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2006 PUBLICATIONS AND COMMUNICATIONS BY LAB. FIRP ASSOCIATES

TOLOSA L., RODRIGUEZ-MALAVAR A. J., GONZALEZ A. M., ROJAS O. J.

Effect of Fenton's reagent on O/W emulsions stabilized by black liquor

J. Colloid Interface Science, **294**, 182-186 (2006)

ABSTRACT: The effect of Fenton's reagent (FR) on surface and dispersion properties of black liquor (BL) was investigated. These properties were compared to those of Indulin C (IC), a commercial lignin, and egg lecithin (Le). FR was applied to two different bulk concentrations of H₂O₂ (160 and 320 mM). At pH 8, a minimum in surface tension for the Fenton treated BL was observed. The dispersant ability of BL, IC and Le in oil-in-water emulsions was studied by measuring emulsion stability and drop size. It was found that the surface activity and emulsifying capability of BL were higher than those of IC. The emulsifying capability of Le was improved by the FR treatment at low H₂O₂ concentration.

SALAGER J. L.

Emulsion Phase Inversion Phenomena.

In *Emulsions and Emulsion Stability*, J. Sjöblöm Ed., 2nd Edition. Chap. 4, pp 185-226. Taylor and Francis, London (2006)

ABSTRACT: Emulsion inversion depends on many different variables which are classified as: formulation, composition, and protocol variables such as the rate of formulation or composition change. The standard inversion is interpreted on a bi or tridimensional map and is related to the phase behavior at equilibrium. Dynamic inversion processes, i.e., transitional and catastrophic inversions are reviewed in details according to the recent literature on phenomenological issues and applications to emulsion formation. The state of the art concerning the understanding of various mechanisms involved in the different types of inversion is discussed.

BRAVO B., MARQUEZ N., YSAMBERTT F., CHAVEZ G., CACERES A., BAUZA R., GRACIAA A., LACHAISE J., SALAGER J. L.

Phase behavior of fatty acid/oil/water systems: I. Effect of the acid alkyl chain length.

J. Surfactants & Detergents **9**, 141-146 (2006)

ABSTRACT: The phase behavior of different fatty acids in pseudo-ternary mixtures (water/fatty acid + alcohol/oil) was studied. The Winsor I-III-II transitions of ionized compounds were attained by varying the FAs composition through the formulation scan. The middle phase volume, the pH of the optimum system and the partition coefficient

increase with the chain length of fatty acids. On the other hand, the acid partition coefficient allows to calculate the Gibbs free energy of transfer of a CH₂ group from oil to water, which is found to be + 0.87 J.mol⁻¹

GRACIAA A., ANDEREZ J. M., BRACHO C., LACHAISE J., SALAGER J. L., TOLOSA L., YSAMBERTT F.

The Selective Partitioning of the Oligomers of Polyethoxylated Surfactant Mixtures between Interface and Oil and Water bulk Phases.

Advances in Colloid and Interface Science **123-126**, 63-73 (2006)

ABSTRACT: Because their affinities for the oil and water phases vary considerably with the number of ethylene oxide units in their hydrophilic group, the ethoxylated nonionic species occurring in commercial products tend to behave in a non-collective way, with the low ethoxylation oligomers partitioning mostly in the oil phase. This results in a surfactant mixture at the interface which is more hydrophilic than the one which was introduced in the system in the first place. The pseudophase model is used to study the partitioning in Winsor III type systems, and to estimate the deviation of the interfacial mixture composition from the overall one. New results indicate that the selective partitioning into the oil phase increases when the oil phase becomes aromatic, when the total surfactant concentration decreases and when the water-to-oil ratio decreases.

TOLOSA L. I., FORGIARINI A., MORENO P., SALAGER J. L.

Combined Effects of Formulation and Stirring on Emulsion Drop Size in the Vicinity of Three-Phase Behavior of Surfactant-Oil Water Systems.

