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## Two dozen papers at Barcelona meeting

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Approximately two dozen papers were presented to general sessions of the 10th meeting of the Spanish Committee on Surface Active Agents, held during March in Barcelona.

Brief summaries of some of the papers are presented below. Further information about the meeting is available from Dr. P. Rihosa Arno, Comité Español de la Detergencia, Secretaria General, Centro de investigación y desarrollo; Patronato Juan de la Cierva, c/o Jorge Girona Salgado, Barcelona-17, Spain. Proceedings of the conference will be published in September and will cost 3,200 pesetas (Approx. US \$45).

*Problems of the Ultimate Biodegradability of Linear Alkylbenzene Sulfonates.* Pavel Pitter and Tomas Fuka, Department of Water Technology and Environmental Engineering, Institute of Chemical Technology, Prague, CSSR.

In this paper biodegradability of LAS was compared with alkyl sulfate, alkene sulfonate and alkane sulfonate. The content of organic substances was specified by determining COD, TOC, MBAS and UV. Substances under study were the only source of organic carbon for the microbes of the inoculum (the inoculum being long adapted activated sludge). Inoculum concentration was always equal to 100 mg.l<sup>-1</sup> of dry matter. The initial LAS concentration ranged from 25 to 65 mg.l<sup>-1</sup> of the active matter. Even a long term adaptation of the activated sludge did not achieve the ultimate biodegradation of LAS expressed in the percentage of COD or TOC. The determined biodegradability amounts on the average to 65.5% COD, 64.0% TOC, 63.3% with the respect to the absorption in the UV region, and 95.5% in terms of the reaction with methylene blue (MBAS). The discussion is concerned with potential effects of the formation of residual substances, the auto-oxidation and autolysis of the microbial inoculum, and the method used upon the degree of biodegradation. The results show a need to change the criteria used to evaluate the degradability of surfactants. The degradability should be considered and evaluated according to the decrease of COD or organic carbon (TOC).

*The Significance of Perfume in a Product Concept for Toilet Soap.* Hans Otto Schmidt, Haarmann + Reimer GmbH, Holzminden.

The significance and task of the soap perfume – like those of color, appearance, and shape – can be quite different in the segment of complexion care or in the freshness sector of the market. Requirements for a perfume that has to convey the message of skin care in white or lightly colored soaps are distinctly different from those of fresheners. In the small but exclusive segment, the significance of the perfume is increased to a point where it almost becomes the basic function of the product and body cleansing the additional value benefit. The product concept of a toilet soap with all its details is determined by the target market position. A detailed briefing about this concept is necessary not only for the advertising agent or the packaging designer, but also for the perfumer to ensure that the right perfume is selected to work harmoniously with other concept details.

*Sorption of Cosmetic Materials onto Hairs and Its Conditioning Effects.* Hiroshi Watanabe, Kao Soap.

Materials that have to be considered are surfactants, fat and oil additives, and other polymers. The amount of these materials remaining on the hair depends on such factors as their characteristics, amount used, formulation, systems, and hair conditions. The following summarizes results of our investigation: (1) As the damage of hair cuticle advanced, the hair surface became more hydrophilic and the sorption of hydrophobic fat and oil materials became more difficult, making the sorption of surfactants easier. (2) Decreasing order for the sorption of surfactants is cationic, anionic, and nonionic surfactants. (3) It was found that the use of cationic or amphoteric surfactants was effective in making the damaged hydrophilic hairs be hydrophobic so that hydrophobic materials could be absorbed onto the hairs.

*The Production of High Performance Detergent Powders by Low Temperature Fluid Mix Agglomeration Technique.* J.L.A. Briggs, Anhydro A/S.

This paper discussed the use of the Anhydro low temperature Fluid Mix agglomeration technique to produce high quality detergent powder, equivalent in most aspects and superior in others, particularly solubility, to that of a leading market brand in the U.K., manufactured by spray drying.

*Modern Methods to Produce Anionic and Nonionic Surfactants from Natural Oils and Fats.* Alfred S. Davidson and Domenico Triberti, Ballestra, S.p.A.

