

MICROEMULSION SYSTEMS

Edited by

HENRI L. ROSANO

City College of the City University of New York
New York, New York

MARC CLAUSSE

Université de Technologie de Compiègne
Compiègne, France

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Preface

The 59th Colloid and Surface Science Symposium and the 5th International Conference on Surface and Colloid Science were held on the campus of the Clarkson College of Technology in Potsdam, New York, June 24-28, 1985. The meeting was organized by the Clarkson's Institute of Colloid and Surface Science. Among the eighteen special topics discussed was one entitled **Microemulsion Systems**; this monograph reports most of the papers presented at that symposium, where the discussions ranged from fundamental research to development efforts. The latest developments in the fields of the mechanisms of formation and stability of microemulsions, characterization, relation between phase behavior and structure, dynamic processes, interfacial properties, theoretical modeling, gas solubility, waterless microemulsions, diffusion phenomena between phases, and many more studies, are reported in this collection of twenty-five chapters on microemulsion systems.

We would like to express our appreciation to all those who participated in preparing this volume, especially the contributors. We would also like to thank Evelyn Cropper for her patience while we were gathering and editing the twenty-five chapters by scientists from the four corners of the world; without her collaboration these proceedings could not have been published. We feel that this universal interest in microemulsion systems, initially in the field of tertiary oil recovery, is starting to find more and more industrial applications, such as in pharmaceuticals, chemical manufacturing, detergents, personal care products, pesticides, herbicides, food products, and in many other important fields.

Henri L. Rosano
Marc Clausse

Contributors

C. A. ANGELL Purdue University, West Lafayette, Indiana

LOÏC AUVRAY* Laboratoire de Physique de la Matière Condensée,
Collège de France, Paris, France

R. C. BAKER† Jealotts Hill Research Station, Bracknell, Berk-
shire, United Kingdom

ALAIN BERTHOD Laboratoire de Chimie Analytique 3, Université
Claude Bernard, Villeurbanne, Lyon, France

CARLOS BORZI‡ Cornell University, Ithaca, New York

AMY L. R. BUG Exxon Research and Engineering, Corporate
Research Science Laboratories, Annandale, New Jersey

MARTINE BUZIER Université de Nancy, Nancy, France

D. CANET Université de Nancy, Nancy, France

E. CAPONETTI** University of Tennessee, Knoxville, Tennessee

Present affiliations:

*Laboratoire Léon Brillouin CEA-CEN, Saclay, Gif sur Yvette,
France

†Coopers Animal Health Ltd., Berkhamsted, Herts, United Kingdom

‡Instituto de Física de Líquidos y Sistemas Biológicos, La Plata,
Argentina

**Postdoctoral Fellow at the University of Tennessee, 1984. Perma-
nent affiliation: Instituto di Chimica Fisica, University of Palermo,
Palermo, Italy

J. CARNALI Lund University, Lund, Sweden

JOHN L. CAVALLO City College of The City University of New York,
New York

A. M. CAZABAT Laboratoire de Physique, ENS, Paris, France

D. Y. CHAO Industrial Technology Research Institute, Hsinchu,
Taiwan, Republic of China

D. CHATENAY Laboratoire de Physique, ENS, Paris, France

S. I. CHEN Industrial Technology Research Institute, Hsinchu,
Taiwan, Republic of China

MARC CLAUSSE Université de Technologie de Compiègne, France

J. P. COTTON Laboratoire Léon Brillouin, CEA-CEN, Saclay, Gif
sur Yvette, France

P. DELORD University of Montpellier 2, Montpellier, France

JEAN-MARC DI MEGLIO Laboratoire de Physique de la Matière
Condensée, Collège de France, Paris, France

M. DUPEYRAT Université Pierre et Marie Curie, Paris, France

GASTON DUPONT Université de Nancy, Nancy, France

J. L. DUSSOSSOY* University of Montpellier 2, Montpellier, France

MAYA DVOLAITZKY Laboratoire de Physique de la Matière Con-
densée, Collège de France, Paris, France

STIG E. FRIBERG University of Missouri at Rolla, Rolla, Missouri

GARY S. GRETT Exxon Research and Engineering, Corporate
Research Science Laboratories, Annandale, New Jersey

P. GUERING Laboratoire de Physique, ENS, Paris, France

J. HEIL[†] Université de Technologie de Compiègne, France

Present affiliation:

*Centre d'Etudes Nucléaires de Cadarache, Saint Paul-Les-Durance,
France

†PUM S.A., Bonneuil sur Marne, France

- U. HENRIKSSON Royal Institute of Technology, Stockholm, Sweden
- E. A. HILDEBRAND Monash University, Clayton, Victoria, Australia
- HU MEI-LONG Xinjiang Institute of Chemistry, Academy of Science of China, Urumuqi, Xinjiang, People's Republic of China
- M. KAHLWEIT Max-Planck-Institut fuer biophysikalische Chemie, Geottingen, Federal Republic of Germany
- M. KAMIONER Université Pierre et Marie Curie, Paris, France
- D. LANGEVIN Laboratoire de Physique, ENS, Paris, France
- F. C. LARCHE University of Montpellier 2, Montpellier, France
- A. LATTES Université Paul Sabatier, Toulouse, France
- LI ZHI-PING Xinjiang Institute of Chemistry, Academy of Science of China, Urumuqi, Xinjiang, People's Republic of China
- YUH-CHIRN LIANG University of Missouri at Rolla, Rolla, Missouri
- BJÖRN LINDMAN Chemical Center, Lund University, Lund, Sweden
- REINHARD LIPOWSKY* Cornell University, Ithaca, New York
- GEORGE B. LYONS City College of The City University of New York, New York
- D. R. MACFARLANE Monash University, Clayton, Victoria, Australia
- L. J. MAGID University of Tennessee, Knoxville, Tennessee
- I. R. MCKINNON Monash University, Clayton, Victoria, Australia
- J. MEUNIER Laboratoire de Physique ENS, Paris, France
- H. NERY Université de Nancy, Nancy, France

Present affiliations:

*Sektion Physik der Universität München, FRG

L. NICOLAS-MORGANTINI* Université de Technologie de Compiègne, France, and Université de Pau et des Pays de L'Adour, Pau, France

RAYMOND OBER Laboratoire de Physique de la Matière Condensée, Collège de France, Paris, France

JEAN-CLAUDE RAVEY Université de Nancy, Nancy, France

I. RICO Université Paul Sabatier, Toulouse, France

HENRI L. ROSANO City College of The City University of New York, New York

J. ROUVIERE Université des Sciences et Techniques du Languedoc, Montpellier, France

S. A. SAFRAN Exxon Research and Engineering, Corporate Research Science Laboratories, Annandale, New Jersey

Y. G. SHEU Industrial Technology Research Institute, Hsinchu, Taiwan, Republic of China

DUANE H. SMITH† Phillips Petroleum Company, Bartlesville, Oklahoma

O. SÖDERMAN Lund University, Lund, Sweden

K. SOHOUNHLOUE Université des Sciences et Techniques du Languedoc, Montpellier, France

PETER STILBS Uppsala University, Uppsala, Sweden

R. STREY Max-Planck-Institute fuer biophysikalische Chemie, Goettingen, Federal Republic of Germany

TH. F. TADROS Jealotts Hill Research Station, Bracknell, Berkshire, United Kingdom

CHRISTIANE TAUPIN Laboratoire de Physique de la Matière Condensée, Collège de France, Paris, France

Present affiliations

*L'Oreal, Aulnay-sous-Bois, France

†Morgantown Energy Technology Center, Morgantown, West Virginia

- D. TOURAUD Université de Technologie de Compiègne, France
- W. URBACH Laboratoire de Physique, ENS, Paris, France
- H. WALDERHAUG Lund University, Lund, Sweden
- T. WÄRNHEIM Royal Institute of Technology, Stockholm, Sweden
- ITZHAK WEBMAN Rutgers University, New Brunswick, New Jersey
- BENJAMIN WIDOM Cornell University, Ithaca, New York
- J. S. ZHOU Université Pierre et Marie Curie, Paris, France
- A. ZRADBA† Université de Technologie de Compiègne, France, and
Université de Pau et des Pays de l'Adour, Pau, France

Present affiliation:

†Ecole Normale Supérieure de Casablanca, Casablanca, Morocco.

Introduction

In reporting the proceedings concerned with "Microemulsion Systems" at the 59th Colloid and Surface Science Symposium and the 5th International Conference on Surface and Colloid Science (Potsdam, New York, June 24-28, 1985), this book also reflects the difficulty experienced by the participants in reaching a common ground on the definition of the term "microemulsions." Over 20 definitions were proposed, but no clear-cut definition emerged. The only common definition described these clear (or sometimes lactescent) systems as containing one polar liquid (generally water or saline), a hydrophobic liquid (the "oil phase"), and a selected (one or several) surface active agent.