Industrial & Engineering Chemistry Research **45**, 3810-3814 (2006)

ABSTRACT: As surfactant-oil-water systems approach Winsor III phase behavior, at the so-called optimum formulation, the interfacial tension decreases, thus allowing the generation of smaller drops upon stirring. On the other hand, the coalescence rate increases, thus favoring the formation of larger drops. The two opposite effects do not alter the drop size in the same way and result in a minimum drop size. Such minimum drop size is found on both sides (for O/W and W/O emulsions) of optimum formulation, whatever the variable used to produce the scan. The location of these minima, which correspond to the most efficient use of the stirring energy to make small droplets, is found to be slightly shifted by a change in stirring energy.

ROJAS O., SONG J., ARGYROPOULOS D. S., BULLON J.

Lignin separation from Kraft black liquors by tangential ultrafiltration.

La Chimica e l'industria, **88**, 88-95 (2006)

ABSTRACT: Lignin from black liquors is a biofuel that is generated during chemical digestion of lignocellulosic materials. There is an increasing interest in separating these lignins into fractions with well-defined properties. Therefore, the objective of this study was to elucidate the characteristics of lignin fractions after ultrafiltration of industrial Kraft black liquor by using tubular ceramic membranes. Overall, this effort represents an embarkation point for a fundamental understanding of membrane ultrafiltration efficiency for the concentration and fractionation of industrial black liquor lignins.

RONDON-GONZALEZ M., SADTLER V., CHOPLIN L., SALAGER J. L.

Emulsion Catastrophic Inversion from Abnormal to Normal Morphology. 5. Effect of the Water-to-Oil Ratio and Surfactant Concentration on the Inversion Produced by Continuous Stirring.

Industrial & Engineering Chemistry Research **45**, 3074-3080 (2006)

ABSTRACT: When the emulsion inversion from abnormal to normal morphology is induced by continuous stirring only, the required duration and the kinetics of the process is found to be related to the water-to-oil ratio of the initial emulsion. The emulsion inversion from O/W to W/O occurs through the formation of a w/O/W multiple emulsion, in which water from the external phase is continuously included as droplets in the dispersed oil phase drops, until a critical dispersed phase value is reached and the inversion takes place. For intermediate initial continuous phase content (0.6 and 0.7 water phase fraction), the water inclusion is found to take place throughout the entire process. In contrast, at high water continuous phase content (0.8 water phase fraction), this inclusion process appears to be delayed and the filling of the oil drops takes place suddenly just before the inversion occurrence. The influence of the surfactant concentration is related with the initial water content too. At $fw = 0.6$, the inversion time is the same whatever the surfactant concentration is, but at $fw 0.7$ and 0.8 , there is an optimal surfactant concentration at which the inversion time is minimal. For systems with a given hydrophilic-lipophilic balance ($HLB = 6$), and 7 wt.% of surfactant concentration, there is, according to conductivity measurements, a unique critical dispersed phase content at which inversion is triggered.

RONDON M., BOURIAT P., LACHAISE J., SALAGER J. L.,

Breaking of water-in-crude oil emulsions. Part 1: Physicochemical phenomenology of demulsifier action.

Energy & Fuels **20**, 1600-1604 (2006)

ABSTRACT: Water-in-oil emulsions formed during oil slicks or petroleum production are known to be stabilized by surfactant molecules that naturally occur in the crude oil, e.g. asphaltenes, which are quite lipophilic in nature. Demulsifier substances combine with naturally occurring surfactants to attain a so-called optimum formulation at which the stability of emulsion is minimum. The attainment of this formulation is related to the hydrophilicity and concentration of the added demulsifier, and a general phenomenology of the demulsification process is outlined.

RONDON-GONZALEZ M., SADTLER V., CHOPLIN L., SALAGER J. L.

Emulsion Inversion from Abnormal to Normal Morphology by Continuous Stirring without Internal Phase Addition. Effect of Surfactant Mixture Fractionation at extreme Water-oil Ratio.

