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High-throughput experiment for the rapid screening of organic phase change materials

Clément Mailhé¹ · Stéphane Gorsse² · Boèce Thirion³ · Elena Palomo^{4,5} · Marie Duquesne³

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Abstract

An emerging dynamic calorimetry method based on infrared thermography (IRT method) has recently proven to be of great interest for the fast establishment of the phase diagrams of binary systems of organic materials (sugar alcohols, fatty acids, fatty alcohols). The methods allowed for their determination in a single 2-h experiment when standard techniques require weeks of measurements hence enabling a significant improvement of the efficiency of material screening processes. As adding a component to the system greatly increases the number of compositions to be studied (21 for binary, 231 for ternary assuming a 5 mol% increment between compositions), the IRT method may be of even greater use with increasing system complexity. This work then aims at studying the applicability of IRT for the fast establishment of phase diagrams of ternary systems with the lauric (LA), myristic (MA), and palmitic (PA) fatty acids ternary system as an illustrative case. A phase diagram in agreement with the results of the literature and with DSC measurements is established using IRT in a few hours-experiments instead of weeks to months with standard methods. The obtained results (*e.g.* 67 mol%LA + 23 mol%MA + 10 mol%PA for the eutectic composition, occurrence of a ternary eutectic reaction at 32 °C) are promising. This technique also successfully allows for rapid exploration of the phase diagram: reconstruction of the liquidus surface, identification of the eutectic transition, 4-phase equilibrium. IRT is a particularly interesting high-throughput characterization method for the establishment of the phase diagrams of ternary (or higher-order) systems.

Keywords Infrared thermography · Phase diagram · DSC · Ternary systems · Eutectic transition

Introduction

Phase diagrams are essential tools in materials science. They define areas of existence of phases according to pressure, temperature, and chemical composition for pure and

multi-component systems of materials [1]. Their establishment hence provides valuable information to the scientist, often decisive depending on the field of application [2–4]. Standard experimental methods are categorized as either static or dynamic and rely on the observation of a material property (structural, physical, thermal, chemical ...) at different temperature levels (static) or during a temperature profile (dynamic) [5]. For multi-component systems of materials, the determination of an accurate phase diagram requires a large number of experiments making the phase diagram establishment process a rather time-consuming task. Indeed, traditional methods are only able to perform single sample characterization which is not competitive in an industrial development context. The use of phase change materials (PCM) in thermal energy storage (TES) is a good example to illustrate the benefits of phase diagrams. Indeed, the thermal behavior and melting temperature of PCMs are critical properties that can be tailored using blends instead of single component PCMs. It is then of great importance with respect to the performance of finding blends without solidification

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interval, i.e. with congruent melting or eutectic. Therefore, phase diagrams are the road map to select blends with the desired melting temperature and solidification behavior. However, the exploration of the vast compositional space of multicomponent systems requires a large number of samples (e.g. 231 for a ternary system and 1771 for quaternary system with a 5 mol% step increment) [6]. To speed up the exploration, we need high throughput techniques that can be automated, parallelized, and miniaturized [7], which is not the case for the most common techniques being used such as differential scanning calorimetry (DSC), differential thermal analysis (DTA), or X-ray powder diffraction (XRD) [1, 2, 5, 8–12]. To address this issue, modeling tools are commonly employed to predict multi-component phase diagrams from known lower-order subcomponent systems [13, 14]. While this approach is well developed for metals and alloys, organic materials are still poorly described in the literature and thermodynamic databases are not currently available for these systems. In order to overcome the limits of traditional methods, a high throughput technique based on infrared thermography (IRT method) was developed. Its application for the assessment of binary phase diagrams has shown interesting results for mixtures of sugar alcohols [15] and of fatty acids and fatty alcohols [16], respectively [15, 16]. It allowed for the rapid screening of the liquidus temperature in a single 2-h experiment while traditional methods would require weeks of measurements. Due to its promising aspect, the method is currently undergoing a campaign of parametric studies [17–19] to enhance and optimize its performance. As the more components, a system includes the more time-consuming the phase diagram establishment process is, the IRT method might be of increasing interest with the growing complexity of the system.