The word "microemulsion" was originally proposed by Jack H. Schulman and co-workers in 1959, although the first paper on the topic dates from 1943. They prepared a quaternary solution of water, benzene, hexanol, and K oleate, which was stable, homogeneous, and slightly opalescent. These systems became clear as soon as a short chain alcohol was added. In the years between 1943 and 1965, Schulman and co-workers described how to prepare these transparent systems. Basically, a coarse (or macro) emulsion was prepared, and the system was then titrated to clarity by adding a cosurfactant (second surface active substance). When the combination of the four components was right, the system cleared spontaneously. Most of the work reported by Schulman dealt with four-component systems: hydrocarbons (aliphatic or aromatic); ionic surfactants; the cosurfactant, generally 4- to 8-carbon chain aliphatic alcohol; and an aqueous phase. Schulman had previously published extensively in the field of monolayers, and applied what he had learned in that field to explain the formation of a microemulsion. He proposed that the surfactant and the cosurfactant, when properly selected, form a mixed film at the oil/water interface, resulting in an

interfacial pressure exceeding the initial positive interfacial tension. The concept of a *negative interfacial tension* was suggested by Schulman. Rosano and co-workers showed that transitory zero (or very low) interfacial tensions obtained during the redistribution of the cosurfactant often play a major role in the spontaneous formation of these systems. This conclusion was based on the differences in the observed results when the order of mixing the components was changed.

To summarize, the basic observation made by Schulman and co-workers was this: when a cosurfactant is titrated into a coarse emulsion (composed of a mixture of water/surfactant in sufficient quantity to obtain microdroplets/oil), the result may be a system which is low in viscosity, transparent, isotropic, and very stable. The transition from opaque emulsion to transparent solution is spontaneous and well defined. It was found that these systems are made of spherical microdroplets with a diameter between 60 to 800 angstroms. It was only in 1959 that Schulman proposed to call these systems *microemulsions*: previously, he used terms such as *transparent water and oil dispersion*, *oleopathic hydromicelles*, or *hydrophathic oleomicelles*.

In 1943, T. P. Hoar and Jack H. Schulman describing an oil/water system wrote: "The disperse phase consists of submicroscopic micelles having a core of soap-in-water solution and a surface monolayer of soap ionic-pairs, interspersed with nonionized amphipathic molecules, with the hydrocarbon portions orientated outwards. The high soap/water ratio ensures that the soap is undissociated. The presence of the water/oil interface containing orientated nonionized amphipathic molecules allows undissociated soap ion-pairs also to orientate there, as an alternative to association in curd filers. The nonionized amphipathic molecules separate the soap ion-pairs sufficiently to prevent the repulsion between them that would otherwise occur, and indeed convert the repulsion in the monolayers into an attraction by forming complexes with the soap."

In order to explain the "spontaneous" formation of these microdroplets and their stability, the following explanation was advanced by Schulman: recall that π -surface pressures can be obtained spontaneously by the monolayer penetration of an alkyl alcohol or cholesterol monolayer with ionic surface active agents such as salts of alkylsulfate and alkylamines and substituted amines injected into the underlying solution (at constant area of the insoluble monolayer). The value of π -surface pressures at the air/water interface can reach values of more than 60 dynes/cm (although the collapse π -pressure for single component monolayers on their own are below 35 dynes/cm). If the surface pressure is held constant, or below the 60 dynes/cm but above the 35 dynes/cm, immediate expansion of the interfacial area takes place as the molecules of ionic surfactant penetrate the

monolayer at the air/water interface. The analogous penetration of the mixed film by oil molecules at the oil/water interface increasing the surface area is the basis for the formation of microemulsions. However, the penetrating pressure in this case must be greater than the oil/water surface tension $\gamma_{o/w}$, which is always less than $\gamma_{a/w}$ tension of 72 dynes/cm, which is approximately 50 dynes/cm for hydrocarbon and 35 dynes/cm for aromatic hydrocarbon compounds.

Therefore, it is progressively easier to obtain negative interfacial tensions for these systems, provided that the oil molecules can penetrate the interfacial film. Rosano et al. measured the change in the water-oil interfacial tension while alcohol was injected into one of the phases. It was found that the interfacial tension may be temporarily lowered to zero while the alcohol diffused through the interface and redistributed itself between the water and oil phases. It would therefore be possible for a dispersion to occur spontaneously (while $\gamma_i = 0$). It is then possible for the interfacial tension of a system to drop to zero for a certain period of time due to redistribution of amphiphatic molecules while the equilibrium γ_i remains positive. A sufficiently low positive value of γ_i is always better for emulsion (macro- or microemulsion) formation; nevertheless, below a certain positive small equilibrium value of γ_i phase separation, sol or gel formation will be produced but not emulsification.