Colloids & Surfaces A. **288**, 151-157 (2006)

ABSTRACT. When the emulsion inversion from abnormal to normal morphology is induced by continuous stirring only, it takes place through the formation of a multiple emulsion in which the external phase is continuously included as droplets in the dispersed phase drops. In the studied case the system exhibits an initial O/W morphology, then a w/O/W multiple one before inverting into a W/O emulsion. The general trend reported in the literature is

that the higher the initial water content, the longer the incorporating process to swell the drops in order to attain the critical dispersed phase volume that triggers inversion. The present study reports, for nonionic systems with a given hydrophilic-lipophilic balance (HLB = 6) and 7 wt.% of surfactant concentration, that this trend is found to be unexpectedly reversed when the initial water content increases beyond 80%. This anomalous result is attributed to an indirect increase in the lipophilicity of the formulation due to the variation of the interfacial surfactant mixture with the water/oil ratio, and to the formation of liquid crystals at high water contents.

SALAGER J. L., ANTON R., AUBRY J. M.

Formulation des émulsions par la méthode HLD (French).

Techniques de l'Ingénieur, Vol. Génie des Procédés, J2-158, 1-15 (2006)

<http://www.techniques-ingenieur.fr/>

ABSTRACT: This chapter which is dedicated to emulsion formulators, first present the strong relationship between equilibrated and emulsified systems. The phase behavior of surfactant-oil-water systems is represented in three types of diagrams: Δ or triangular, γ or fish, χ or formulation-composition. The different types of emulsions are represented in phase diagram maps, and the inversion line characteristics are discussed for the different types of diagrams. Dynamic inversions (transitional and catastrophic) and their characteristics are presented next. Finally the principles of different emulsification processes taking advantage of the phase behavior transitions and emulsion inversions are discussed.

SALAGER J. L.

Microemulsions with surfactant mixtures. Synergy or disaster.

Invited keynote lecture, Symposium on Microemulsions, 97th American Oil Chemist Society Annual Meeting. Saint Louis USA. April 30 – May 3, 2006.

ABSTRACT: Mixing surfactants often produce a more or less collective behavior between mixed species and some intermediate formulation averaging effect takes place. Nevertheless, in some cases uncollective behavior occurs, with each specie going its own way, and results in attractive new features or disastrous outcomes. Various examples are reviewed which deal with different cases and applications: fractionation of polyethoxylated surfactants, anionic-nonionic mixtures to attain insensitivity to temperature, pH sensitive acid/amine type mixtures, charge antagonism in anionic-cationic mixtures, extremely complex case of acid/amine mixtures vs pH.

SALAGER J. L., ANTON R. E.

Microemulsions insensitive to temperature.

Invited lecture, Symposium on Microemulsions, 97th American Oil Chemist Society Annual Meeting. Saint Louis USA. April 30 – May 3, 2006.

ABSTRACT: Ionic surfactants tend to become more hydrophilic as the temperature increases, whereas the opposite occurs with polyethoxylated nonionic. As a consequence the contrary effects might cancel out for a properly selected mixture of ionic and nonionic species. It is shown that there is no need to start a trial and error formulation campaign to find such formulation. Instead, some basic modeling of known formulation effects described through the surfactant-affinity-difference (SAD) quantification concept allows to built up an

impressive forecast on how to design a system insensitive to both temperature and mixture composition, particularly to form bicontinuous microemulsions.

BRICEÑO M. I., CELIS M. T., SALAGER J. L.

Use of Particles as Stabilizers of Emulsions and Foams.

Invited Conference. *Particles 2006 Symposium*. Orlando FL May 13-16, 2006

ABSTRACT. Although the stabilizing effect of colloidal-size nanoparticles on emulsions and foams has been known since the early twentieth century, it has been the last 10 years that industry and academia have acknowledged the vast potential of using particles as surfactants or co-surfactants in the preparation and stabilization of dispersed systems. In fact, particles are the main stabilizers of naturally occurring emulsions and foams, such as the ones encountered in water-in-crude oil emulsions and froths in fermentation processes. In the first part of the lecture, the mechanisms by which particles may stabilize a surface or an interface are reviewed, as well as the effect of particle wettability, size and concentration on stability. Some applications in the pharmaceutical and food industry are presented in the second part.

