

Sugar Alcohol based Materials for Seasonal Storage Applications

## NEWSLETTER

### An insight in SAM.SSA's current research

<b>Project acronym:</b>	SAM.SSA
<b>Project full title:</b>	Sugar Alcohol based Materials for Seasonal Storage Applications (FP7-ENERGY-2011, collaborative project)
<b>Project No.:</b>	296006
<b>Project duration:</b>	36 months (01/04/2012 – 31/03/2015)
<b>Requested EU Contribution:</b>	2,941,997 €

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### EDITORIAL

The SAM.SSA project aims at developing new Phase Change Materials (PCMs) for thermal energy seasonal storage applications in the range of medium temperatures.

PCM are rarely proposed for seasonal thermal energy storage due to i) insufficient energy densities and ii) the high risk of PCM solidification during the storage period caused by poor insulation inadequate for maintaining temperatures beyond the melting point. These problems will be overcome by the materials proposed in SAM.SSA.

SAM.SSA is developing molecular alloys based on sugar alcohols. These molecular alloys allow for adjustment of the melting point and lead to a significantly increased energy density compared not only to that of their components, but also to that of n-alkanes molecular alloys. Furthermore sugar alcohols permit high levels of undercooling thus minimizing the risk for spontaneous PCM solidification and at the same time reducing insulation requirements as well as thermal losses during long term storage. The application of a local shock or ultrasound will induce nucleation and subsequent crystallization thus provoking easy discharge of the storage system.

Like most PCM, sugar alcohols due to their low thermal conductivity (typically  $< 1 \text{ W/m/K}$ ) impose a principal heat transfer problem on the storage design. SAM.SSA research will overcome this problem by increasing the MASA thermal conductivity using low-cost, tailor-made carbon porous structures, and increasing the specific area of heat exchange through MASA macro- or micro-encapsulation with organic, inorganic and hybrid shells.

## Sugar Alcohols Blends

"Sugar replacer", "sugar alcohol", "hydrogenated carbohydrate", and "polyols" are synonyms for a sub-class of carbohydrates present in foods. The defining characteristic is the occurrence of an alcohol group ( $>CH-OH$ ) in place of the carbonyl group ( $>C=O$ ) in the aldose and ketose moieties of mono-, di-, oligo- and polysaccharides. The most commonly used polyols are Sorbitol, Mannitol, Xylitol, Lactitol, Malititol, Erithritol and Isomalt.

The potential of polyols for compact thermal energy storage at low/medium temperature have been investigated using available thermodynamic data for mono- and disaccharide polyols. Polyols with linear, cyclic or ramified carbon chain structures, and number of carbons atoms ranging from 3 to 12, have been considered. As shown in Fig. 1, there is an upper limit for the compactness achievable from sugar alcohols which is defined by monosaccharide polyols with linear structure. Moreover, these polyols can be roughly classified into three groups: a) polyols with more than 5 carbon atoms, whose melting point is above  $100^{\circ}C$  and that have very high energy density ( $>130 \text{ kWh/m}^3$ ); b) polyols with less than 5 carbon atoms, whose melting point is below  $100^{\circ}C$  and the energy density is less than  $80 \text{ kWh/m}^3$ ; and c) polyols with 5 carbon atoms, whose melting point is close to  $100^{\circ}C$  and that have energy density ranging from  $80-120 \text{ kWh/m}^3$ .

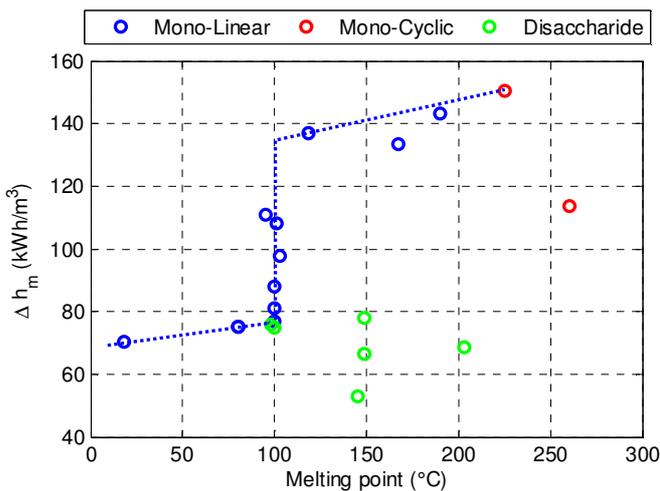


Figure 1: Volumetric enthalpy change by melting vs. Melting point.

