooling demands which otherwise are met by fossil-based means^{[1], [6], [7]}. Renewable energy sources such as solar can be effectively integrated to a TES system (e.g. in solar thermal systems^{[8], [9]}) to resolve the challenges due to their intermittent nature. TES also incorporates flexibility into an energy system by permitting peak shaving and load shifting^{[10], [11], [12]}, also with potential cost benefits.

Phase Change Materials (PCMs) are one effective TES choice, allowing compact storage with larger storage densities e.g. as compared to sensible TES. Numerous PCMs-based TES applications have been investigated over the years^(e.g. [13], [14], [15], [16], [17], [18]). Among these, many PCMs were analyzed to be incorporated in buildings for improving the indoor thermal comfort^[13]. An example is where PCMs suitable for building indoor thermal comfort applications were obtained via the fractionation of natural-gas based products to obtain alkenes-based PCMs^[14]. The intermittency in renewable energy sources like solar thermal and concentrated solar power production can also be effectively mitigated by employing TES, among others, employing PCMs^[15]. Among such investigations, e.g. dodecanoic acid as a PCM has exhibited 20 % more TES capacity than a water storage system in a solar thermal system^[16]. The overall energy efficiencies of today's industrial processes can also be achieved by means of integrating TES with PCMs, by means of waste heat recovery^[18]. Thermal management in batteries with PCMs^[17] is another emerging technology of

TES. To accomplish truly sustainable TES solutions from PCMs, besides their energy-related advantages, materials of renewable origin are of great interest as PCMs. Polyols are one such renewable material category emerging as candidate PCMs^{[19], [20], [21], [22], [23], [24]} with attractive thermal properties suitable for cooling as well as heating applications.

The polyols erythritol, xylitol, and a eutectic among their blends^{[23], [24], [25]} are found as attractive PCM candidates, with melting temperatures (77-120 °C) and enthalpies (~200-370 kJ·kg⁻¹) suitable for low-temperature heating. These materials should also have satisfactory thermal conductivities for fast charging and discharging of heat/cold in the TES system. The thermal conductivity of erythritol and xylitol are available in literature, however, with considerable disparities and often lacking the details on the specific measurement conditions essential for repeatability^(e.g. [19], [24], [26], [27], [28], [29], [30]).

Numerous methods are being used today to measure thermal conductivity of solids and liquids. From these, some only measure thermal conductivity at steady-state while some others measure at unsteady-state (i.e., transient conditions)^[31]. The steady-state methods measure thermal conductivity employing heat flows that are one dimensional (using e.g. a guarded hot plate, an unguarded hot plate, or various other configurations for linear heat flow) or radial (e.g. cylindrical, spherical or ellipsoidal)^[31]. From the transient methods, some only measure thermal diffusivity, using which thermal conductivity is indirectly calculated (e.g. Ångström, Laser Flash Apparatus (LFA), and Laser Step (LS) methods). Whereas, some other transient methods measure thermal conductivity (e.g. Hot Wire Method (HWM)), or both thermal conductivity and thermal diffusivity (e.g. transient hot strip (THS) and transient plane source (TPS) method)^[31].

Among these transient methods, those which can only measure thermal diffusivity are limited so because the applied power to the sample cannot be determined (e.g. LFA, Ångström and LS methods). The other transient methods do not have this limitation, as in those methods the applied power can be precisely determined to measure the thermal conductivity and sometimes even the thermal diffusivity (e.g. THS and TPS methods)^[31]. Therein, such transient methods clearly come with advantages. Considering these transient methods, longer sample sizes are necessary for the hot wire and hot strip methods, whereas in the TPS method the sample size requirement is minimized by using a sensor made as a double spiral^[31]. Another practical advantage of the TPS method is that both low and highly conductive powders, liquids, solids and metals can be tested. In-contrast, e.g. in the THW method, the range of samples is narrowed to especially low conductive powders, liquids and solids that can be easily injected to the sample holder, unlike in the TPS method. Therefore, the TPS method appears to have clear advantages over many other thermal conductivity measurement methods today.

The thermal conductivity properties of erythritol, xylitol and their blends of the compositions 25 mol% and 80 mol% erythritol were presented very concisely in a recent publication by the present authors^[25]. However, in that study, focused on the determination of the erythritol-xylitol phase diagram, comprehensive details of the thermal conductivity assessment of the system were excluded. Driven by the disparities observed in various literature, herein, the thermal conductivity

investigation of erythritol, xylitol, 25 mol% erythritol (a blend close to the eutectic in the system) and 80 mol% erythritol using the TPS method is presented in explicit detail. This procedure and the obtained results are then critically compared with the available thermal conductivity data in literature. Thereby, the aim of this work is to exemplify the dependence of the final results on the employed testing conditions and the underlying data evaluation procedure, and the key information crucial for transparency and repeatability, all vital in PCMs characterization and design. Although the present study is limited to the TPS method, it is expected here to reach certain common conclusions in this respect for unified thermal conductivity assessments and thus results in the PCM design context.

2 Materials and Methods

Meso-erythritol ($C_4H_{10}O_4$, CAS number 149-32-6) and xylitol ($C_5H_{12}O_5$, CAS number 87-99-0) of 99% purity each^[32], and their blends in the compositions 25 mol% erythritol (i.e., a close-to-eutectic blend) and 80 mol% erythritol were investigated (hereon, mol% erythritol will be referred to as mol% Er). The molar compositions of these blends were within the expanded uncertainty of 1.3 mol% at a 0.95 confidence level^[25].