CELIS M. T., GARCIA-RUBIO L.

Droplet size distribution of liquid-liquid systems from UV-VIS spectra.

Particles 2006 Symposium. Orlando FLA. May13-16, 2006

ABSTRACT: The stirring of a surfactant/water/oil system result in the generation of droplet populations determined primarily by the nature and amount of emulsifier, mixing characteristics and emulsion preparation. The estimation of the droplet size and droplet size distribution is important data, not only because they are linked with the manufacturer process, but also they give valuable information on the properties of the dispersed phase. This research demonstrates the potential of spectroscopy as a tool for characterization of liquid-liquid systems in terms of droplet size distribution as a function of the oil concentration and of physicochemical variables. The opportunity of obtaining information from a single multiwavelength measured makes UV-Vis spectroscopy a powerful tool for characterization of dispersed systems.

RODRIGUEZ-ABREU C., DELGADO-LINARES J. G., BULLON J.

Properties of Venezuelan Asphaltenes in the bulk and dispersed State.

J. Oleo Science **55** (11) 563-571 (2006)

ABSTRACT: The properties of asphaltenes derived from Venezuelan vacuum residues have been investigated using several techniques. X-ray diffraction and Differential Scanning Calorimetry (DSC) and infrared spectrometry results indicate that asphaltenes are complex mixtures that form nanostructures with some (low) degree of ordering, probably consistent with a sheet-like arrangement of aromatic nuclei. Nanostructures seem to be also present in solutions of the asphaltenes in toluene, and an increase in their concentration can induce phase separation by flocculation. Asphaltene aggregates show an isoelectric point at around pH=4 and their charge is highly negative at high pH. This is attributed to the presence of ionisable acidic groups that also are responsible of low water/toluene interfacial tensions at basic conditions. A decrease in interfacial tension is also induced by increasing asphaltene concentration or the addition of electrolyte. The results are correlated with the stability of water-in-oil emulsions, which seems to be determined by interfacial adsorption.

SALAGER J. L., FORGIARINI A., LOPEZ-MONTILLA J. C., MARQUEZ L.

Low-energy Emulsification by Phase Inversion Techniques – Trends and Tricks.

Paper# 023. 4th World Congress on Emulsion, Lyon France. Oct. 3-6, 2006

ABSTRACT: There are different methods to produce an emulsion (particularly nano-emulsion) by “persuasion” rather than “brute force” stirring. All of them use a situation in which the emulsion is evolving more or less spontaneously as a change in formulation or composition or equilibrium state takes place. Some are transitions whereas other are instabilities involving or not spontaneous emulsification. The different mechanisms are outlined and the ways they can be harnessed to lead to a fine and/or stable (nano)emulsion are discussed. Some of them are well known such as the PIT method, whereas others have been understood only recently and some are still puzzling, such as some dilution methods. These methods are applied to real processes such as waterborne resin manufacturing, pesticide conditioning or cutting oils are discussed.

PIZZINO A., CATTE M., VAN HECKE E., AUBRY J. M., SALAGER J. L.

La Rétrodiffusion de Lumière: Une nouvelle technique non invasive de détection de la Température d’Inversion de Phase des émulsions.

Paper # 095. 4th World Congress on Emulsion, Lyon France. Oct. 3-6, 2006

RÉSUMÉ: L’inversion de phase d’une émulsion correspond au passage d’une morphologie huile-dans-eau (H/E) à une morphologie eau-dans-huile (E/H) ou vice versa. L’inversion de phase est utilisée dans l’industrie, comme méthode d’émulsification sous faible agitation pour l’obtention de petites gouttes, dans la fabrication de peintures ou de cosmétiques. Les phénomènes associés à cette inversion ne sont pas tous parfaitement maîtrisés, mais il est bien connu qu’elle peut être induite par un changement de température. La Température d’Inversion de Phase (TIP) des systèmes contenant des tensioactifs nonioniques polyéthoxylés, est en général déterminée par conductimétrie, toutefois avec certaines difficultés d’interprétation, en particulier en absence d’électrolytes. La nouvelle méthode présentée utilise une sonde de rétrodiffusion de lumière, plongée au sein de l’émulsion, qui permet de détecter l’inversion de phase même dans des systèmes exempts d’électrolyte. La méthode est appliquée à l’inversion de phase thermo-induite du système C₆E₂/tétradécane/eau. Les résultats concordent avec les données de conductivité. Contrairement à l’idée généralement admise, on constate de fortes variations de la TIP avec le sens de variation de la température et la cinétique de chauffage et/ou refroidissement. Ce phénomène d’hystérésis est interprété sur la base du comportement de phase des systèmes à l’équilibre.