Regarding seasonal storage applications, none of these polyols is ideal. Those with very high energy density have melting point above  $100^{\circ}C$ , while those with melting point below  $100^{\circ}C$  provide moderate storage compactness. Therefore, binary systems of linear monosaccharide polyols (25 systems) have been investigated. As shown in Fig.2, eight eutectic blends with melting point less than

$100^{\circ}C$  have been identified. Among them, there is four with enthalpy change by melting higher than  $240 \text{ kJ/kg}$ . They can likely provide total energy density (sensible plus latent heat) as high as  $200 \text{ kWh/m}^3$  under expected working conditions and are studied in deep.

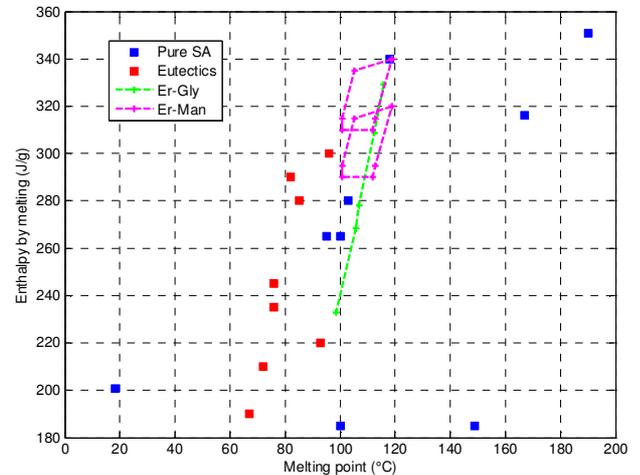


Figure 2: Sugar alcohols and sugar alcohol blends: gravimetric enthalpy change by melting vs. melting point.

The characterization of the new blends as PCM has been carried out. This includes the determination of the melting point and the latent heat, as well as measurements of the specific heat, the density, the viscosity and the thermal conductivity as a function of the temperature. The physical properties are studied in solid and liquid state, as well as in meta-stable state at sub-melting temperatures. Thermochemical stability is still under study.

The thermal performances in such systems are strongly dominated by the nucleation and crystal growth kinetics. Therefore, ongoing work focuses on the experimental study and the modelling of both spontaneous and induced crystallization of these products. This involves quantifying nucleation rates and crystal growth rates as a function of the subcooling. Different techniques to provide a practical and cost-effective way to trigger the crystallization of these products are also under development.

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## Microencapsulation of Sugar Alcohols

In this project, the microencapsulation of sugar alcohols is evaluated as a procedure to increase the specific area of heat exchange and maximize the effectiveness of sugar alcohols as thermal energy storage materials.

Due to the large number of techniques and methodologies developed for the microencapsulation of different compounds and different industrial sectors (cosmetic, food, pharmaceutical, etc.), a screening study on the microencapsulation of sugar alcohols using organic, inorganic or organic-inorganic hybrid shells is being carried out.

Several techniques have been selected to study their feasibility, according to the ease of the process and the up-scaling viability for sugar alcohols with the objective to evaluate microencapsulation procedures for sugar alcohols and analysis of the thermal properties depending on the type of shell.

Different microencapsulation techniques (chemical and physical) have been studied for sugar alcohols with organic shells. Although not optimized results are obtained, the possibility of obtaining sugar alcohol microparticles with organic shells has been demonstrated (Figure 3). The main difficulty is the high reactivity of the  $-OH$  groups in the sugar alcohol, which determines in most cases the type of polymer and the ratio to be used for the microencapsulation. Future work will address the optimization of the experimental parameters for a maximum thermal energy storage and durability.