Finally, stability, in turn, is not dependent on the value of the interfacial tension, but on the structure of the interfacial film surrounding the individual droplet. Therefore, low interfacial tension appears to be required for stability to occur at any degree. This finding is similar to observations of Shinoda and Saito, who first suggested forming their emulsification process using nonionic surfactants at the phase inversion temperature (PIT) where minimal interfacial tension is reached and then lowering it for further stability. At the PIT, γ_i is minimum favor to o/w interfacial area formation. In Rosano's et al. work on microemulsification, the same concept seems to apply: low interfacial tension is initially produced to lower the initial work requirement but through transfer and redistribution at the interface. Eventually the interfacial tension increases while the o/w interface curls and droplets are formed, allowing a barrier to form at the interface and preventing coalescence. This observation was also described mathematically by Defay and Sanfeld.

In order to obtain spontaneous microemulsion formation, the selection of the primary surfactant and cosurfactant and the right procedure capable of favoring redistribution between phases is important. Rosano et al. have stressed that the spontaneous formation of these dispersed swollen micelles is not dependent on simple thermodynamic stability, but rather—at least in part—on the occurrence of kinetic conditions favorable to the dispersion of the dispersed phase into the o/w system.

Essentially, the pioneering work of Schulman and later, his students, was to correlate chemical structure of the various components in the microemulsion system to their spontaneous formation. As was mentioned above, the use of the word "microemulsion" has been disputed and other labels have been preferred: transparent emulsion micellar emulsion, micellar solution, swollen micellar solution. Those who prefer to use micellar solution or swollen micellar solution consider the fact that systems of the same kind are made of one surfactant with double chain (sodium dioctylsulfosuccinate-Aerosol OT). It is possible to obtain a microemulsion-type system by either incorporating a hydrocarbon into the aqueous micellar solution or putting water into an organic solution containing the surfactant (*inverse micelles*). For these authors, microemulsion formation must be considered a micellization or micellar solubilization process of a macroemulsion when a selected cosurfactant is added. Winsor has described and studied monophasic media composed of water/organic salt/additives/hydrocarbon. He has described these systems as "solubilized." On the other hand, Friberg et al. and several others have, from the analysis of phase diagrams, underscored the fact that systems studied by Schulman (water/surfactant/cosurfactant/oil) evolve from ternary monophasic solutions (water/ionic surfactant/alcohol).

In the final analysis, there is no real opposition to the concept of microemulsion versus swollen micellar solution. It seems that the word "microemulsion" is used more often to describe any multicomponent fluids made of water (or a saline solution), a hydrophobic liquid (oil), and one or several surfactants, these systems being stable, with a low viscosity, transparent, and isotropic. On the one hand, from an application point of view, "spontaneous" formation of these systems (high energy drinks, O₂-carrier, imaging solutions, inks, household and personal care products, and so on) remains an important characteristic. On the other hand, from a purely scientific point of view a so-called microemulsion fluid may exist as one phase of mutual solubilization of all the components. It may also constitute one phase of a polyphasic system. The other phases can either be water or organic phases in excess (Winsor systems), or mesophases (lyotropic liquid crystals). With certain polyphasic systems, two such phases may be of the microemulsion type. It should be noted that the word "microemulsion" has been used to describe media with no strong amphiphile (waterless microemulsions) and also media where formamide or glycerol were used instead of water. The lack of discrimination in the use of a precise definition of the word "microemulsion" may be unacceptable to some but it has not prevented people from investigating these systems. We must be grateful to the late Jack H. Schulman for offering such a catchy word to his colleagues. Maybe the ambiguity of this word, which is easy to

remember, has helped to awaken the scientific and industrial world to consider the fundamental and practical applications of these systems. To be sure, the word has helped to some degree to stimulate research on these systems, and it is hoped that this book will encourage more.

On June 20, 1967, the announcement of the tragic death of Jack H. Schulman, Professor at Columbia University, came as a great shock to his many friends and colleagues. We in the field of colloid and interface science hold him in high esteem for his impressive record of important achievements. It is fitting for us to dedicate this book to his memory.

Henri L. Rosano

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