RONDON-GONZALEZ M., SADTLER V., CHOPLIN L., SALAGER J.L.

Emulsion inversion produced by continuous stirring without internal phase addition. Effect of the phase viscosity ratio.

Paper # 312. 4th World Congress on Emulsion, Lyon France. Oct. 3-6, 2006

ABSTRACT: Emulsion inversion, i. e., the emulsion morphology swap from oil-in-water (O/W) to water-in-oil (W/O) or *vice versa*, could result from the continuous stirring of a so-called abnormal system, and is an alternative to attain fine and concentrated emulsions, particularly with a high viscosity fluid. The inversion mechanism, as well as the stirring time required for inversion, is reported to be affected by the phase viscosity ratio. For

systems containing nonionic surfactants, and a phase viscosity ratio near unity, the inversion takes place through the formation of a multiple emulsion (w/O/W) in which a portion of the external phase is continuously included as droplets in the dispersed phase drops. Consequently, the dispersed phase apparent volume increases until a critical value is reached and the inversion is triggered. As the internal phase viscosity is increased, the dispersed phase content required to trigger the inversion decreases, and the inversion time remains constant up to a viscosity ratio about 10 beyond which it decreases. This results are interpreted on the basis of the deformability of the oil drops. On the other hand, as the viscosity of the external phase is increased by the addition of carboxymethylcellulose, the critical dispersed content decreases as well as the viscosity of the resulting emulsion. The experimental evidence indicates that the mechanism of inversion is more complex than the mere formation of a multiple emulsion.

FORGIARINI A., MARQUEZ L., CELIS M. T., SALAGER J. L.

Nanoemulsification of triglyceride oil by mean of a inversion method.

Paper #318. *4th World Congress on Emulsion*, Lyon France. Oct. 3-6, 2006

ABSTRACT: Emulsions are mixtures of two immiscible liquids, one of them being dispersed as droplets in the other. When droplet size is in the range of 20 to 500 nm it is often called nano-, mini- or submicron emulsion. Different methods have been proposed to prepare nanoemulsions, among which the transitional inversion (at fixed composition) so-called phase inversion temperature (PIT) method, commonly used in systems formulated with nonionic ethoxylated surfactant. In general and in particular with other types of surfactant, the transitional inversion can be carried out by changing any other formulation variables likely to alter the hydrophilic-lipophilic deviation (HLD) of the system with respect to optimum formulation for three-phase behavior, like the salinity of the aqueous phase, the surfactant or surfactant mixture hydrophilicity or the cosurfactant effect.

The present report deals with the attainment of O/W nanoemulsions with a nonionic surfactant/triglyceride oil/water system. The initial systems were formulated at different HLD values and different Span/Tween nonionic surfactant mixtures. Emulsification was carried out by dilution, i.e., by a water-to-oil ratio change. The nanoemulsion drop size and stability are related to the nonionic surfactant mixture HLD and to the existence of a lamellar liquid crystalline phase during the emulsification process. The data highlight the particular importance of lamellar phases in nanoemulsion formation and stabilization.

BRICEÑO M. I., CELIS M. T. SERVIGNA F., SALAGER J. L.

Evolution of physical properties of concentrated o/w emulsions during mixing: influence of physico-chemical formulation.