The thermal conductivity of these pure polyols as well as their blends was analyzed experimentally using the TPS method using a Hot Disk Thermal Constants Analyzer TPS-2500S. This employed set-up is shown in **Figure 1 (a)**. In this case, the Hot Disk sensor 7577 (2 mm radius) which is insulated with polytetrafluoroethylene (PTFE) was used. A specially in-house built sample holder made of aluminum (volume: 7 ml, diameter: 24 mm, height: 15 mm) was used, which was mounted on a stainless steel (SS) base^[33], as shown in **Figure 1 (b)**. This sample holder was built, according to the TPS-2500S manufacturer guidelines, to test PCMs at various temperatures besides room temperature (because the manufacturer's PCM testing holder is limited to room temperature measurements).

Figure 1. The employed TPS (a) experimental set-up and (b) sample containment.

The Hot Disk Thermal Constants Analyzer TPS-2500S instrument and TPS sensor was verified using benchmark tests conducted on distilled water. In this case another Hot Disk sample holder for only liquid samples had to be used. In these thermal conductivity verification tests using the TPS method, each sample test was repeated up to three times at each chosen power and time condition set. The obtained measured values, reference values (from the NIST^[35]) and the standard deviations are summarized along with the measurement power, time in **Table 1**.

Table 1. Standard deviations of the measured thermal conductivities of distilled water the TPS method^a

Temperature (°C)	Power (mW)	Time (s)	Averaged Thermal Conductivity (W·m ⁻¹ ·K ⁻¹) for 9 repetitions	Reference Thermal Conductivity ^[35] (W·m ⁻¹ ·K ⁻¹)	Standard deviation (W·m ⁻¹ ·K ⁻¹)
5	25, 28, 32	2, 3	0.571	0.570	0.005
10	25, 28, 32	2, 3	0.579	0.580	0.003
15	25, 28, 32	2, 3	0.588	0.589	0.002
20	25, 27, 30	2, 3	0.593	0.598	0.004
25	25, 27, 30	2, 3	0.598	0.607	0.002
30	22, 25, 28	2, 3	0.613	0.615	0.003
35	18, 20, 22	2, 3	0.623	0.623	0.004
40	18, 20, 22	2, 3	0.628	0.631	0.002

^ain this study the experimental pressure was not controlled beyond atmospheric pressure (101±2 kPa)

As can be seen in **Table 1**, the difference between the obtained experimental results therein and data from the National Institute of Standards and Technology (NIST)^[35] for distilled water was less than 0.7 % which is significantly below the measurement error of instrument set to be ± 5 % (~ 0.05 W·K⁻¹·m⁻¹). In these benchmark tests, density and specific heat capacity were used as input values for the post processing of thermal conductivity results. The use of this knowledge of the volumetric heat capacity (i.e., the product of density and specific heat) in the data post processing decreases the thermal conductivity measurement error below 2 %. It is important to underline that this post processing method was used to both verify the validity of the Hot Disk TPS-2500S instrument measurements, as well as to prove that this TPS testing method can be applied for liquid samples in a wider temperature range. These benchmark tests are concentrated on the liquid-state measurements, because, liquid-state thermal conductivity measurements are more complex to handle generally in all the thermal conductivity measurement methods.

In this investigation, erythritol and xylitol were considered as the reference cases for 80 and 25 mol% Er blends, respectively. However, when using these reference measurement parameters for testing the blends, some indicated pronounced convection or insufficient signal penetration into the sample. Thus, other specific testing conditions were used in those cases as detailed in **Table 2**. The

uncertainty of these thermal conductivity measurements is 5% as specified by the Hot Disk manufacturer^[34].

The material samples tested were prepared in this work as following. For the liquid-state measurements, the powdered solid samples were filled into the TPS container, while keeping the sensor vertical, and sandwiched between the powders. Then the open areas of the container (the areas where white powder can be observed as in **Figure 1 (b)**) were then covered with aluminum foil. Each respective sample then inside the TPS container was then inserted into the thermal bath with a silicon oil connected to the TPS apparatus, maintained at the respective measurement temperature as specified in **Table 2**. These were then maintained in the thermal bath at this set temperature, for several hours, to ensure complete melting, before the measurements were conducted.

Table 2. The employed TPS measurement conditions^a on pure erythritol, pure xylitol and their blend compositions: 25 mol% erythritol and 80 mol% erythritol

<i>Sample</i>	Liquid/ Solid	Temperature (°C)	Power (mW)	Time (s)
<i>Erythritol</i>	Liquid	125 ^b	17, 19, 20, 22, 25	2, 3, 4
	Solid	20 ^c	18, 20, 21, 22, 25, 30	2, 3, 4
<i>80 mol% Er^d</i>	Liquid	125 ^b	18, 20, 22, 25	2, 3, 4
	Solid	20 ^c	18, 20, 22, 25	2, 3, 4
<i>Xylitol</i>	Liquid	110 ^b	18, 20, 21, 22, 25, 30	2, 3, 4
	Solid	20 ^c	18, 20, 21, 22, 25, 30	3, 4
<i>25 mol% Er^d</i>	Liquid	110 ^b	18, 20, 25	2, 3, 4
	Solid	20 ^c	18, 20, 21, 22, 25	2, 3, 4