In this paper, we present high-throughput experiments that allow simultaneous measurement of the liquid–solid transition temperatures of several blends, and to quickly establish the liquidus surface of multi-component systems. Our method combines materials libraries with discrete compositions and rapid tests enabling to accelerate the screening tests. This approach is applied to the fatty acids (FA) ternary system with Lauric acid, Myristic acid, and Palmitic acid. The validity of the results is discussed by comparison with DSC measurements and with data from the literature.

Materials and methods

Materials

The ternary system studied in this work is the Lauric acid + Myristic acid + Palmitic acid system. Information regarding the properties of the pure compounds is given in Table 1. Indicated melting temperatures and their corresponding standard deviation are obtained by DSC following the protocol described hereafter. Temperature ranges found in the literature [20–29] are also stated for comparison.

Each binary system obtainable from the 3 materials considered has already been studied with the IRT method [30, 31] and the results obtained have been validated by comparison with DSC measurements and literature data. With a 5 mol% increment, the number of samples to be studied is 231 (171 to be studied in this work and the 60 other samples being studied for the establishment of the binary systems phase diagrams in previous work [30, 31]). Although this number may still appear high for standard methods such as DSC, analyzing all the samples can be managed in a matter of hours with the IRT method.

The preparation of each sample is made using a METTLER TOLEDO weighing scale with a 0.03 mg uncertainty. Batches of 200 mg are prepared by first weighing the first compound in its solidified form after being melted in an aluminum weighing pan and then weighing the second compound following the same process.

IRT method

A recent innovative high-throughput technique based on infrared thermography has recently proven to be particularly adapted for the fast establishment of phase diagrams of binary systems of organic materials [15–19, 31]. The infrared thermography (IRT) method has successfully provided the phase diagrams of sugar alcohols, fatty acids, and fatty alcohols binary systems in a single experiment of less than two hours. Its advantage resides in its ability to study multiple samples at a time, hence greatly accelerating the step of phase change characterization. The process of phase diagram establishment requires the study of an extensive amount of compositions. If for binary systems a reliable determination can be made based on a manageable number

Table 1 Properties of the pure compounds studied

Material	Acronym	Supplier	Purity/%	Melting temperature/°C	
				This work	[20–29]
Lauric acid	LA	Acros Organics	99	43.40 ± 0.11	42.44–45.29
Myristic acid	MA	Acros Organics	99	53.48 ± 0.04	55.23–55.58
Palmitic acid	PA	Sigma-Aldrich	99	62.14 ± 0.02	61.45–64.35

of samples, the requirements become increasingly demanding as new compounds are added to the system. In that frame, the IRT method, whose efficiency has already been demonstrated for binary systems [15–19, 31], could prove to be of great interest for the phase diagram establishment of systems including more than two compounds. It could also effectively participate in the large gathering of experimental data, supplying modeling databases with useful information regarding organic materials. The principle of the method has been thoroughly described in [15] resulting from various studies [32] dedicated to the study of phase transition processes for the study and development of phase change materials. The presented experimental parameters have been chosen according to the results obtained in these previous studies. The readers are encouraged to consult these studies for an in-depth understanding of the choices made regarding the operating conditions.

Multiple samples of the system of materials are deposited in the form of droplets onto a thermally conductive plate. This plate is then submitted to a slow heating ramp monitored with a thermocouple placed on the support plate. In this study, the heating rate is set at 1 °C min^{-1} from 20 to 75 °C in order to encompass the majority of occurring transitions. An infrared camera placed above the plate simultaneously records the thermal scene as the samples are submitted to the ramp. The camera used in this work is the FLIR X6580SC, and the acquisition rate is set at 7 Hz. An image sequence is then obtained containing the data of 30 to 40 samples at a time in a single experiment lasting less than 2 h. Considering the 5 mol% increment chosen for this work, the 171 samples can all be studied within 12 h of experiments. Figure 1 presents a scheme of the experimental setup.