Paper # 344. *4th World Congress on Emulsion*, Lyon France. Oct. 3-6, 2006

ABSTRACT: In this work we evaluated the evolution of droplet size and rheological behavior of a coconut oil-in-water emulsion during the mixing process as a function of physico-chemical formulation. The emulsions were stabilized by means of mixtures of nonionic surfactants. The proportion of these surfactants was varied as to obtain different HLD values (Hydrophilic-Lipophilic Difference). The latter is a non-dimensional parameter associated to formulation; it can be calculated and depends on the nature of the oil and water phases, as well as on surfactant type and temperature. The emulsions were prepared as follows. First, the oil was slowly added to the aqueous surfactant solution using low

mixing speed; a rather coarse 80 % oil dispersion was obtained. Then, the mixing speed was increased and small samples of emulsions were taken during the stirring process. Droplet size distribution was measured for every sample as well as the rheological dynamic behavior performing a stress sweep under oscillatory conditions. The resulting Sauter diameter, yield stress τ_0 and elastic modulus G' were analyzed as a function of mixing time and HLD value. It was found that, as expected, droplet size diminished as mixing time increased up to an asymptotic value. However, the rate of droplet reduction was much greater as HLD increased. Droplet size decrease was related to diminishing values of τ_0 and G' . Further, for similar droplet size, τ_0 and G' tend to increase as HLD is reduced. As a fact, higher HLD is associated to lower interfacial tension. Therefore, both droplet size and rheological behavior seem to be controlled by interfacial tension during the mixing process.

DEL GAUDIO L., ALBONICO P., BELLONI A., MARQUEZ L.

A low energy method for water-in-oil nanoemulsion preparation.

Paper #476. 4th World Congress on Emulsion, Lyon France. Oct. 3-6, 2006

ABSTRACT: Nanoemulsions are characterized by a droplet size between 20 and 500 nm. They are not thermodynamically stable but they can possess long term stability if adequately prepared. Over the last few years there has been increasing interest in the development of commercial applications of nanoemulsions in the cosmetic, pharmaceutical and chemical fields. Many publications have appeared recently, underscoring the importance of developing new and low cost routes to stable nanoemulsions. The production of very small droplets in emulsions is difficult and requires the application of a large amount of energy that can be achieved either by low energy or by high energy methods. To date, relatively less work has focused on the preparation of water-in-oil (W/O) nanoemulsions by low energy methods. In this paper a new method is described for preparing water-in-oil nanoemulsions by a low energy approach based on phase inversion induced by phase boundary crossing. The preparation consists of three steps in which different zones of the phase diagram are crossed. These three steps must be well controlled to favor fast nanoemulsion formation and to obtain stable nanoemulsions. In the first step a concentrated additive solution is prepared by mixing a fixed amount of oil, water and surfactant. In the second step the remaining water is added and a new solution is obtained (called the precursor). After the final dilution with oil a delay time is necessary to obtain nanoemulsions. Nanoemulsions with different formation kinetics and stabilities have been made. The preparation route developed appears to be quite general. It can be applied to different surfactants and oils, opening a route to the preparation of W/O nanoemulsions of interest in many industrial applications.

CARDENAS A., SALAZAR F., BULLON J., RENNOLA L.

Membrane/Crude oil behavior in dilute oil/water emulsion filtration.

Paper 508. 4th World Congress on Emulsion, Lyon France. Oct. 3-6, 2006

ABSTRACT: Crude oil emulsions are often found in different operations of the oil industry. In crude oil production, after previous treatments for water/oil separation, like in API separators, very dilute crude oil-in-water emulsions are obtained. These emulsions should, in many cases, be further treated if water is to be reused or if it is disposed in the environment. One of the processes that could be used for this treatment is membrane filtration, where water can be separated from the small oil droplets. In this work different

organic membranes were used to filter dilute crude oil-in-water emulsions and their behavior is studied. Three different types of crude oil were used: light, medium and heavy with API gravities of 33.4°, 24.6° and 13.8°. Zeta potential, contact angle and interfacial tension measurements were carried out. Results of the filtration experiments show that when the membrane is more hydrophobic, there is more adsorption of crude oil and then more membrane fouling. This agrees with the contact angle measurements that show that as wetting of the membrane with oil increases, the fouling also increases. Also, when physico-chemical conditions are changed, as pH for example, the wetting of the crude oil conditions change and fouling can be reduced.