^ain this study the experimental pressure was not controlled beyond atmospheric pressure (101±2 kPa)

^{b,c,d}with the respective expanded uncertainties with 0.95 confidence level: ^b5 °C; ^c0.1 °C; and ^d1.3 mol%

For the solid-state measurements, the samples were prepared using some additional steps. The respective solid samples were first melted by heating in an oven respectively at 145 °C- for erythritol and 80 mol% Er, and at 120 °C -for xylitol and 25 mol% Er¹, until each became a single liquid². Simultaneously, the TPS sample container attached with the sensor, was also subjected to the same respective heat-treatment. Once molten, each of these respective samples were carefully poured into the TPS container cavity (until reaching a few mm below its maximum height), while keeping the sensor vertical and trying to avoid air-bubbles formation (especially around the sensor). Afterwards, the heating of erythritol and 80 mol% erythritol was gradually changed into cooling, using 10-15 minutes time steps into 120, 110, 95, 80 and 65 °C. The aim of this gradual cooling process was to avoid metastable phases that could be triggered in erythritol and 80 mol% erythritol samples if they were directly cooled to ambient temperature from 145 °C. As xylitol and 25 mol% Er samples have a much slower solidification rate compared to erythritol-rich samples, these were

¹ As these have a lower melting temperature, and to minimize thermally-activated change in these, a temperature lower than 145 °C was used here.

² This melting was a must to ensure that the blends are in-fact blends rather than a physical mixture of the two pure components, whereas, the same was performed for the pure components, to maintain a standard procedure.

given only one intermediate cooling step at 70 °C for 10 minutes. At the end of such cooling steps, a very small amount of the starting powder material was seeded to each melt, to further encourage the stable solid-state formation. Erythritol and 80 mol% erythritol completed solidification fast (within less than an hour), whereas xylitol and 25 mol% Er samples required several hours in room temperature till complete solidification. Then these samples were also covered with aluminum foils at the top of the TPS container.

With such a careful solidification process, the samples were sandwiching the Hot Disk sensor well, ensuring a satisfactory contact with it. This solidification process however did not pressurize the sensor too much, as it mostly started at the sensor and propagated outwards into the TPS cavity which had sufficient volume for the crystals to grow. The prepared solid samples as such were then transferred to the TPS thermal bath at ambient temperature (~24 °C), and then were maintained in the bath at 20 °C for one hour. Afterwards, the TPS measurements were performed on their solid state, also using the conditions as specified in **Table 2**.

3 Results and Discussion

The thermal conductivities measured in this work using the TPS method are compared with literature data, in **Table 3**, beside available measurement conditions. As **Table 3** indicates, in the PCM literature as a whole, the thermal conductivities of erythritol and xylitol, in solid or liquid state, were presented with considerable discrepancies consequent in a large range of values. The thermal conductivity of solid erythritol has major discrepancies between various literature, found within the range 0.31-0.89 W·m⁻¹·K⁻¹. The solid state of xylitol has also been proposed with numerous thermal conductivity values, consequent in the largest range of discrepancies found, within 0.44-1.31 W·m⁻¹·K⁻¹.

In this work, the thermal conductivities of solid erythritol and xylitol respectively were found to be 0.59 W·m⁻¹·K⁻¹ and 0.37 W·m⁻¹·K⁻¹, however, with not many comparable values available in literature (c.f. **Table 3**). The 25 mol% Er was examined using the TPS method by just one study: del Barrio et al., 2017^[24], presenting the liquid and solid thermal conductivities to be 0.42 and 0.38 W·m⁻¹·K⁻¹. The thermal conductivities of the 25 mol% Er for both liquid and solid states obtained in the present work are comparable with del Barrio et al., 2017^[24]. However, the pure component results are considerably different in the case of erythritol for both solid and liquid states, and of solid xylitol. The 80 mol% Er was not studied by any other authors thus far.

The thermal conductivity measurements found in literature were performed using many different methods such as: TPS, C-Therm Thermal Conductivity Analyzer, LFA, Thermo Con Tester M100, and Thermal Conductivitymeter EP2C (c.f. **Table 3**). With LFA, as was mentioned in section 1, only thermal diffusivity can be measured directly. Thus, thermal conductivity is an indirect result in LFA obtained as a function of thermal diffusivity, combined with specific heat (c_p) and density^[26] (which are also experimentally measured using various techniques).

Table 3. The final thermal conductivities of erythritol, xylitol, 25 mol% Er and 80 mol% Er measured in this work compared with literature (L: Liquid, S: Solid, Er: erythritol, na: not available, RT: room temperature)

