

Fragrance in Emulsion and Surfactant Systems

Steve Herman

AFF International, Totowa, N.J., USA

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ABSTRACT: *Fragrance is a complex system of aroma chemicals whose potential to interact with the base in formulated products is reviewed in this article, with a focus on fragrance in emulsion systems and surfactant systems.*

Much has been written on fragrance chemistry and applications, but many formulators remain unaware of the detailed mechanisms by which aroma chemicals interact with bases. With the daunting complexity of fragrances, no text will resolve all the situations that are encountered. Nevertheless, an understanding of the underlying chemistry can notably enhance the probability of success.

Fragrance is used in most personal care products. Sometimes, its purpose is simply to mask an undesirable base odor; more often it is a key sensory attribute. For some products, such as shampoo and body wash, fragrance may be the most important element in consumer appeal.

Formulators usually view fragrance as a single ingredient, a liquid in an amber bottle, often added at the end of the development process with little regard for its properties. The marketing department treats fragrance by its odor description, demographic appeal and cost. In reality, fragrance often is the most chemically complicated component of the formulation, and understanding its technical aspects is essential for the creation of an acceptable product.

Fragrance Materials

In constructing a fragrance, volatility is a key factor. The materials that are

most volatile, and last the shortest time during use, are called top notes. Materials of intermediate volatility comprise the middle notes. The least volatile, longest-lasting components make the bottom notes. A pleasing blend of a few materials is called an accord. A fragrance, in its most basic form, can be constructed from a bottom accord, a middle accord and a top accord. More than 40 years ago, Jean Carles¹ provided a classic summary of fragrance construction that is still valid.

Many fragrances are created first as hydroalcohols, for which the term *perfume* usually is used. If the fragrance is successful, line extensions are made into skin, hair and bath products. The materials that can be employed—and the balancing of the components—must be modified for aesthetic, economic and technical considerations.² It is easy to understand the economics. While functional fragrances frequently derive from fine fragrances, the fine fragrances have much higher price points. Expensive nuances in the fragrance usually will not be used in personal care. The aesthetic and technical aspects of translating fragrance into successful applications will occupy the remainder of this article.

Perfumers, even if they are blissfully unaware of chemistry, learn to solve many common problems through training and experience. When modify-

ing fragrance for a specific base, some materials must be removed because of their negative impact on stability. Materials must be added to the fragrance or subtracted from it to compensate for the physical or olfactory qualities of the base. Raw material studies can identify problematic ingredients. Databases give the stability of aroma chemicals over a wide pH range or in common applications. Compendiums from aroma chemical suppliers frequently include indications of appropriate applications.

Although it is possible to solve problems without a theoretical foundation, the curious formulator will still desire a deeper understanding of why fragrances behave as they do. Fragrances embrace a widely diverse range of chemical types. Some characteristic examples of those types are shown in **Figure 1** on page 60. Aroma chemicals often contain more than one functional group, and they each contribute to the properties of the material. The literature includes numerous books³⁻⁸ that treat the chemistry, synthesis and properties of aroma chemicals in detail.

THE CREATION OF A FRAGRANCE

The classic tale of accidental perfume creation concerned the earliest successful fragrance, Eau de Cologne. Allegedly, a perfumer in Cologne fell down some stairs while carrying a tray of various flower extracts. The resultant mixture seemed not only pleasant, but noticeably stronger than any of the individual ingredients. The effect of the whole being found to be greater than the parts, modern perfumery was born.⁵⁴

Fragrance in Emulsion Systems

Since the 1940s, application chemists have studied the effects of fragrances in emulsion systems.⁹⁻¹¹ With emulsions, the obvious consequences of undesirable fragrance interactions were the thinning and breaking of emulsions, and discoloration. Odor changes taking place over time include the loss of strength and character, and the development of sour or rancid notes. An acidic note also may be detected. Surfactant systems experienced viscosity changes, solubility issues in clear systems, color changes and interactions with certified colors. Exposure to heat and UV light often exaggerated these problems.

