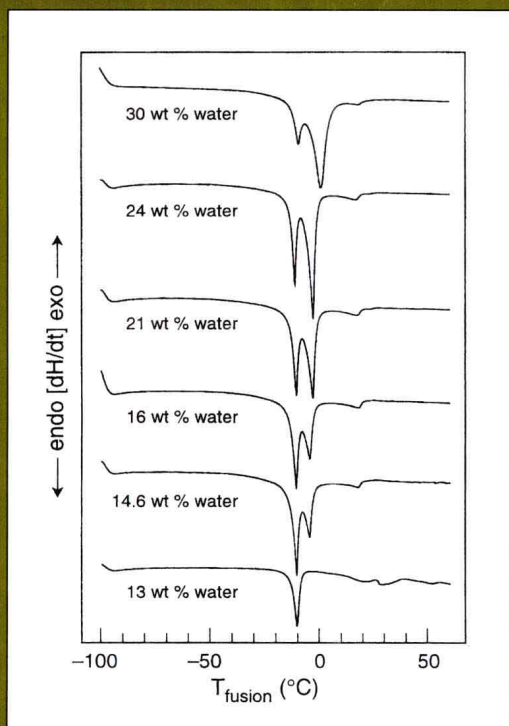


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volume **93**

# THERMAL BEHAVIOR OF DISPERSED SYSTEMS



edited by  
Nissim Garti

# **THERMAL BEHAVIOR OF DISPERSED SYSTEMS**

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edited by

Nissim Garti

*The Hebrew University of Jerusalem  
Jerusalem, Israel*



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# Preface

Many important everyday materials are known to be colloidal heterogeneous systems. Milk, margarine, ice cream, mayonnaise, cosmetic creams, hand lotions, blood, ink, paint, and other substances are heterogeneous systems that can flow or become solid, and contain structural entities with at least one linear dimension in the size range of several nanometers to tens of microns.

Colloidal systems consist of a dispersed phase of particles, droplets, and bubbles in a second continuous phase called the dispersion medium.

Dispersed systems are said to be stable if, over a certain period of time, there is little detectable aggregation or settling of particles. In many colloidal systems, a stable thermodynamic state is reached only when all particles or droplets have become united in a single homogeneous lump of dispersed phase and, therefore, any apparent stability must be regarded as purely kinetic phenomenon.

Dispersed systems are stabilized by a third component known to have amphiphilic properties and surface activity. The amphiphiles migrate to the interface and modify it to reduce the interfacial free energy and to minimize interactions between particles and droplets.

It is obvious that any temperature change will affect the mobility of the amphiphiles from the continuous phase to the interface, and vice versa, and will affect the thermodynamic and geometric parameters of the interface. Therefore, the thermal behavior of dispersed systems is an essential parameter in studying structural and thermodynamic aspects.

For generations, attempts have been made to heat and cool foams, emulsions, dispersions, and heterogeneous colloidal systems, and to learn about the stability of the systems through their thermal behavior. Differential scanning calorimetry (DSC) and differential thermo gravimetry (DTG) are classical instruments that

through complex heating-cooling protocols provide important information on the behavior of the components of the dispersions.

Dispersions (mostly emulsions and microemulsions) are used as microreactors or nanoreactors for important organic and enzymatic processes, and serve as reservoirs for the solubilization of materials. The behavior of the solubilized matter is also dramatically affected by thermal fluctuations. Hydration or solvation, as well as other interactions of cosolvents, are also studied through thermal treatment. It is therefore important to bring to the reader's attention the options and the scope, as well as the limitations, of the thermal behavior of dispersed systems.

This book calls attention to some of the recent studies that have been carried out on heterogeneous colloidal (dispersed) systems.

Chapter 1, by Turco Liveri (Italy), reviews calorimetric investigations on reversed micelles, in which the apolar molecules interact by dispersion forces that are always attracted independently of their relative orientation. The author describes the tendency of the apolar medium in reverse micelles to form a long-range, ordered molecular arrangement in condensed phases. The amphiphilic molecules, characterized by the coexistence of spatially separated polar and apolar moieties, work together to drive the intermolecular aggregation, giving rise to dimensionally limited supramolecular aggregates.

From a thermodynamic point of view, self-aggregation of amphiphilic molecules in apolar solvents involves a favorable enthalpic term due to intermolecular bonding counteracted by an unfavorable entropic term as a result of partial loss of molecular translational and rotational degrees of freedom.

V. Turco Liveri discusses structural aspects, the state of water and other solutes in reversed micelles, intermicellar interactions and percolation phenomena, solubilization of nonionic solutes, and the reversed micelles as nanoreactors.

The second chapter, by D. Vollmer (Germany), brings a quantitative comparison of experimental data and theoretical predictions on thermodynamic and kinetic properties of microemulsions based on nonionic surfactants. Phase transitions between a lamellar and a droplet-phase microemulsion are discussed. The work is based on evaluation of the latent heat and the specific heat accompanying the transitions. The author focuses on the kinetics of phase separation when inducing emulsification failure by constant heating. The chapter is a comprehensive, detailed study of all the aspects related to the phase separation phenomenon in microemulsions.

In Chapter 3, Ezrahi et al. (Israel) discuss the use of subzero temperature behavior of water in microemulsions as an analytical tool to enable better understanding of the interfacial behavior of the surfactant. Microemulsions are cooled to subzero temperatures and the water in the internal reservoir freezes. In the heating cycle the thawing of the water is measured. The authors critically discuss the problems related to the use of this technique and the advantages derived from it.

Chapter 4, by Schulz et al. (Argentina and Mexico), describes the use of DSC techniques for studying binary and multicomponent systems containing surfactants. The authors explain how DSC helps to elucidate such properties as type of transition, phase boundaries, enthalpies of phase transition, and heat capacity of systems in heterogeneous states.