MARQUEZ R., BULLON J., MARQUEZ L., BRICEÑO M. I., FORGIARINI A.

More efficient emulsification procedure to obtain parenteral emulsions.

Paper 511. 4th World Congress on Emulsion, Lyon France. Oct. 3-6, 2006

ABSTRACT: A more efficient emulsification procedure to obtain parenteral emulsions composed by lecithin, soybean oil, water and glycerol was investigated making use of the know-how in low energy emulsification by phase transition from a W/O to a O/W system. For relationships S/(S+O) greater than 3.5, a change was found in the physicochemical behavior of the system. A viscoelastic liquid crystal mesophase was studied through the measurement of conductivity and viscosity through out the emulsification procedure. We prepared a lipid emulsion with a droplet size distribution with diameters below 4.2 μm using an emulsification procedure which is more efficient from an energetic point of view than those used in industry.

CHAVEZ G., PIÑA I., BRAVO B., CÁCERES A., YSAMBERTT F., SALAGER J. L., MARQUEZ N.

Development of a procedure of analysis to study the EON distribution of nonionic surfactant mixture of the polyethoxylated alcohol type by HPLC-IRD and its application in the partition coefficient determination (Spanish)

Ciencia 14 (2) 223-243 (2006)

RESÚMEN: Desarrollo de un procedimiento de análisis para estudiar la distribución de EON de mezclas de surfactantes no-iónicos de tipo alcohol polietoxilado por HPLC-IRD y su aplicación en la determinación del coeficiente de reparto.

Se desarrolló un procedimiento de análisis para determinar la distribución del número de óxido de etileno (EON) de mezclas de surfactantes no-iónicos de tipo alcohol polietoxilado (APE) utilizando la cromatografía líquida de alta resolución (HPLC) con detección de índice de refracción (IRD). Se estudió la separación de las cadenas polietoxiladas con columnas analíticas de fase normal (gel de sílice) y fase inversa (C8) variando la proporción de los componentes de la fase móvil. La mejor separación de la parte hidrocarbonada y resolución de la distribución de cadenas polietoxiladas fue obtenida a 25°C utilizando la cromatografía líquida en fase reversa con la mezcla acetonitrilo/agua 80/20. La concentración mínima detectable fue de 50 $\mu\text{g mL}^{-1}$ para el análisis de muestras comerciales de APE, y de 40 mg mL^{-1} para el análisis de APE en sistemas agua/microemulsión/aceite. El procedimiento propuesto es aplicable para el control de calidad de muestras comerciales de surfactantes con distribución de EON entre 1 y 20 unidades.

PIERINI C., BRACHO C., GONZALES G.

Formulation of Asphalt Emulsion with Lignin Derivatives and its Behavior with Mixtures of Stony Materials. (Spanish)

Revista Técnica Ing. Univ. Zulia **29** (2) 134-143 (2006)

RESÚMEN: Formulación de emulsiones asfálticas con derivados de lignina y su comportamiento con mezclas de materiales porosos.

En el presente trabajo se evaluó la utilización de los derivados de ligninas en la formulación de emulsiones asfálticas y su comportamiento como mezclas con agregados pétreos de buena calidad. En esta investigación de naturaleza descriptiva, se formuló la emulsión asfáltica con experimentos en laboratorio y planta piloto, y el comportamiento con mezclas de materiales pétreos por el método Marshall modificado de Illinois. Se utilizaron tres tipos de ligninas comerciales y una recuperada en el laboratorio a partir del licor negro mediante un proceso de extracción con ácido sulfúrico. La formulación condujo a una emulsión del tipo no iónica, con un comportamiento adecuado al ser mezclado con materiales pétreos con propiedades controladas. Se concluyó que la mezcla encontrada presenta propiedades óptimas como capa base en pavimentación, limitado su uso para carpeta de rodamiento por el porcentaje de pérdida de estabilidad, donde el factor determinante podría ser el tiempo de curado.