Early investigators published many specific examples of the interaction of aroma chemicals with cosmetic bases. Pickthall¹² noticed the destabilizing effects of fragrance materials such as alcohols, phenols, acids and some esters. Hydroxycitronellal and α -terpineol are two prominent examples. Hydroxycitronellal contains two polar groups: hydroxyl and aldehydic. Its polarity enables it to compete for space on the surface of the micelle, leading to either a decrease in viscosity or a breaking of the emulsion. The α -terpineol has one hydroxyl group on a relatively rigid molecule, less disruptive than hydroxycitronellal but still

capable of destroying the balance of the emulsion. It also is possible for perfume ingredients to produce closer packing of

the hydrophobes on the micelle surface, in which case emulsion stability will improve.

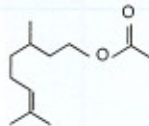
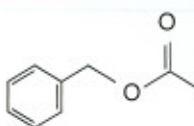
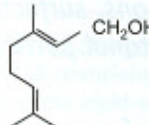
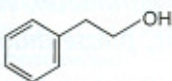
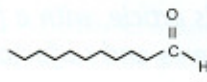
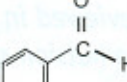
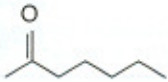
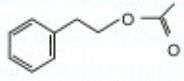

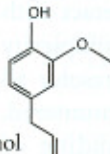
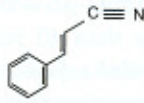
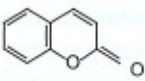
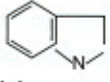
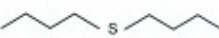
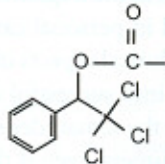
Chemical type	Examples		
Esters	 Geranyl acetate (aliphatic)	 Benzyl acetate (aromatic)	
Alcohols	 Geraniol	 Phenylethyl alcohol	
Aldehydes and ketones	 Aldehyde C-11 (ylid)	 Benzaldehyde	 Methyl n-amyl ketone
Acetates	 Phenylethyl acetate		
Phenols	 Phenol	 Eugenol	
Nitriles	 Cinnamyl nitrile		
Lactones	 Coumarin	A cyclic ester. γ or δ hydroxy acid forms 5- or 6-member ring by intramolecular esterification.	
Heterocyclics	 Indole		
Acids	CH_3COOH Acetic	$\text{C}_2\text{H}_5\text{COOH}$ Propionic	$\text{C}_3\text{H}_7\text{COOH}$ Butyric
Sulfur compounds	 Dibutyl sulfide		
Halogen compounds	 Rose crystals		

Figure 1. Some chemical types in perfumery

Karas¹³ studied TEA-stearate lotions. He observed immediate separation with phenylethyl alcohol and hydroxycitronellal, breaking after five days with linalool, amylcinnamic aldehyde, terpineol and methyl ionone. Methyl anthranilate showed discoloration and slight creaming.

Wellenkamp¹⁴ used acetals rather than the corresponding aldehydes to aid emulsion stability. He considered the acetals of phenyl acetaldehyde, phenyl propylaldehyde, cyclamen aldehyde, amyl cinnamic aldehyde, hydroxycitronellal and hydratropic aldehyde. The acetals were found to be longer-lasting, less harsh and more delicate than the aldehydes. Significantly, the acetals did not discolor soaps and creams.

In a 1980 paper,¹⁵ Bonadeo proposed K_d to explain the effect of fragrance on emulsions. The K_d is the water/dioxane partition coefficient. The aromatic material is considered part of the oil phase, and an HLB_f is assigned, corresponding to the required HLB commonly used as a guide for nonionic emulsification. Bonadeo proposed one additional concept: the critical limit of the water phase (CLWP), which quantifies the water that can be tolerated by the emulsifier system.

Bonadeo concluded that the olfactory properties of a fragrance in a w/o or o/w emulsion are the same if the system is properly balanced; otherwise, a mixed system is formed, upsetting the fragrance distribution. He firmly places the fragrance in the oil phase, becoming a determinant in calculating the total HLB , and he concluded that the CLWP of each perfume component contributes to the limit of water that can be tolerated.