Material	L/ S	Measurement conditions and input parameters				Thermal conductivity (W·m ⁻¹ ·K ⁻¹)	Sources
		Temperature (°C)	Power (mW)/ other conditions	Time (s)/other conditions	Volume (ml)		
Erythritol	L	125 ^a	20	3	~6.7	0.321	This work ^{f, c}
		120-160, 140, 110, ~120	na, na, na, na	na, na, na, na	na, ~0.23, na, ~30	0.327-0.339, 0.330, 0.350, ~0.370	[27] ^f , [26] ^{j,p} , [36] ^{h,q} , [24] ^{s,r}
	S	20 ^b	20, 18-25	3, 2-4	~6.7	0.586, 0.589 ^e	This work ^{f, c}
		na ^g , na ^g , na ^g , 30, 25, na ^g , RT ^g , 25, 30 (15-60), RT, na ^g , 27, 25, 29, na, 20	na, na, na, na, na, na, ASTM D5470, 1000 ^m (na ⁱ), na, na, ASTM D5470, 25, na, na, na, na	na, na, na, na, na, na, na, 60 ^m (na ⁱ), na, na, na, 4, na, na, na, na	na, na, na, na, na, ~0.16, na, na, na, na, na, na, ~30, na, na, na, ~0.23	0.310, 0.326, ~0.369, 0.390, 0.650, 0.703, 0.710, 0.720 ^{m,j} , 0.722 (0.756-0.688), 0.733, 0.730, 0.761, 0.770, 0.800, 0.810, 0.890	[28] ^l , [37] ^f , [38] ^p , [36] ^{h,q} , [39] ^j , [40] ^f , [41] ^l , [42] ^{m,j} , [27] ^f , [19] ^j , [43] ⁿ , [24] ^f , [44] ^j , [45] ^j , [29] ^{i,k} , [26] ^{i,p}
80 Er	L	125 ^{a,d}	20	2, 2-3	~6.7	0.363	This work ^{f, c}
	S	20 ^{b,d}	20, 18-25	2, 2-3	~6.7	0.553, 0.535 ^e	
Xylitol	L	110 ^a	20, 20-22	3, 2-3	~6.7	0.412, 0.433 ^e	This work ^{f, c}
		110, 140, ~90-110, 100 (30-100) ^r	na, na, na, na	na, na, na, na	na, 0.23, ~30, ~30	0.350, 0.360, ~0.395, ~0.400 (0.390-0.450) ^r	[36] ^{h,q} , [26] ^{j,p} , [24] ^{s,r} , [30] ^{s,r}
	S	20 ^b	20	3	~6.7	0.373	This work ^{f, c}
		30, 20, 27, 25	na, na, 25, 25	na, na, 4, na	na, ~0.23, ~30, ~30	0.440, 0.520, 1.314, 1.300	[36] ^{h,q} , [26] ^{j,p} , [24] ^f , [30] ^f
25 Er	L	110 ^{a,d}	20	2	~6.7	0.402	This work ^{f, c}
		~80	na	na	~30	~0.420	[24] ^{s,r}
	S	20 ^{b,d}	20, 20-25	3, 3-4	~6.7	0.391, 0.394 ^e	This work ^{f, c}
		27	25	4	~30	0.380	[24] ^f

^{a,b,d} expanded uncertainty with 0.95 confidence: ^a5 °C; ^b0.1 °C; and ^d1.3 mol%, ^ean uncertainty of 5%^[31], ^caverage over the range

^{f,h,j,l,m,n,o,s} used method: ^fTPS (Transient Plane Source) method using TPS 2500S, ^hC-Therm Thermal Conductivity Analyzer using modified TPS method, ^jLaser Flash Apparatus, ^lThermo Con Tester M100, ^mThermal Conductivitymeter EP2C (Neo Tim), ⁿOne-dimensional Steady-state method, using own apparatus, ^oTransient Hot Wire (THW) method, ^sHot-Disk based method as proposed in literature^{[46], [47]}

^gthe state (whether solid or liquid) was not specified, however, given details imply that the measurements were done of the solid at around room temperature

^kfood-grade material, ^ptechnical-grade material, ^qbulk-grade materials, ^rincluding supercooled liquid

Among these numerous evaluations in literature, except for some, a majority do not detail the corresponding measurement conditions (as identified with na-not available), as **Table 3** indicates. Such a lack of procedural reporting has been observed also beyond the context of polyols, by other authors (e.g. Warzoha and Fleischer, 2014a^[46]). Possible main reasons for the results discrepancies in literature are thus here considered as related to the different measurement conditions used, and the unavailability of these details alongside the results for later evaluations to produce repeatable results.

Furthermore, it appears that a major lack exists in relevant standards to abide to, for comparability. Thermal conductivity directly influences the heat exchanger design and thus the TES system functionality. Hence, incomparable thermal conductivities could negatively affect the expected TES system performance.

3.1 Validation of Erythritol-Xylitol Thermal Conductivity Measurements using Hot Disk Thermal Constants Analyzer TPS-2500S

For comparability of experimental data, transparent and repeatable data and procedure reporting is imperative. This is equally true for the experimental investigations of thermal conductivity of PCMs. To exemplify the significance of such transparent and repeatable data reporting, the detailed thermal conductivity assessment in this work using the TPS method with the Hot Disk Thermal Constants Analyzer TPS-2500S, on erythritol, xylitol and the 25 mol% Er composition is discussed here. The detailed assessment on the 80 mol% Er is however excluded due to the lack of reference literature data.

3.1.1 Accurate Data Analysis Approach in Using the TPS Method

In the Hot Disk Thermal Constants Analyzer, the Hot Disk sensor has two main functions. It acts as the heat source for increasing the temperature of the sample, and as a resistance thermometer that records the time-dependent transient temperature increase in the sample. For a certain applied power within a given time duration, the sensor sends in 200 pulses and measures the thermal response of the sample for each pulse. It is important to choose the radius of the Hot Disk sensor according to respective experiment and sample type. First of all, the sensor radius must be considerably larger than the porosity or the void structure of the sample, if the material in question is not dense or homogenous. For homogenous materials where the structure variations are at atomic or molecular levels (such as metals), Hot Disk sensors with radii between 3.2 and 15 mm should be used. Whereas, liquids should be tested using smaller radii from approximately 0.5 to 3.2 mm^[31].