As only the data of the samples is useful and as each sample is represented by hundreds of pixels, image, and signal processing techniques are applied prior to analyzing the data in order to facilitate the interpretation of the results. As explained in [15], the structure modifications occurring during the phase change process induce a variation of the emissivity of the material. This change in emissivity can then be noticed in the infrared signal recorded by the camera and consequently lead to the identification of phase transitions. The detail of applied processes is available in [15]. Following their implementation, the data interpretation for each sample consists in detecting abrupt trend changes in 2–3 eigenfunctions, attributed to phase transitions. The eigenfunctions are obtained through a singular value decomposition (SVD), which is a common tool in signal processing when dealing with large multi-variable datasets that allow separating variables and highlighting variations in the raw data.

Figure 2a shows the raw infrared signals for a sample of pure Palmitic acid. As is, the signals are not manageable and cannot allow for a reliable interpretation of transition

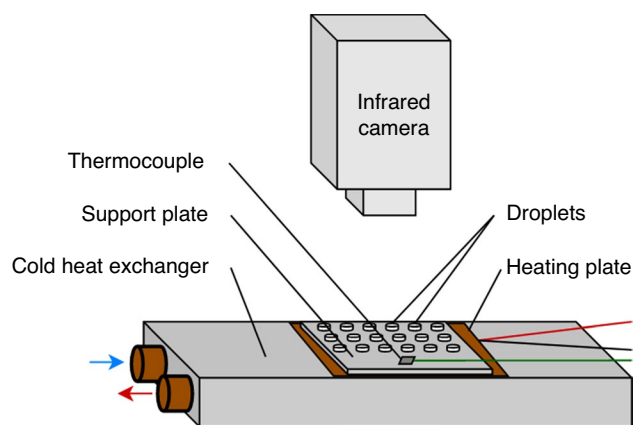


Fig. 1 Scheme of the experimental setup

temperatures. In that case, a transition can either be misplaced or lost in the great amount of data gathered. After SVD, we notice in Fig. 2b that the first three eigenfunctions show easily identifiable variations which greatly facilitates the interpretation. As demonstrated in [15], the first three eigenfunctions are sufficient to detect most occurring transitions, the first eigenfunction generally being a representation of the average signal while the other two express variations from it. This consideration is all the more valid when several transitions occurring successively have to be distinguished in the cluster of data (which is often the case for mixture samples).

Validation

The IRT method is an emerging technique whose results are to be validated by measurements obtained by established standard methods. The most commonly used for phase diagram establishment is DSC. It is consequently the technique used in this work to validate the results obtained with the IRT method as well as the results extracted from literature.

The device used is a DSC 131 model provided by SETARAM, calibrated for reliable measurements in the 20–330 °C range using Gallium (Purity: 99.9999%), Indium (Purity: 99.995%), Tin (Purity: 99.999%), and Lead (Purity: 99.999%). The samples passing through the DSC come from the same batches used for the samples of the IRT experiment. A total of 22 compositions are chosen in order to provide an adequate depiction of the ternary phase diagram as analyzing all the samples studied in the IRT experiment would not be possible due to the time-consuming aspect of the DSC. For each sample, a mass between 10 and 15 mg is weighed and placed in 30 μL aluminum crucibles and 3 heating–cooling cycles are performed at 1 °C min^{-1} in order to match the operating conditions of the IRT method. Each cycle begins at 20 °C and ends at 75 °C to be sure encompass most occurring transitions. The first cycle is used