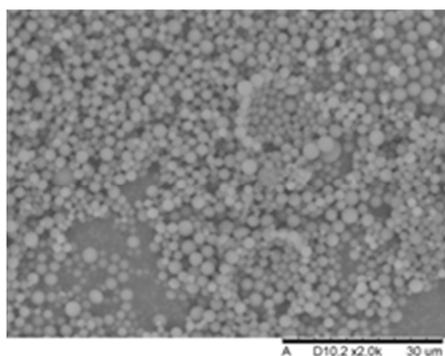


Figure 3: SEM micrograph for D-Mannitol/PMMA microcapsules prepared by solvent evaporation.

A process enabling microencapsulation of sugar alcohols in inorganic ZnO shell with flower like structure based on hydrothermal process followed by spray drying has been developed (Figure 4).

The formation of new compounds between D-Mannitol and ZnO may be considered to explain the XRD and DSC spectra of the microcapsules. Also, future works are needed to better understand the process and optimize the

experimental parameters to increase the encapsulation degree. Moreover, experiments on the encapsulation of sugar alcohols in silica as inorganic shell using micro-inverse emulsion synthesis are being carried out (Figure 5).

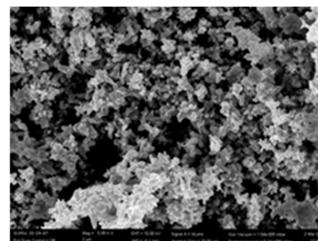


Figure 4: SEM micrographs of D-Mannitol microcapsules in ZnO shell with flower like structure obtained by hydrothermal synthesis followed by spray drying.

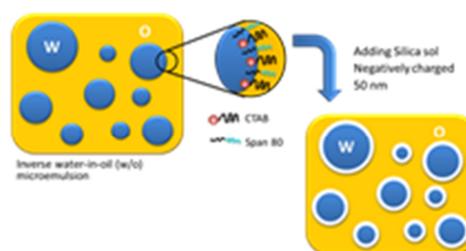


Figure 5: Scheme of the strategy of encapsulation using a negatively charged silica sol.

Encapsulation experiments with hybrid shells (ORMOCER®s) are being carried out using the extrusion equipment. The core material and the shell material are extruded together through a special nozzle (Figure 6). The shell material is UV cured subsequently. The exposition time to the UV radiation is very short so that the core material is altered only to a very low extent.

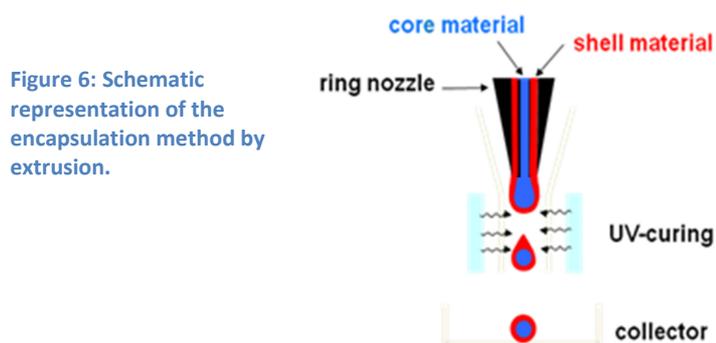


Figure 6: Schematic representation of the encapsulation method by extrusion.

María Dolores ROMERO & Mónica FUENSANTA (AIDICO),  
Roxana PITICESCU (IMNR), Thomas BALLWEG  
(FRAUNHOFER ISC)



## Carbon foams: towards porous materials for thermal management

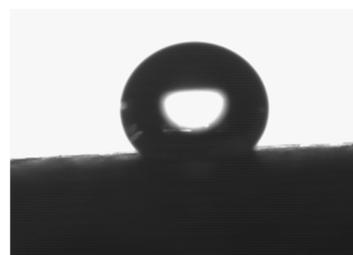
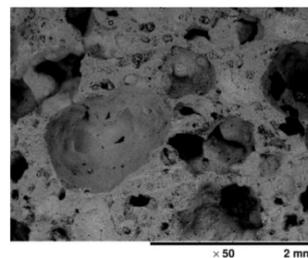
In the framework of the present project, the aim of our work was to provide porous matrices able to host as large amounts of phase-change-materials (PCMs) as possible for storing and recovering heat, through melting and crystallization of PCMs, respectively. Such a feature simultaneously requires high porosity and high thermal conductivity for the porous matrices. However, the latter, two, properties are antagonistic, since the conductivity dramatically decreases as soon as part of the material is replaced by voids. An additional requirement was that the PCMs should remain in the molten state at temperatures far below their equilibrium melting points, in order to recover the heat at the best, desired, moment. For reaching such unusual behaviour, the surface of the inner porosity needs to be properly treated for, in some way, repelling the liquid PCM from the surface. However, the thermal contact should not be lost. A last requirement was to get a material as cheap as possible.