By accounting the applied power, time, temperature change in the sample, the sensor radius and the probing depth, the Hot Disk Thermal Constants Analyzer software iteratively calculates the thermal conductivity, thermal diffusivity and also the specific heat capacity of the sample. The detailed thermal conductivity calculation procedure in the TPS method is found in the Hot Disk Thermal Analyzer TPS-2500S manual^[31]. This calculation is performed for a selected amount out of 200 data points representing the temperature increase during the transient test. These calculated values are compared with the measured values corresponding to the selected data points and are

represented by means of a ‘residual plot’ by the software. In the residual plot, the temperature variations in the sample (in K) is plotted versus the square root of time (in $\sqrt{s}$). **Figure 2** is an example of a typical residual plot, which has a perfect random scatter of the data points around the 0 K temperature variation. Obtaining this type of a random scatter in the residual plot is essential for a correct thermal conductivity evaluation with the TPS method. In contrast, the residual plot data points shifting towards positive or negative temperature variations is characteristic of one or more undesirable conditions such as: too little or too high power; too short or too long time; effects of free (i.e., natural) convection; or signal reaching the sample boundary^[31]. The way these undesirable conditions relate to the applied power and time are summarized in **Figure 3**.

Figure 2. A typical residual plot produced after thermal conductivity measurements in a Hot Disk Thermal Constants Analyzer (redrawn based on the Hot Disk Thermal Constants Analyser-Instruction Manual^[31])

Free convection effects or uneven signal penetration in the samples give rise to a data points scatter in the residual plot taking a sine-wave like shape. Free convection often occurs in liquid samples, whereas, if the solid samples contain voids, the entrapped moist air could also give rise to some free convection effects. For solids without considerable voids, uneven signal penetration also causes a sine-wave shaped residual plot. The thermal conductivity will be overestimated (as compared to the real thermal conductivity of the sample) if: free convection occurs, the heat signal reaches the sample boundary; a too high power is applied; and/or a too long measurement time is used. Conversely, thermal conductivity will be underestimated if a too low power and/or a too short measurement time is used^[31].

Figure 3. The influence of too low/high power and too short/long time in the Hot Disk measurements on (a) solid-state and (b) liquid-state (Here, dT: temperature variations (in K) with + and – indicating the direction of shift, FC: free convection, BE: signal exceeds the sample boundary and USP: uneven signal penetration)

Therefore, when testing samples in liquid state, it is of highest importance to avoid free convection process. One way of accomplishing this is to limit the testing time to so short durations that any movement in the fluid induced by heating the sensor is undetectable. Because of the restrictions on measurement times, it is normally advisable to use sensors with the smallest possible radii. As was mentioned, another important parameter while testing the thermal conductivity using Hot Disk Thermal Constants Analyzer is the applied heating power. Simply, the heating power is the constant effect sent to the sensor during a measurement. The aim of correctly setting the heating power is to acquire a suitable total temperature increase, typically 2-5 K when using a TPS-2500S^[31]. The thermal conductivity of the material influences the level of power that should be used. For example, insulation materials having low thermal conductivity require only a very small power in mW range, while highly conductive materials can handle much higher powers without risking sensor damage^[31].

Another important factor to consider while measuring the thermal conductivity is the measured probing depth. The measured probing depth needs to be smaller than the available probing depth. This is necessary to avoid the heat wave reaching the sample boundary or sample holder wall during the measurement time, which if happens, violates the infinite medium assumption^[31].

As another important step when analyzing the residual plot to obtain the correct thermal conductivity, a few of the very first data points and very rarely a few of the last points of the plot need to be excluded from the analysis. This ‘cutting’ procedure is necessary since the first few data points may contain interferences caused by the extra resistance of the sensor insulation material. Whereas, the last few points may contain misinterpretations as the heat signal has most possibly

reached the sample boundary by then. Once these adjustments are performed, the correct thermal conductivity is calculated by the Hot Disk software^[31].

3.1.2 Erythritol-Xylitol Thermal Conductivity Measurements Validation

In the investigation by del Barrio et al., 2017^[24] the same instrument (TPS-2500S) and the same sensor Hot Disk 7577 (2 mm radius) insulated with Kapton has been used for testing the same materials (except for the 80 mol% Er blend). Thus, in this study it was decided to work with same size of sensor (7577), whereas, more rigid insulation material (polytetrafluoroethylene, PTFE) was used to keep the coil of the sensor in position. Among the studies employing the TPS method (c.f. **Table 3**), only del Barrio et al., 2017^[24] have specified both the heating power and time used in the solid-state thermal conductivity investigations. However, even in their work, the specific input parameters used for the liquid-state measurements were not clear, besides abiding to the methodology proposed by Warzoha and Fleischer, 2014^{[46], [47]}. Thus, in the present work, the same sized sensor was used, aiming to reproduce previously reported data using the reported heating power and measurement time (by del Barrio et al., 2017^[24]) and develop the testing method for both solid and liquid states of the remaining polyol blends. Although the liquid-state parameters were unclear in literature, in this study it was decided to start with the same solid-state conditions of del Barrio et al., 2017^[24] for the liquid-state as well. The consequent thermal conductivity measurements are discussed in detail herein.