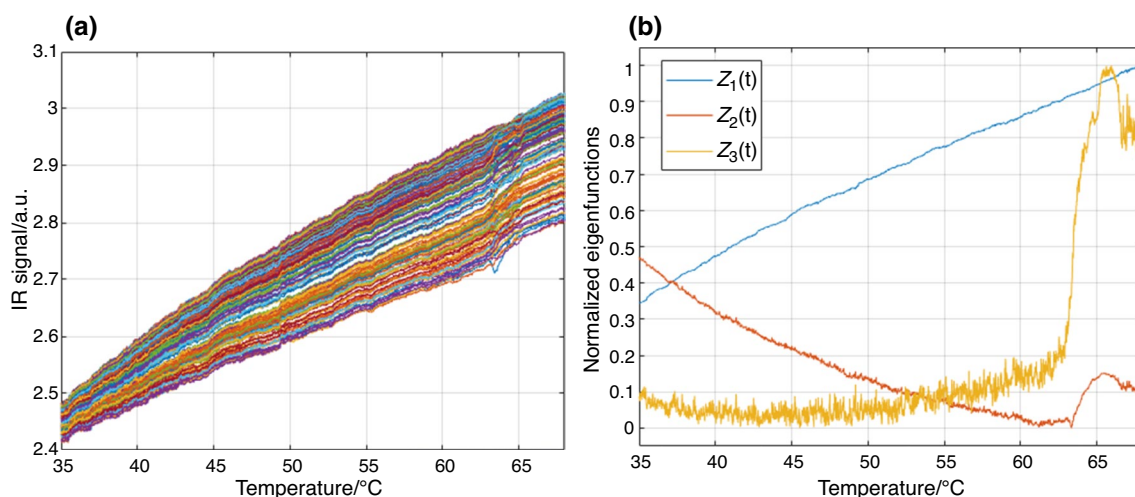


Fig. 2 **a** Raw infrared signals for a sample of Palmitic acid; **b** First three time-eigenfunctions for the same sample.

to ensure the sample adheres to crucible and to delete any thermal history.

The interpretation of the DSC curves is made based on the guidelines in [33] stating that for transitions temperatures, the onset temperature is considered if a clear baseline can be identified and the peak temperature is chosen otherwise.

The liquidus temperatures for 22 blends extracted from IRT and DSC, respectively, are compared in Fig. 3. This graphical representation highlights the good agreement that has been found so far between both techniques.

Results and discussion

Figure 4a shows the complex surface connecting the 231 liquidus temperatures obtained by IRT for the LA+MA+PA ternary system. In ternary systems, the liquidus surface bounds two-phase Liquid + Solid (L+S) fields. Considering that the binary sub-systems involved in this ternary include eutectic and peritectic reactions [30, 34–36], there are numerous two-phase regions (L+S) whose edges meet to form a common line bounding three-phase regions. The most obvious that can be identified in Fig. 4a is the eutectic valleys emerging from the neighboring binary eutectic reactions $L=LA+PA$, $L=LA+MA$, and $L=MA+PA$. Another interesting feature in Fig. 4a is the presence of a minimum at around 32 °C corresponding to the lowest melting point of the ternary system at 70 mol%LA + 20 mol%MA + 10 mol%PA. This reveals the occurrence of either an isomorphous congruent minimum melting or a ternary eutectic reaction $L=LA+MA+PA$.

In addition to the liquidus surface, the IRT method evidenced isothermal transitions spanning over large composition ranges at around 32, 37, and 51 °C (Fig. 4b). In ternary