The paper described methods to transform natural fats and oils, particularly nonedible fats and oils, into their methylesters and to use the methylesters either to produce alkylolamides or alpha-sulpho-methyl-fatty-acid-ester. Methods were given for prerefining of the fatty raw materials, such as tallow, palm oil and also coco-oil, especially for their transformation into methylester. Furthermore, a short description for the ester-interchange process is given and the analytical control methods for the process. Transformation of the various methylesters into alkylolamides was described and some information given for the application of the various alkanolamides in finished detergents. The sulphonation process, based on SO<sub>3</sub>-converter gas for the production of fatty alpha-sulpho-methylesters, was described based on extensive pilot tests in a standard cascade sulphonation system.

*Human Safety Testing of Surfactant/Detergent Systems. I.* Smith, Inveresk Research International, Edinburgh.

Human safety testing of chemicals such as surfactants is mainly the evaluation of irritation and sensitization potentials on the skin. The irritancy and sensitization (or allergic) evaluations can be initially carried out using the relevant animal tests, but to avoid some uncertainties with the interpretation of the animal vs. human response, human tests are carried out, mainly by patch test methods intended to exaggerate the exposure over the course of 3-4 weeks with a challenge made to the skin after a 2 week period. The classical statistical correlation by Henderson and Riley between a negative patch test results from a representative sample, and the in-use consumer population

has shown that even if there were no positive reactions to a product in a test panel of 100 subjects, the rate of positive reactions in the general population is not likely to exceed 2.9% at the 95% confidence level. A negative test result on a panel of 1000 volunteers would reduce the likelihood to 0.3% — but the cost would limit cost/benefit to the point where no company could feasibly afford this extent of testing for every new product. It is thus important that the test design (concentration, exposure, panel numbers) must be as reliable as possible to detect a sensitizer if one exists in the product. This paper set out to detail optimal procedures and high quality assurance necessary to conduct testing to identify products/ingredients capable of sensitization.

*On Zeolite/Surfactant Systems.* N. Filipovic-Vincekovic, M. Cebulc, Lj. A. Despotovic, R. Despotovic, B. Subotic, V. Tomasic, D. Zitnik, Z. Demetar, and L. Sekovanic, Rudjer Bokovic, Institute.

In the past four years, the synthetic zeolite, type (NaA or NaX), with large capacity for multivalent ions, has showed the excellent properties for phosphate substitution. Conventional detergents are composed of water soluble components, but synthetic zeolite is water insoluble. It is of utmost importance to prepare zeolites with convenient surface properties (stable colloid suspension during washing) to prevent incrustation on fabrics. Crystalline zeolite of high capacity for  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  ions were dispersed in hard water ( $10^{\circ}\text{d}$ ) of aqueous solutions containing standard detergent surfactant sodium p-dodecylbenzenesulfonate both with and without sodium tripolyphosphate. The dispersed zeolite (negative colloid) is sufficiently stable in hard water during the period of time needed for standard washing operation. The mechanism of interaction of inorganic sols and surfactant is a complex one and depends on the type and the concentration of present surfactant. Interactions of oppositely charged inorganic sols and surfactant depend on surfactant concentration in the sense of flocculation, stabilization and mutual coagulation of sols. Similar phenomena were observed in the systems with the same charge of sols and surfactant. The turbidity measurements of systems zeolite + sodium dodecylbenzenesulphonate (negative colloid + micellar solution of anionic surfactant) show a high stability of dispersed material. The chosen concentration of surfactant was in the range of mutual interaction of sols. In the measured time interval (150 minutes), this interaction was not yet finished (the mutual coagulation is a kinetic process). The latest recommendation is to produce the detergents with partially substituted phosphate. Several experiments were performed in order to examine the stability properties of the system zeolite + sodium tripolyphosphate + surfactant. The present sodium tripolyphosphate does not decrease the stability of negatively charged zeolite for one hour, but in 150 minutes stability of system decreased about 40%. After the systems were shaken, the turbidity was unchanged. It means that in combination with washing machine operation (shaking of washfloat), the combination of zeolite + tripolyphosphate is usable in the light of the results obtained for the stability of systems. The present experiments confirmed good properties of synthesized zeolite, but for a definite statement it is necessary to examine the complex interaction of colloid zeolite with other components in detergent formulation.

*Evaluation of Physico-Chemical Properties of Synthetic Zeolites Useful in Washing Processes.* Z. Demeter and L. Sekovanic, KGK, Karlovac, Croatia, Yugoslavia.