A useful method for adjusting the perfume to compensate for the effect

of the substrate was presented by Saunders.¹⁶ The smell of a fragrance out of a bottle is different from the smell after it is applied to a substrate or put in a base. What is being smelled corresponds to the headspace composition. Saunders compared the headspace proportion of each component out of the bottle and in the air over the base, deriving a ratio for each material for use as a correction factor. The perfumer's formula then is adjusted by these correction factors, resulting in a new formula. The new formula's headspace analysis in the base should be the same as the headspace of the neat oil made from the original formula. This is illustrated in Table 1.

Polarity

It is clear that, with the obvious exception of the interaction of functional groups, the key to understanding the behavior of aroma chemicals in diverse media is polarity. The simplest division of materials is into two groups, polar or nonpolar, but either solubility parameters (SP) or the water/octanol partition coefficient (P) is much more informative. P usually is expressed as its calculated logarithm, $\log P$.

Solubility parameters: Vaughan has covered the personal care applications of SP in a valuable series of articles.¹⁷⁻²¹ The SP is a measure of all the cohesive forces in a molecule. Its consequence can easily be seen in the size of a drop. Using a uniform pipette, the weight of a drop of water, mineral oil or ethyl alcohol will be different, and the difference can be correlated directly to the SP of the materials.

In a simplified way, where aroma chemicals might partition in an emul-

sion system can be seen using the solubility parameter. Table 2 shows the solubility parameter of selected fragrance materials and some common emulsion ingredients.

The most common type of emulsion is o/w, with anionic or nonionic emulsifiers. The emulsion has an oil phase of small droplets covered by emulsifier molecules, an external phase and liquid crystal structures. These different phases must be kept in mind when examining the fate of fragrance in the system.

A simplified view of the location of aroma chemicals in an emulsion is shown in Figure 2 on page 63. Once the fragrance mixes into the emulsion, the individual components of the fragrance partition in varying

Table 2. Solubility parameters (SP) of selected fragrance materials and some common emulsion ingredients

Ingredient	SP
White mineral oil	7.09
Stearic acid	7.75
β -Pinene	8.03
Amyl acetate	8.44
Citronellal	8.83
Stearyl alcohol	8.90
Citral	9.34
Linalool	9.62
Amyl alcohol	10.84
Benzaldehyde	11.00
Eugenol	11.12
Dipropylene glycol	11.78
Phenylethyl alcohol	11.79
Benzyl alcohol	12.31
Vanillin	12.34
Propylene glycol	14.00
Water	23.40

Table 1. Analysis of headspace composition when a fragrance mixture is placed in a base (From Reference 16)

Fragrance component	a Headspace of fragrance	b Headspace over fragranced base	c a/b	d Original fragrance formula	e Adjusted fragrance formula
Limonene	31.9	15.9	2.0	2.0	4.0
Linalool	13.2	12.9	1.0	2.0	2.0
Linalyl acetate	12.3	7.7	1.6	2.0	3.3
PEA	6.2	22.6	0.3	3.5	1.1
Benzyl acetate	11.6	18.7	0.6	2.1	1.3