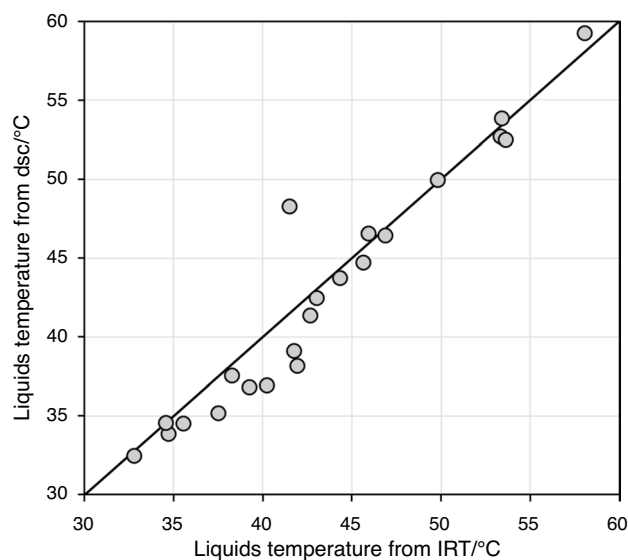


Fig. 3 Graphical representation of the agreement between the IRT method and a standard method (DSC)

systems, 2-dimensional isothermal regions connect the compositions of phases participating in four-phase equilibria. Even though it is not possible to extract the compositions of the phases from Fig. 4b, the isotherm detected at around 32 °C in the LA-rich compositional space allows discriminating the type of phase transition that occurs at this minimum melting temperature as a ternary eutectic transformation. The nature of the other ternary four-phase equilibrium at 37 and 51 °C cannot be determined with the current data, however, the peritectic reactions extending from the binary sub-systems suggest that ternary four-phase equilibria are class II, i.e. intermediate between eutectic and peritectic equilibria, and/or class III, i.e. ternary equivalent of peritectic equilibrium.

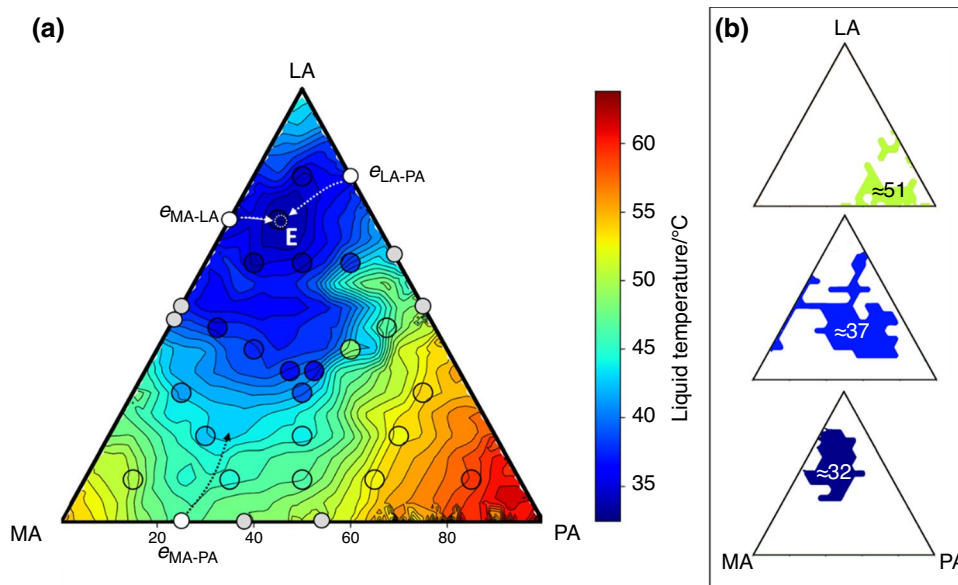


Fig. 4 Phase diagram reconstruction from IRT. **a** Liquidus surface temperature map of the ternary system LA+MA+PA obtained 231 samples simultaneously analyzed by the IRT method. The DSC results, represented by large circles, are superimposed on the liquidus surface. The same colormap is used for both methods to show the coincidence. The smaller white and gray circles represent invariant

reactions in the binary sub-systems (binary eutectics and peritectics). Binary eutectic and ternary eutectics are denoted by *e* and *E*, respectively. Dotted arrows are guidelines to visualize the eutectic valleys emerging from the binaries and meeting at the ternary eutectic point. **b** Ternary four-phase equilibrium at 32, 37 and 51 °C