Among prepared zeolites we have chosen three zeolites as the most promising ones for the substitution of sodium tripolyphosphate. Two of the chosen zeolites have identical

chemical and structural characteristics (crystalline zeolite A). Variation of gel composition and the conditions of crystallization process enabled the crystalline zeolites A to be produced in different crystallization times. Depending on time needed for complete crystallization, the zeolites have particles of various sizes. These zeolites show excellent exchange capacity for calcium and magnesium ions, and slow adsorption capacity for organic dyes. The third chosen zeolite has a high exchange capacity for calcium and magnesium ions and excellent adsorption capacity for organic dyes. A number of zeolites with low or medium exchange capacity but of high adsorption capacity for organic dyes were prepared too. All three chosen zeolites possess good colloidal stability in water and in the presence of different ions. The present surface active agents influence the colloid stability of zeolites. The results of investigations on physico-chemical properties of produced synthetic zeolites show that the chosen zeolites are suitable substitutes for sodium tripolyphosphate in the washing formulations. The described zeolites can be used separately or as various mixtures of prepared zeolites.

*Trends in Formulation and Use of Household and Institutional Automatic Dishwasher Detergents in the U.S.A.* Robert J. Fuchs, FMC.

Formulation changes in household automatic dishwasher detergents were reviewed, with emphasis on phosphates, silicates, and active chlorine compounds. The slowing market growth of automatic dishwasher detergents in the U.S. from 16-18% per year to 5-6% per year were discussed. Recent trends in energy conservation and their possible impact on household and institutional products were discussed.

*Process Technology of the Manufacture of Phosphate-Containing Powdered Detergents.* G. Sorbe, Hoechst AG.

The process technology can be divided into processes in which the individual components are worked up into agglomeration products (spray-mixing plants) and processes in which the individual components are combined to form a slurry or solution comprising several substances (spray-drying plants). More than 90% of the washing powders produced throughout the world are spray-dried-detergents. The preparation of the slurry and the properties of the phosphates are decisive with regard to the economical operation of the spray tower and the final quality of the product. In preparing the slurry, the addition of sodiumtriphosphates leads to a change in the viscosity of the slurry which is due to the hexahydrate formation. Sodiumtriphosphates, the grain size distribution, purity and impurity, phase-I-content, hexahydrate-content all influence slurry viscosity. The main reaction in the slurry is the hydration of pentasodiumtriphosphate to pentasodiumtriphosphate-hexahydrate. Hydration is influenced by several parameters including the quality of sodiumtriphosphates and slurry conditions. Simultaneously with hydration, hydrolysis processes take place in the slurry. These are regarded as secondary reactions.

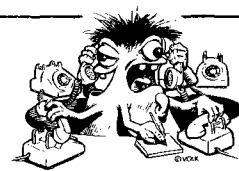
*Various Fields of Industrial Application of Surfactants.* H. Stache and S. Scholz-Weigl, Chemische Werke Hüls.

The wide spectrum of surfactants for industrial application was broken down into sections with special attention given to the fiber and textile industry, the fur and leather industries, and the wood pulp and paper industry. The industrial cleaning operations range from commercial laundering and dry-cleaning, the cleaning of large surfaces in institutional buildings to industrial cleaning processes. Secondly, the use of surfactants as emulsifying agents was examined in the construction material industry, the paint and varnish industry, pest control, and the metal-working

industry. In the chemical industry, emulsifying agents are used in polymerization processes. Third, the use of surfactants in the foodstuffs industry was dealt with, with particular emphasis on emulsification and improving solubility of hydrophobic substances. Alkylbenzene sulphonate is still

the number one attraction among surfactants because of its availability, its properties and its price. The portion of nonionics is steadily increasing due to the great variability of the molecular structure. Cationics and ampholytes are used as special products on a limited scale. ●

# SD&C Industry News



The Food and Drug Administration has found nitrosamines in some cosmetic products and is considering what action to take in view of studies that link nitrosamines to animal cancer.

In the Federal Register of April 10, the FDA said the potential hazard could be substantially reduced by reformulation to avoid use of nitrosating agents in products containing alkanolamines. Meanwhile, the agency asked for comments, scientific data or other information regarding nitrosamine contamination in cosmetics, specifically seeking toxicological data on *N*-nitrosodiethanolamine (NDELA), its penetration through human skin, and chemistry.