Fouconnier et al. (France) introduce us in Chapter 5 to dispersed systems that are not stable thermodynamically, such as emulsions and double emulsions, and teach us how to carry out DSC measurements properly in order to obtain valuable information on the stability of the emulsions.

Various physical and chemical phenomena that occur during cooling and heating have been pointed out. They may be associated with either the dispersed, the continuous, or the interfacial phase. A tentative description of some of these events is presented, and a correlation was made with the resulting properties of the emulsions themselves. Simple water-in-oil (W/O) or oil-in-water (O/W) emulsions, mixed emulsions, and multiple emulsions can be found during the fabrication process. Their storage and their use are considered.

Senatra (Italy), who was a pioneer in the use of DSC as a technique to study interfacial phenomena, offers in Chapter 6 some interesting physical parameters and the essence of these observations.

Chapter 7 is an interesting review by Kodama and Aoki (Japan) on the behavior of water in phospholipid bilayer systems. The authors distinguish between nonfreezable interlamellar water and freezable intralamellar and bulk water, and estimate the number of molecules of water in each category. They also examine the relationship between lipid phase transitions and ice-melting behavior in lipid-water systems. The behavior of water is also discussed in the gel phase of systems such as DPPC, DMPE, and DPPG.

Part II concentrates on solid-liquid interfaces.

Chapter 9, by Király (Hungary), attempts to clarify the adsorption of surfactants at solid/solution interfaces by calorimetric methods. The author addresses questions related to the composition and structure of the adsorption layer, the mechanism of the adsorption, the kinetics, the thermodynamics driving forces, the nature of the solid surface and of the surfactant (ionic, nonionic, HLB, CMC), experimental conditions, etc. He describes the calorimetric methods used, to elucidate the description of thermodynamic properties of surfactants at the boundary of solid-liquid interfaces. Isotherm power-compensation calorimetry is an essential method for such measurements. Isoperibolic heat-flux calorimetry is described for the evaluation of adsorption kinetics, DSC is used for the evaluation of enthalpy measurements, and immersion microcalorimetry is recommended for the detection of enthalpic interaction between a bare surface and a solution. Batch sorption, titration sorption, and flow sorption microcalorimetry are also discussed.

Chapter 10, by Dékány (Hungary), describes the microcalorimetric control of liquid sorption on hydrophilic/hydrophobic surfaces in nonaqueous dispersions.

The dispersed systems are mostly silicates. The author discusses interparticle interactions as a tool for evaluating the stability of dispersions. Parameters such as heat of immersion at solid-liquid interfaces and adsorption capacity are determined, and the mathematical treatment for determining the enthalpy isotherms is described. The heat of wetting in amorphous silica dispersion and on zeolites is discussed.

Füredi-Milhofer (Israel), in Chapter 11, provides a broad overview of the role of thermal analysis techniques in basic and applied studies of the formation and transformation of crystalline dispersions. Crystalline dispersions are formed by a succession of some of the following precipitation processes: nucleation, crystal growth, flocculation, Ostwald ripening, and/or phase transformation. After a brief elaboration of the theories underlying these processes, a review is given of experimental studies on the formation and transformation of ionic precipitates from bulk electrolyte solutions.

At relatively high supersaturations, compounds that include hydrophilic cations (such as Ca, Al, Fe, etc.) are likely to form highly hydrated amorphous precipitates via homogeneous nucleation and subsequent flocculation. A number of important crystalline compounds, such as hydroxyapatite or zeolites, are formed by phase transformation via amorphous and/or gel-like precursor phases. Thermal analysis techniques yield information on the amount of incorporated water, and mechanism and strength of bonding, and pore sizes of such amorphous and poorly crystalline materials. In some cases they have been successfully used to detect the initiation of phase transformation, such as the formation of ordered subunits of a quasicrystalline zeolite phase within amorphous aluminosilicate precursors.

At low and medium supersaturations, hydrophilic cations form different crystal hydrates by heterogeneous nucleation and subsequent crystal growth and phase transformation. Dehydration curves give information on the modes of water incorporation resulting from different modes of crystallization. A useful application of thermal analysis is the analytical approach: by determining the mass loss due to dehydration, it was possible to quantitatively determine the proportion of different calcium oxalate hydrates in mixtures, which have been qualitatively analyzed by other techniques (X-ray powder diffraction, IR spectroscopy, etc.). The method yielded excellent results in studies of the kinetics of phase transformation and has been successfully used to demonstrate the potential of surfactant micelles to control the nature of the crystallizing phase.

Chapter 11 also deals with crystallization in O/W emulsions and W/O micro-emulsions.

Filipović-Vinceković and Tomašić (Croatia) have contributed Chapter 12, "Solid-State Transitions of Surfactant Crystals," which discusses the effect of surfactants on the crystallization of materials in aqueous and nonaqueous solutions. The chapter describes the crystalline structure of surfactant and its thermal

behavior, and the effects related to its crystallization. Single- and double-chain surfactants are reviewed, and the differences in their thermal behavior are elucidated.

Chapter 13 is the only chapter that discusses thermal behavior of real complex systems. Raemy et al. (Switzerland and Israel) in "Thermal Behavior of Foods and Food Constituents," reveal the complexity of studying such systems using different thermal calorimetric techniques.

In conclusion, this book presents only a very small fraction of the options, scope, and limitations of using thermal behavior of dispersed systems as an analytical and physical tool for the evaluation of phenomena occurring at the interface between the dispersed phase and the dispersion phase.

It must be noted that much more work is required to enable better understanding of complex systems and real dispersions. These systems will be discussed in a separate book that will be devoted to complex dispersion systems that have been converted into commercial products.

*Nissim Garti*

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