The work presented in [37] shows a DSC-determined ternary eutectic composition of 67.13 mol%LA + 22.83 mol%MA + 10.05 mol%PA with a melting point around 31 °C. Further DSC analyses aiming at corroborating the observations made with the IRT method, confirm the existence of a eutectic reaction at this composition (67 mol%LA + 23 mol%MA + 10 mol%PA) and with a melting point matching the one obtained with IRT (Table 2). In addition, the previously mentioned 37 °C isothermal region has successfully been retrieved with DSC measurements, at a temperature level consistent with IRT observations. On the other hand, the thermal event at 51 °C is only detected with the IRT method. The detection appears to be rather reliable considering the small standard deviation but its presence cannot yet be confirmed by DSC. This detection is however, not surprising as similar observations are also made for the binary systems in previous works [30, 31].

The author in [37] attempts predicting eutectic compositions of multi-compounds systems with thermodynamic modeling. For this system, the predicted eutectic composition for this system was 58.69 mol%LA + 28.68 mol%MA + 12.63 mol%PA which is far off the actual composition determined experimentally. This deviation highlights the limits of modeling techniques and the need for cost-effective and time-efficient experimental methods such as IRT.

With the encouraging results obtained for the establishment of a ternary phase diagram with the IRT method, we can conclude that this method can allow the rapid and efficient screening of complex multi-component systems. The transition detection with the IRT method indeed appears to be quite reliable with a retrieved liquidus showing no discontinuities and isothermal regions detected with acceptable standard deviations, within range of standard DSC measurements and spanning over several distinct

Table 2 Average temperature, standard deviation, and number of samples for the isothermal transitions detected with the IRT method and DSC measurements

	$\overline{T_{tr1}}/^{\circ}\text{C}$	$\sigma_{tr1}/^{\circ}\text{C}$	<i>n</i>	$\overline{T_{tr2}}/^{\circ}\text{C}$	$\sigma_{tr1}/^{\circ}\text{C}$	<i>n</i>	$\overline{T_{tr3}}/^{\circ}\text{C}$	$\sigma_{tr1}/^{\circ}\text{C}$	<i>n</i>
IRT	31.96	0.77	43	37.13	0.57	49	50.77	0.62	13
DSC	31.93	0.58	12	36.81	0.65	10	–	–	–

With $\overline{T_{tr1}}$ the average transition temperature of transition *n*°1 in °C, σ_{tr1} the standard deviation of the transition temperature for the transition *n*°1 and the *n* number of samples for which the transition has been detected

experiments. Now that the proof of concept is done, we can proceed to the screening of other ternary / quaternary systems in order to explore their employability in various applications

Conclusions

In this work, we have shown that the IRT method is an effective high-throughput technique allowing the automatic, rapid, and low-cost characterization of a broad spectrum of discrete compositions of a blend. It has been successfully implemented for the rapid screening of an LA + MA + PA ternary blend. Its phase diagram obtained in this work consisted of 171 data sets obtained in less than 12 h of experiments which is considerably less than the time that would have been required with the use of standard methods (weeks/months). An isotherm detected around 32 °C in the LA-rich compositional space suggested the occurrence of a ternary eutectic reaction whose location, both in terms of temperature and composition, was confirmed and found to match literature data and DSC measurements. At this eutectic composition, the ternary system behaves like a single phase with a congruent fusion. Being able to rapidly obtain both the eutectic composition and its melting temperature is a tremendous asset of the IRT method. Indeed, it enables the easy identification of material properties in temperature-based applications such as thermal energy storage and motivates the choice to either further study the composition or continue the screening of other systems. This technique also allows for rapid exploration of the phase diagram: reconstruction of the liquidus surface, identification of the eutectic, 4-phase equilibrium. Both pave the way for the modeling and prediction of phase diagrams in these systems and for the rapid screening of the composition space in the search of suitable blends in a wide range of applications.

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