Carcinogenicity of NDELA has been established in two animal species, the FDA said, and other studies have indicated it penetrates the skin of live monkeys and "penetrates excised human skin from an aqueous vehicle." An analysis of 29 suspect cosmetics detected NDELA in 27 of the 29, at levels up to 48 ppm, the FDA said. A second study of 191 selected cosmetics showed 114 were free of NDELA.

"The most highly contaminated cosmetic products usually contained as ingredients both an alkanolamine, generally triethanolamine, and a nitrosating agent," the FDA said. Lower levels of contamination are more likely when the nitrosating agent is not an intentional ingredient, but a contaminant of a regular ingredient.

The FDA is continuing to study the identity, source and formation of nitrosamines, including NDELA, in cosmetics, as well as the potential risk to public health and the extent of human exposure to nitrosamines through cosmetics.

Nitrosamine-contaminated cosmetics could be considered adulterated and subject to regulatory action, the FDA said, but said it first wanted to see if voluntary industry action would be sufficient to eliminate the problem. ●

## Cosmetic, toiletry market reviewed

Cosmetic and toiletry raw materials sales in the United States are expected to rise approximately 16% between 1978 and 1982, according to a market study by Charles H. Kline & Co.

The report forecasts 1982 sales at \$810 million on a volume of 1.6 million pounds, compared to \$695 million in 1978 sales on a volume of 1.3 billion pounds.

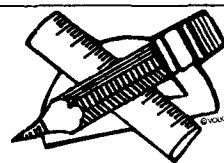
Kline said that more than 100 different products, classified into nine products groups, are purchased by more than 900 manufacturers of cosmetics and toiletries. Sixty per cent of the raw materials sold fall within three product groups—fragrances, surfactants, and organic commodities. Of the 100 raw material suppliers, the thirteen largest companies (with annual sales of \$15 million or more) account for 43% of total industry sales. In terms of physical volume, sales are expected to rise by 4.7% through 1982, Kline said, with surfactants increasing at a 6.6% annual rate.

The 570-page report is available from Kline & Co., 330 Passaic Ave., Fairfield, NJ.

TABLE I  
Estimated U.S. Consumption of Cosmetic and Toiletry  
Raw Materials by Product Type, 1978

Product type	\$ Million	% Of total
Fragrance compounds	\$170	24%
Surfactants	160	23
Organic commodities	90	13
Organic specialties	65	9
Inorganic chemicals	60	9
Fatty chemicals	55	8
Propellants	50	7
Biocides	25	4
Petroleum products	20	3
Total	\$695	100%

# SD&C Abstracts



EFFECT OF A SKIN CREAM CONTAINING THE SODIUM SALT OF PYRROLIDONE CARBOXYLIC ACID ON DRY AND FLAKY SKIN. J.D. Middleton and M.E. Roberts. *J. Soc. Cosmet. Chem.* 29(4), 201-5 (1978). Humectants added to skin creams can increase the moisture retention of isolated corneum and reduce the incidence of dry and flaky skin in vivo. Results are given of an investigation into the efficacy of a humectant, the sodium salt of pyrrolidone carboxylic acid (NaPCA), which occurs naturally in the corneum. A product containing 5% of NaPCA increased the water-holding capacity of isolated animal corneum.

PRECIPITATION OF INSOLUBLE SALTS ON FABRICS LAUNDERED IN

HARD WATER IN THE PRESENCE OF DIFFERENT ALKALIS AND BUILDERS. S.V. Vacck, *Tenside Deterg.* 15(6), 286-90 (1978). Cotton and polyester-cotton fabrics were laundered 20 times in hard water at 85 C in the presence of an anionic surfactant and different concentrations of alkalis and sequestering agents. When normal alkalis and pyrophosphate were used, high levels of insoluble salt deposits were found. Sequestrants reduced the level of insoluble salt deposits to negligible values already at low concentrations. Neutral electrolytes also reduced the level of salt deposits. Tripolyphosphate caused increased levels of deposits at low concentrations and negligible values above 2 g/l. An aluminosilicate (Sasil) showed a similar behavior but the deposits level did not decrease below 1%. Distinct differences in the greying behavior were also observed. ●