Carbon is a very unique element, as it allows obtaining materials having extremely versatile and controllable properties. Carbonaceous materials can thus present chemical and thermal resistance, electrical conductivity, conductive or insulating thermal character, airtightness or permeability, porosity or not, surface areas sometimes exceptionally high, weightlessness, high mechanical resistance or ductility. This non-restrictive list of sometimes very antagonistic properties is the result of the outstanding variety of the textures that the carbon element may generate. To each particular texture corresponds one given functionality, and the modification of the texture induces that of the related properties. Just compare the black, opaque and ductile graphite, to the outstandingly hard, transparent and colourless diamond. All is a matter of texture.



From the aforementioned requirements and from the possibilities offered by the carbon element, we thought that carbon foams might reach such targets, although very difficult to meet as soon as they are combined. This is indeed one special peculiarity of our work, imposing that all features are obtained together. Now, commercial graphite foams meet most of the requirements but are prohibitive, and their thermal conductivity is uselessly too high. Additionally, their

chemical reactivity is low, hindering any surface treatment in favour of the long-time conservation of PCMs in the liquid phase. In contrast, carbon foams made from hard carbon are cheap, can have their surface easily modified, but are excellent thermal insulators. The idea was thus to prepare materials having the advantages of both kinds of foams, with as less drawbacks as possible. And this is what we did. These formerly non-existing materials, are intermediary between hard and graphite foams.



The unique strategy that we developed is now patented. We could successfully meet the targets in terms of porosity (> 85%), thermal conductivity (> 3 W/m/K), surface functionalization (superhydrophobicity), and cost (< 6 €/kg). More details will be given in a forthcoming newsletter, explaining how the targets could be reached through the combination of several techniques of solid-state chemistry. The present work now consists in characterizing more deeply these new materials for better understanding their behaviour and finding out how they can be further improved.

*Alain CELZARD, Vanessa FIERRO & Prasanta JANA (CNRS-IJL)*

## Storage requirements and cost analysis

Storage requirements for a sugar alcohol storage being included in a solar thermal system were defined in order to give information for guidance of materials design. Additionally a cost estimation based on the chosen system was carried out to clarify if the system can compete against other techniques.

Figure 7 shows an example of the system configuration. The solar collectors deliver heat to the PCM-storage consisting of sugar alcohols. In source as well as in load circuit water is used as heat transfer fluid. A gas boiler serves as auxiliary heater if the heat delivered by the PCM-storage is not sufficient to cover the heating load of the building.

Different system configurations with varying collector size and storage size were analysed by simulation study.

Energy costs were calculated for two system configurations. It includes energy consumption costs (natural gas at 7 c€/kWh) and investments costs considering a 20 years amortization. Investments costs were calculated assuming the following prices: sugar alcohols (1730 €/m<sup>3</sup>), solar collectors (200 €/m<sup>2</sup>), gas boiler (4000 €), tank and tubing (1000 €/m<sup>3</sup>), electrical system (800 €), regulation (600 €) and labour (2000 €).

Results of the cost estimation are presented in Figure 6. It is interesting to note that even at the early stage of the technology; the energy price of the system developed in the project is not so high (~11 to 13 c€/kWh). Indeed, energy prices for other technologies were estimated to be in the range of 9 to 17 c€/kWh (own estimation). Compared to other technologies the system proposed in the project offers high share of renewable energy and is less sensitive to fossil energy price increase. This study shows that depending on the configuration chosen, even if investments costs are high (19 k€ for case1 and 33 k€ for case 2), the technology can be profitable over the years.