Examples of the residual graphs are presented below for the tests performed in the present work on solid erythritol at a temperature of 20 °C with two different test settings: heating power 25 mW and time 4 s (c.f. **Figure 4**) and heating power 20 mW and time 3 s (c.f. **Figure 5**).

Figure 4. Residual plot for a single thermal conductivity test performed for solid erythritol at 20 °C (heating power 25 mW, time 4 s), indicating a thermal conductivity of 0.583 W·m⁻¹·K⁻¹

Figure 5. Residual plot for a single thermal conductivity test performed for solid erythritol at 20 °C (heating power 20 mW, time 3 s), indicating a thermal conductivity of $0.585 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$

As was mentioned in section 3.1.1, in the ideal case, the residual plot should be a random scatter around a horizontal line of 0 K variations. When this condition is obtained, the model fits the measurement data and the results are concrete. However, there are several common deviations (detailed in section 3.1.1) such as: large fluctuations in the initial part of the graph, pronounced sine wave shape, and deviations in the later part of curve. As seen in **Figure 4**, the residual plot shows slight sine wave-like shape that may be the result of uneven penetration of signal within the sample resulting in some misleading results or free convection when too high heating power or/and too short/long time are applied. Thus, it is recommended to repeat the measurement after allowing sufficient waiting time so that the sample temperature decreases to the initial testing temperature (controlled by the thermostat bath) while trying to apply other heating powers and/or times. In **Figure 4**, the whole curve is shifted more toward positive temperature difference in the y-axis, and therefore, based on the previous experience of testing different materials it can be decided that lower heating powers should be applied. Moreover, too many agglomerated points (uneven scattering of points) can indicate the use of a too high heating power.

Interestingly, in the present investigation, applying the same measurement conditions (i.e., input parameters) as in the study by del Barrio et al., 2017^[24] did not result in similar results. That is, the measured thermal conductivities which are up to 25% lower than those of del Barrio et al., 2017^[24], for four repetitions with the same chosen input parameters on pure erythritol, xylitol and 25 mol% Er, were obtained.

Therefore, it was decided to repeat the measurement and apply a lower heating power of 20 mW and a shorter time of 3 s. The resultant residual plot for this thermal conductivity test performed for solid erythritol at 20 °C is presented in **Figure 5**. Additionally, applying different input parameters for solid erythritol resulted in very similar thermal conductivity results giving more

confidence in these obtained results. These obtained thermal conductivity results, summarized in **Table 3**, vary compared to other previously reported values by numerous studies^{[19], [26]-[29], [36]-[45]}.

As was mentioned before, the liquid-state investigation input parameters used by del Barrio et al., 2017^[24] were unclear in their published work. Nevertheless, in the present study it was decided to apply the same high heating power and long time (25 mW and 4 s) used on the solid erythritol to validate the testing methodology that could be applied for liquid erythritol. An example of these results is presented in the residual plot in **Figure 6**. As seen in **Figure 6**, the residual plot exhibits a sharp sine wave-like shape that may be the result of the unstable temperature in the sample or free convection when too high heating power or/and too short/long time were applied. Thus, the thermal conductivity tests were repeated at different heating powers and/or times. Based on the previous experience of testing liquids at high temperatures, it was here decided to decrease both the heating power and testing time. **Figure 7** presents the residual plot for one such single test performed for liquid erythritol at 125 °C with a heating power 20 mW and a time of 3 s. Applying the same testing input parameters as in the case of solid erythritol resulted in thermal conductivity results for liquid erythritol similar to those reported by Wang et al., 2016^[27], Höhlein et al., 2017^[26] and Puupponen et al., 2016^[36].

Figure 6. Residual plot for a single thermal conductivity test performed for liquid erythritol at 125 °C (heating power 25 mW, time 4 s), indicating a thermal conductivity of 0.486 W·m⁻¹·K⁻¹

Figure 7. Residual plot for a single thermal conductivity test performed for liquid erythritol at 125 °C (heating power 20 mW, time 3 s), indicating a thermal conductivity of $0.320 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$

Figure 8, a residual plot of a single thermal conductivity test performed for liquid xylitol at temp 110 °C (heating power 25 mW, time 4 s), also shows the effect of free convection, with a distinct sine wave shape. Moreover, in that, slightly lower thermal conductivity values were obtained as compared to del Barrio et al., 2017^[24]. Here, despite decreasing either the heating power or time, some free convection was still observed. Thus, tests were repeated at different heating powers between 20 and 22mW, and times 2 and 3 s to further decrease the free convection occurrence and confirm the thermal conductivity results. As shown in **Figure 9** the most coherent thermal conductivity results for liquid xylitol were obtained for the input parameters of 20 mW and 3 s. Remarkably, applying these same input parameters as in the case of liquid erythritol resulted in thermal conductivity results for liquid xylitol similar to those reported by del Barrio et al., 2017^[24], Höhle et al., 2017^[26] and Puupponen et al., 2016^[36].