SALAGER J. L.,

Formulation Concepts for the Emulsion Maker,

In *Pharmaceutical Emulsions and Suspensions*, edited by F. Nielloud & G. Marti-Mestres, Eds., 2nd Edition, Chap. 2, Taylor & Francis, London (2006).

ABSTRACT: This is a self-study text on the basic concepts that an emulsion maker should know to handle the intricacies of physico-chemical formulation, whatever the application, pharmaceutical or other. It spans from very simple concepts such as HLB number to complex but up-to-date SAD numerical expression that allows to take into account the effect of all variables (including salinity, temperature and even pressure). A section on experimental methods surveys the formulation scan technique and how to use it in practice. This chapter contains 21 figures, 126 references and 3 tables of numerical data.

SALAGER J. L.,

Emulsion Properties and related Know-how to attain them,

in *Pharmaceutical Emulsions and Suspensions* edited by F. Nielloud & G. Marti-Mestres, Eds., 2nd Edition, Chap. 3, Taylor & Francis, London (2006).

ABSTRACT: It is a self-study text which presents the state of the art in relating the emulsion properties to formulation, composition and emulsification protocol, whatever the application, pharmaceutical or other. The emulsion characteristic properties (type, conductivity, drop size, viscosity and stability) are first analysed. Then the influence of physico-chemical formulation, as SAD, on all emulsion properties is discussed. The case of emulsions containing a large amount of either oil or water is treated next on a bidimensional map (formulation-composition) that is presented as the general phenomenology map for emulsion properties forecasting. The variations of this map with other variables (phase viscosity, surfactant concentration, stirring energy) is outlined. The last section is dedicated to an introduction on dynamic phenomena leading to transitional and catastrophic inversion of the emulsion. This chapter contains 23 fig. and 118 refs.

BRICEÑO M. I.,

Rheology of Suspensions and Emulsions,

in *Pharmaceutical Emulsions and Suspensions* edited by F. Nielloud & G. Marti-Mestres, Eds., 2nd Edition, Chap. 16, Taylor & Francis, London (2006)..

ABSTRACT: This is a self-study text that introduces the reader to basic rheology concepts before describing the main features exhibited by emulsions and suspensions. It starts with the definition of Newtonian and non Newtonian behaviors, particularly those exhibited by dispersed systems as shear thinning, viscoplastic, as well as thixotropic. Viscoelastic behaviors is outlined, with stress relaxation, creep compliance and recovery features. A section on rheometry discusses the typical arrangements used in apparatuses: concentric cylinders, cone and plate, parallel plates, capillary, rolling ball. The following section describes the main rheological characteristics of emulsions and suspensions, and specifies the effects of the different physical variables (internal phase content, particle size and shape, particle or drop size distribution). The bimodal distribution influence on emulsion viscosity is described in details. Physico-chemical phenomena, such as the interparticle forces commanding aggregation and flocculation mechanisms, as well as electroviscous and solvation effects, are discussed as well. The chapter ends with two practical sections in which the reader can find advice on how to avoid common mistakes as far as the interpretation of the data is concerned, how to select the best apparatus in each case, and how the formulation can be related to the product rheology.

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Diseño y construcción de una planta piloto para el tratamiento de aguas residuales por lodos activados y su puesta en marcha para tratar vinanzass de una destilería.

Ciencia e Ingeniería **27**: 145-151 (2006)

RESÚMEN: Se diseño y construyó una planta piloto para el tratamiento de aguas residuales por lodos activados, constituida por un tanque de alimentación, una cámara de reacción, un sistema de aeración, un sedimentor, una bomba de reciclo y accesorios de conexión. La planta piloto puede ser utilizad para el tratamiento aeróbico de diversas aguas residuales. El caudal de alimentacion puede variar entre 0.1 y 5 L/h. Dada la problemeatica ambiental ocasionada por las vinazas de las destilerías se utilizó la planta para tratar dichas aguas residuales utilizando *Candida utilis* como cepa biológica. La remoción de DBO fue de 90 % y con respecto a DQO mayor que 33 %.

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