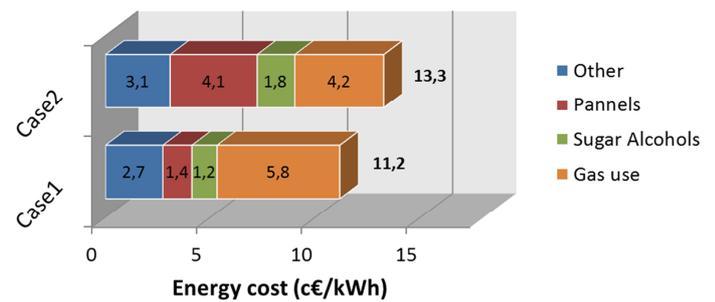


Figure 8: Energy prices for the proposed systems (20 years amortization).

Hannah NEUMANN (Fraunhofer ISE) & Jean-Christophe GABELLE (Solvay)

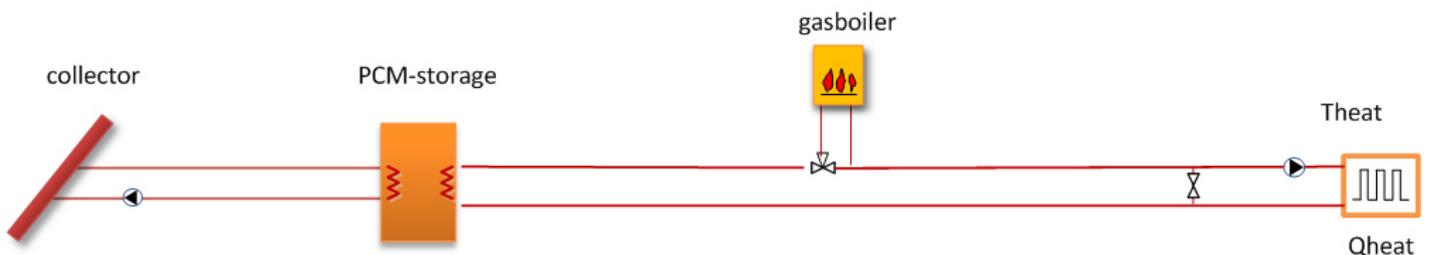


Figure 7: system configuration including PCM-storage consisting of sugar alcohols.



## Publications (selection)

### Ultralow cost reticulated carbon foams from household cleaning pad wastes

*P. Janaa, V. Fierroa, A. Celzarda*

(CARBON, Vol. 62; Year 2013, Elsevier, p. 517-520)

A very simple method is described for preparing ultra lightweight and ultralow cost carbon foams (density 0.04–0.075 g cm<sup>-3</sup> and porosity 98–96%) by impregnation with sucrose of household cleaning pad wastes used as sacrificial templates, followed by pyrolysis in inert atmosphere. Scanning electron microscopy showed that the resultant reticulated vitreous carbon (RVC) foams had a fully open and interconnected porous structure. These materials are more thermally insulating (thermal conductivity 0.042–0.065 W m<sup>-1</sup> K<sup>-1</sup>) than commercial RVC foams having similar compressive strengths (0.11–0.23 MPa) and similar densities.

<http://www.sciencedirect.com/science/article/pii/S0008622313005290>

### First-principle prediction of anisotropic crystal/melt interfacial free energy of sugar alcohols

*Zhang, H., Nedeia, S. V. and Rindt, C. C. M.*

(conference proceeding of IHTC15, 2014)

Sugar alcohols are extensively used as phase change materials in long term heat storage systems. The thermal performances in such systems are strongly dominated by the nucleation and crystal growth kinetics, which is further linked to the crystal/melt interfacial free energy (also called the solid – liquid surface tension). We report a novel technique using first principle calculations to accurately predict the crystal/melt interfacial free energies of xylitol and  $\delta$ -d-mannitol, as well as their anisotropy. The new technique is based on a wall cleaving method (Davidchack et.al. 2012) which is successfully implemented in monoatomic and rigid polyatomic molecules. This method essentially creates an artificial interface at the melting temperature by gradually putting the crystal and melt phases into contact under the guidance of a cleaving potential. Since the volumetric free energy at the melting temperature for both phases are equal, the free energy difference between the initial and end states only comes only from the interface. We extend this method for the calculation of flexible polyatomic molecules. We base our simulations on a well-proven generalized AMBER force field, and designed specific cleaving potentials. Lots of efforts were made to achieve reliable equilibrium Boltzmann sampling and reduction of hysteresis, including the selection of transition paths. The samples of transition states are processed based on the Bennett Acceptance

Ratio method. The predicted free energies concede with available experimental estimations and the large anisotropy is found to be responsible for the needle shaped growth of d-mannitol. The results also proved that the wall cleaving method can serve as a good adjunct to the increasingly powerful nanoscale experimental probes for interfacial phenomena.