Figure 8. Residual plot for a single thermal conductivity test performed for liquid xylitol at 110 °C (heating power 25mW, time 4s), indicating a thermal conductivity of $0.377 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$

Figure 9. Residual plot for a single thermal conductivity test performed for liquid xylitol at 110 °C (heating power 20mW, time 3s), indicating a thermal conductivity of $0.405 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$

Figure 10 and **Figure 11** present the residual plots each for a single thermal conductivity test performed for solid xylitol at 20 °C using two different heating power and time combinations. As seen in **Figure 10**, the residual plot for a single thermal conductivity test performed for solid xylitol at temperature of 20 °C (heating power 25 mW, time 4 s) also displayed the effect of free convection. Thus, tests were performed at different heating powers between 18 and 30 mW and times between 2 and 3 s for solid xylitol at 20 °C. From those, the most coherent results were obtained for the input parameters 20 mW and 3 s as shown in **Figure 11**. Interestingly, applying the same input parameters as in the study by del Barrio et al., 2017^[24] did not result in similar

results. In contrast, a significantly lower thermal conductivity value, consecutively for four repetitions using the same input parameters, was obtained. Curiously, in the present work, the application of the same input parameters used on liquid xylitol (i.e., 20 mW and 3 s) gave rise to thermal conductivity results for the solid xylitol similar to those reported by Höhlein et al., 2017^[26] and Puupponen et al., 2016^[36].

Figure 10. Residual plot for a single thermal conductivity test performed for solid xylitol at 20 °C (heating power 25mW, time 4s), indicating a thermal conductivity of $0.359 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$

Figure 11. Residual plot for a single thermal conductivity test performed for solid xylitol at 20 °C (heating power 20mW, time 3s), indicating a thermal conductivity of $0.375 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$

Figure 12 and **Figure 13** present the residual plots each for a single thermal conductivity test performed for the solid 25 mol% Er using two different heating powers and times. The thermal conductivity tests (heating power 25 mW, time 4 s) performed for the solid 25 mol% Er showed

slightly higher values compared to the results of del Barrio et al., 2017^[24]. Nevertheless, the thermal conductivity results obtained by using the input parameters of 20 mW and 3 s showed results rather similar to those reported by del Barrio et al., 2017^[24].

Figure 12. Residual plot for a single thermal conductivity test performed for solid 25 mol% Er at 20 °C (heating power 25 mW, time 4 s), indicating a thermal conductivity of $0.390 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$

Figure 13. Residual plot for a single thermal conductivity test performed for solid 25 mol% Er at 20 °C (heating power 22 mW, time 3 s), indicating a thermal conductivity of $0.387 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$

Figure 14 presents the residual plot for a single thermal conductivity test performed on the 25 mol% Er in the liquid state, using the heating power 20 mW and time 2 s. As seen (c.f. **Table 3**), the thermal conductivity tests using different heating powers and times showed rather similar results compared to the values presented by del Barrio et al., 2017^[24] for 25 mol% Er.

Figure 14. Residual plot for a single thermal conductivity test performed for liquid 25 mol% Er at 110 °C (heating power 20mW, time 2s), indicating a thermal conductivity of $0.410 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$

Besides the examples presented here, numerous residual plots obtained within the entire measurement regime were comprehensively scrutinized in this work. The most coherent residual plots were chosen through such a careful scrutiny, while discarding the other over- and under-estimating residual plots. Thereby, the optimal measurement conditions and thus the final thermal conductivities of these polyols were identified. The over- and under-estimating residual plot examples shown here (e.g. **Figure 4**, **Figure 6**, **Figure 8**, **Figure 10** and **Figure 12**) only concern the use of too high powers and too long times. Whereas, the entire measurement regime (c.f. **Table 2**) consists of too low powers and too short times as well, besides the optimal conditions. However, residual plot examples for such too low powers and too short times are excluded here in this discussion, to avoid repetition, because these are similar by appearance to the presented examples herein.

Overall, as shown along numerous examples of the residual plots obtained throughout the thermal conductivity evaluation of this polyols system, it is clear that each and every material required a sequence of input parameters to finally achieve coherent results. Therefore, it is always essential to see how the variations in the input parameters influence the residual plot and to choose the correct data scatter, avoiding free convection and still ensuring sufficient signal penetration. Concerning the TPS method, it is therefore of utmost importance to specify the measurement conditions such as solid/liquid state, the measurement temperatures, and the specific input parameters, i.e., the heating power and time, used to conclude on each material's thermal conductivity.

The solid-state thermal conductivity of materials is influenced by the actual solid-state that is analyzed, if the material can have metastable states and/or different polymorphic structures. Many materials in nature have such metastable and/or polymorphic structures almost all the time. These various states formation is governed by their solidification (i.e., the cooling) rate. That is because,

materials are prone to metastability when the cooling rates are insufficiently slow to reach thermodynamic equilibrium. In reality, materials often solidify under such too fast conditions, unless this is specifically controlled. Thus, the thermal conductivity discrepancies between many of these literature and this work could also be due to the differences in the solid state that was analyzed, triggered by different cooling rates. Therefore, on one hand, the systematic control of the sample cooling rates (to achieve stable phase changes) along with transparent reporting of that procedure and the thermal conductivity results is imperative. On the other hand, for practical applications that very often employ fast cooling rates, which thus could trigger metastable states in PCMs, it is crucial to determine the thermal conductivity of these metastable states as well.