### Experimental analysis of heterogeneous nucleation undercooled melts by infrared thermography

*M. Duquesne, A. Godin, E. Palomo del Barrio, J. Daranlot*

(Conference Proceedings: 12th International Conference on Quantitative Infrared Thermography; Reference A9167EP)

A new experimental technique for quantitative analysis of heterogeneous nucleation in undercooled melts is proposed in this paper. It is based on the observation by infrared thermography of the thermal behavior on cooling of a large population of small droplets deposited on a substrate. The method allows analyzing the influence of different parameters such as the size of the droplets, the cooling rate, and the wettability of the substrate on the nucleation rate. The fundamentals of the method, the associated experimental set-up, and the mathematics required for data processing are described in the paper. The results of the nucleation analyses carried out using erythritol deposited on different substrates are also presented and discussed.

### High Pressure Solutions-Turning the energy levels of inorganic-organic interfaces may open new applications in life sciences and energy storage

*R.R. Piticescu*

(Science & Technology, vol. 7, June 2013, pp. 108)

Advanced materials are one of the key enabling technologies enabling design and fabrication of a large number of systems and components. Many of them require a perfect control over the interfaces and involve activities from modeling to process development and components integration. Nano-enabled surfaces are therefore one of the most interesting value chain for multi-sectorial applications. It is important to note that high pressure chemistry solution ensure the kinetic control toward different morphologies such as nano-rods, nano-whiskers, flower-like structures, etc. An example is the control of graphite-zinc oxide adhesion energy leading to interfaces with design hydrophobicity and thermal properties for energy storage systems. Furthermore other applications may be developed using the new research infrastructure developed by the institute with help from the Structural Funds Project High PTMET.

<http://www.paneuropeannetworks.com/ST7/#108/z>



### **Simple and versatile one-step synthesis of highly interconnected graphitized macroporous carbon foam**

*M. Karthik, V. Roddatis, S. Doppiu*

(Conference Proceedings: Annual International World Conference on Carbon (Carbon 2013))

The highly interconnected graphitized macroporous carbon foams were successfully synthesised by one-step low temperature process using resorcinol and formaldehyde as carbon precursors and metal nitrate as a graphitization catalyst. The commercially available polyurethane foam was used as a sacrificial polymer template. The obtained three-dimensional (3D) highly interconnected graphitized carbon foams were extensively characterized by using powder X-ray diffraction (XRD), nitrogen adsorption-desorption measurement, transmission electron microscopy (TEM), scanning electron microscopy (SEM), raman spectroscopy and thermogravimetric analysis (TGA). XRD and TEM results clearly demonstrated the presence of a well-defined graphitic framework. The surface morphology of the macroporous carbon foam was studied by using SEM and the results clearly showed the presence of a highly interconnected porous network throughout the structure. The thermal stability of the carbon foam was also examined by TGA. The effects of various parameters such as precursor ratio, curing time & temperature, concentration of the metal nitrate and graphitization temperature were investigated. The physical properties of the carbon foam as such density, pore size and total porosity were also studied and the salient results are discussed.

[http://www.carbon2013.org/site/wp-content/uploads/Carbon2013\\_accepted\\_abstracts.html](http://www.carbon2013.org/site/wp-content/uploads/Carbon2013_accepted_abstracts.html)

### **Analysis of crystal growth kinetics in undercooled melts by infrared thermography**

*A. Godin, M. Duquesne, E. Palomo del Barrio*

(12th International Conference on Quantitative Infrared Thermography Reference: A9201AG)

A new experimental approach based on infrared thermography is proposed in this paper for studying crystal growth kinetics in undercooled melts. The crystallization of a thin sample of an undercooled melt at constant bulk temperature is induced by a small crystal seed and the growth of the solid phase is observed using infrared camera. The recorded thermal images allow determining the position of crystallization front at any time, the velocity of advancement of the front at any point and time, and the temperature at the points of the interface at any time. Contrary to experimental techniques based on optical microscopy or video cameras, infrared thermography provides detailed analysis of the interface temperature which is essential when discussing the temperature dependence of experimentally determined growth rates.

The appropriateness of infrared thermography for crystal growth kinetics analysis is illustrated through the experimental analysis of erythritol crystallization.

### **Fast estimation of the phase diagram of a binary system using infrared thermography**

*R. Cadoret, E. Palomo del Barrio, J. Daranlot*

(Conference Proceedings: 12th International Conference on Quantitative Infrared Thermography; Reference A9167EP)

A method based on infrared thermography for fast estimation of the phase diagram of solid-liquid binary systems has been developed. The method involves heating simultaneously a set of small crystals of a system A/B, with compositions ranging from 0%B/(A+B) to 100%B/(A+B), and to observe their thermal behavior with an infrared camera. Recorded data are then analyzed by Singular Value Decomposition techniques to identify liquidus and solidus lines. Different binary systems have been investigated and the results achieved have been compared with the phase diagrams determined by standard DSC testing, as well as with the phase diagrams predicted by thermodynamic modelling.



### **Workshops and conferences (where SAM.SSA was presented)**

26-27.04.2012 **2012 Annual Conference of Euroheat & Power and RHC-Platform, Copenhagen** (Copenhagen, Denmark), Zafer Ure (PCM Products)

16-18.05.2012 **Innstock 2012 12th International conference on Energy Storage** (Lleida, Spain), Silvia Nedeá, Camilo Rindt (TU/e)

12-13.09.2012 **FOAMS 2012 -10<sup>th</sup> International Conference on Foam Materials & Technology** (Barcelona, Spain), Alain Celzard (CNRS-IJL)

29.11.2012 **NANO4DESIGN Workshop** (London, UK), Zafer Ure (PPL)

21-13.04.2013 **4th European Conference on Renewable Heating & Cooling** (Dublin, Ireland), Elena Palomo (CNRS-I2M)

19-21.06.2013 **2nd International Conference on Sustainable Energy** (Dublin, Ireland), Roxana M. Piticescu, Maria D. Romero (IMNR, AIDICO)

14-19.07.2013 **Carbon 2013: Annual International World Conference on Carbon** (Rio de Janeiro, Brazil), Mani Karthik (CICe)

27.08.2013 **BIOFOAMS 2013 - 4th International Conference on Biofoams** (Toronto, Canada), Alain Celzard (CNRS-IJL)

02-05.09.2013 **International Conference on Diamond and Carbon Materials** (Riva del Garda, Italy), Radu Piticescu, Elena Palomo del Barrio, Alain Celzard, (IMNR,CNRS)

23-25.09.2013 **International Conference on Solar Heating and Cooling for Buildings and Industry** (Freiburg, Germany), Hannah Neumann (Fraunhofer ISE)

23-25.09.2013 **International Conference on Solar Heating and Cooling for Buildings and Industry** (Freiburg, Germany), Hannah Neumann (Fraunhofer ISE)

28-30.05.2014 **Eurotherm Seminar n°99: Advances in Thermal Energy Storage** (Lleida, Spain), Silvia Nedeá, Camilo Rindt (TU/e)

7-11.07.2014 **12th International Conference on Quantitative Infrared Thermography** (Bordeaux, France), Marie Duquesne, Alexandre Godin, Elena Palomo (CNRS-I2M)



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TUE - Technische Universiteit Eindhoven, The Netherlands



CICe - Centro de Investigación Cooperativa de Energías Alternativas Fundación, Spain

**Upcoming Workshop**

February/March 2015 - **Workshop & On-site demonstration Workshop at CICE in Vitoria, Basque Country**

For further information please contact:

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### The Consortium



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For further information on the project, please visit the project website:

[www.samssa.eu](http://www.samssa.eu)

### Disclaimer

The SAM.SSA project (Project Identifier: 296006) is funded by the European Commission under the Seventh Framework Programme.

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