In this work, the sample solidification was performed by natural cooling of the hot liquid samples within room temperature, without any systematic control of the cooling rate otherwise. However, a minute amount of the original crystals in powder form were seeded into these liquid samples to facilitate the growth of their stable crystalline form. In the blends' case, with the same expectations, the physically mixed polyol powders of the corresponding blend composition were seeded into the supercooled liquids. This overall sample solidification procedure is nevertheless one aspect that can be improved in future, to obtain more accurate and repeatable thermal conductivity results.

Repeatability of the measurements directly influence the measurement reliability. In this thermal conductivity evaluation using the TPS method, each sample test was repeated at least up to four times at each chosen power and time condition set. However, at the end of these tests, it was observed that not all the tests yielded coherent results for each power and time input parameters set. Thus, in the cases when there were not many coherent results per parameters set, some other power and time conditions were also compared (e.g. for solid erythritol, solid xylitol and 25 mol% Er in liquid state). Their standard deviations are summarized along with the measurement power, time and measured thermal conductivities in **Table 4**. For the liquid state of erythritol and 25 mol% Er, however, only two instances of coherent result were obtainable per composition, from seven repetitions. A possible main reason for these incoherent results relates to the challenge in avoiding free convection in the liquid-state.

All the measured compositions nonetheless produced very consistent results with very small standard deviations (within 0.001-0.036 W·m⁻¹·K⁻¹) between these repetitions, as **Table 4** shows. Thus, it can be concluded that the measurements in the present work have very satisfactory repeatability.

Although this thermal conductivity analysis is restricted to the TPS method and polyols, it indeed exemplifies the necessity of transparency in the procedural aspects that are essential to maintain repeatability and thereby to achieve comparable results. This necessity is universally valid for all the thermal conductivity assessment methods, where, each method will have its own set of conditions crucial for repeatability. Then again, for each material, a number of evaluations are inevitable before coherent results can be obtained, and there are no universal measurement conditions that fit all materials.

Table 4. Standard deviations of the final measured thermal conductivities of erythritol, xylitol, 80 mol% Er and 25 mol% Er in this work using the TPS method for up to four measurements repetitions. (S: solid, L: liquid, Er: erythritol)^a

Material	S/ L	Power, Time (mW, s)	Thermal Conductivity ^c (W·m ⁻¹ ·K ⁻¹) for up to 4 repetitions	Average (W·m ⁻¹ ·K ⁻¹)	Standard deviation (W·m ⁻¹ ·K ⁻¹)
Erythritol	S	22,3	0.593; 0.592; 0.593; 0.593	0.593	0.001
	L	20,3	0.322; 0.320	0.321	0.001
80 mol% Er ^b	S	20,2	0.558; 0.546; 0.553; 0.553	0.553	0.004
	L	20,2	0.364; 0.364; 0.363; 0.360	0.363	0.002
Xylitol	S	20,3	0.378; 0.371; 0.372; 0.370	0.373	0.003
	L	22,2; 20,2; 20,2; 20,3	0.454; 0.388; 0.479; 0.412	0.433	0.036
25 mol% Er ^b	S	20,3; 20,3; 20,4; 20,4	0.383; 0.399; 0.396; 0.395	0.393	0.006
	L	20,2	0.404; 0.401	0.402	0.001

^ain this study the experimental pressure was not controlled beyond atmospheric pressure (101±2 kPa)

^bwith the expanded uncertainty with a 0.95 confidence level: 1.3 mol%, and ^can uncertainty of 5%

Round Robin Tests are one effective way of achieving common standards in these experimental investigations of thermal conductivity, where one such is currently being instigated at the IEA Annex 33/Task 58^[48]. Similar international experts' collaborative networks are valuable platforms for reaching standardization of thermal conductivity investigations (among other experimental assessments) in the PCM design context.

4 Conclusions

The thermal conductivities of xylitol, its blends with erythritol: 25 mol% erythritol and 80 mol% erythritol, as well as erythritol are found in this work using the TPS method, in the solid state respectively to be: 0.373; 0.394; 0.535 and 0.589 W·m⁻¹·K⁻¹, and in the liquid state respectively to be: 0.433; 0.402; 0.363 and 0.321 W·m⁻¹·K⁻¹. When comparing these thermal conductivities with available literature in the PCM-context so far, clearly, major discrepancies exist particularly on the values for erythritol and xylitol. The main reasons for these dissimilarities are the missing measurement and sample preparation conditions, and standards to abide to, for comparability. Thermal conductivity directly influences the heat exchanger design and thus the TES system functionality. Incomparable thermal conductivities could hence negatively affect the expected TES system function. Therefore, as shown here, not only on polyols, but overall in the PCM-context, a major need exists in standardizing the thermal conductivity measurement approaches and data reporting, for accurate design of TES systems.

Moreover, this study shows that it is necessary to test the same materials with different input parameters to verify the obtained results as well as to check the sample sensitivity to free convection especially in the liquid state. For instance, in the use of the TPS method, a material

needs to be investigated while varying the input parameters such as the heating power and time. Further studies of the thermal conductivity of different polyols are recommended to validate the data presented herein. The systematic control of the sample's cooling rates, even beyond simple steps of cooling and seeding as used herein, to obtain the expected solid states is another important future consideration to obtain more consistent thermal conductivity measurements.

Overall, to achieve comparable thermal conductivity results, abiding to transparent and repeatable data and results reporting is primordial. For that, establishing and maintaining standards in the relevant context is imperative. This could be achieved through international collaboration platforms working towards such common goals.

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