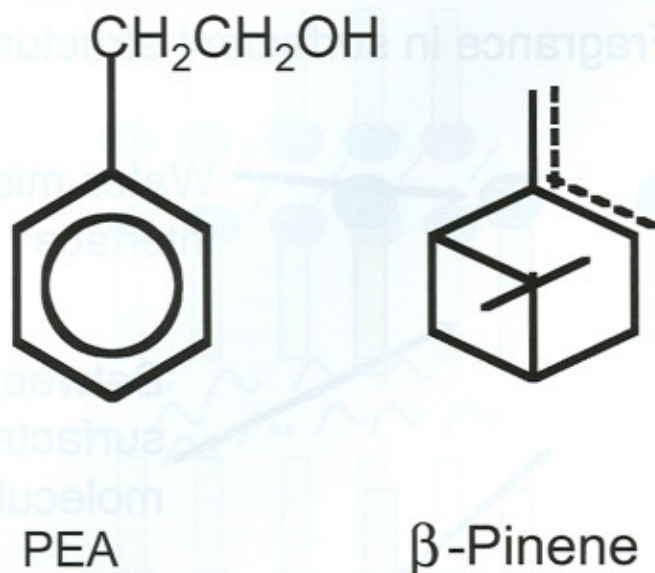


Figure 2. The location of aroma chemicals in an emulsion

VANILLIN

SMILES: COc1cc(C=O)ccc1O

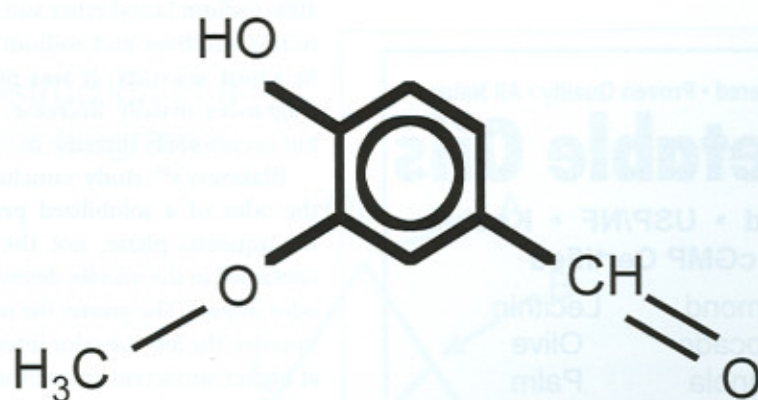


Figure 3. SMILES notation for vanillin

degrees into the different structures of the product. Pinene, with an SP under 9 and no oxygen groups, will migrate into the internal oil phase. Hydroxycitronellal, with its surface activity, can concentrate on the micelle surface; there it can displace some emulsifier, lower the viscosity or break the emulsion. Phenylethyl alcohol and vanillin, with SP greater than 11, can have substantial presence in the external aqueous phase. The amyl alcohol, with a fatty chain and hydroxyl group, can partition into the liquid crystal structure. The partitioning takes time, accounting for the aging necessary before one can accurately evaluate the effect of the base on the fragrance's olfactory performance.

Water/octanol partition coefficient: The water/octanol partition coefficient was conceived as a wet chemistry process, but the "calculated" coefficient in clogP refers to the fact that it now is more commonly determined by an algorithm of functional groups in a computer program. Because most chemical structures cannot be typed on a keyboard for computer entry, they first must be converted using programs such as the Simplified Molecular Input Line Entry System (SMILES).

In SMILES, case is important: aliphatic regions are upper case and aromatics are lower case. Hydrogen is not notated. Logically enough, a single bond is C-C and a double bond is C=C, but a triple bond requires special treatment, and is C#C. Rings are denoted by a number at the start and end: c1ccccc1Br is bromobenzene.

A more recent tool is the Graphical Input of SMILES, or GRINS. It allows

Table 3. ClogP analysis for vanillin, calculated at www.daylight.com/daycgi/clogp

Class	Type	Log P	Value
Fragment	#1	Ether	-0.610
Fragment	#2	Aldehyde	-0.420
Fragment	#3	Alcohol or hydroxy	-0.440
Carbon		1 aliphatic isolating carbon	0.195
Carbon		6 aromatic isolating carbons	0.780
ExFragment	Hydrog	6 hydrogens on isolating carbons	1.362
ExFragment	Bonds	1 chain and 0 alicyclic (net)	-0.120
Electronic	SigRho	4 potential interactions; 1.95 used	0.787
Ortho	Ring 1	1 normal ortho interaction	-0.250
Result	4/22+	All fragments measured	1.284

some ring structure to be inserted in a process similar to using a chemical drawing program. Figure 3 shows the SMILES notation and Table 3 shows a detailed analysis of the clogP for vanillin acquired from a Web-based calculator.²²

The clogP of fragrances preferred in specific applications are specified in many patents. A notable example is the Procter & Gamble patent²³ protecting the Febreze[®] cleaners. Although the inventors claim cyclodextrin as the principal odor control agent, they devote more space to specifying the preferred fragrance composition. The ideal fragrance will consist of a preponderance of hydrophilic materials, especially those with clogP of 3 or less. These aroma chemicals are most compatible with aqueous systems.

Fragrance in Surfactant Systems

The study of fragrance in surfactant systems has a long history. Munden²⁴ published a study on the effect of

** Febreze is a registered trademark of Procter & Gamble.*

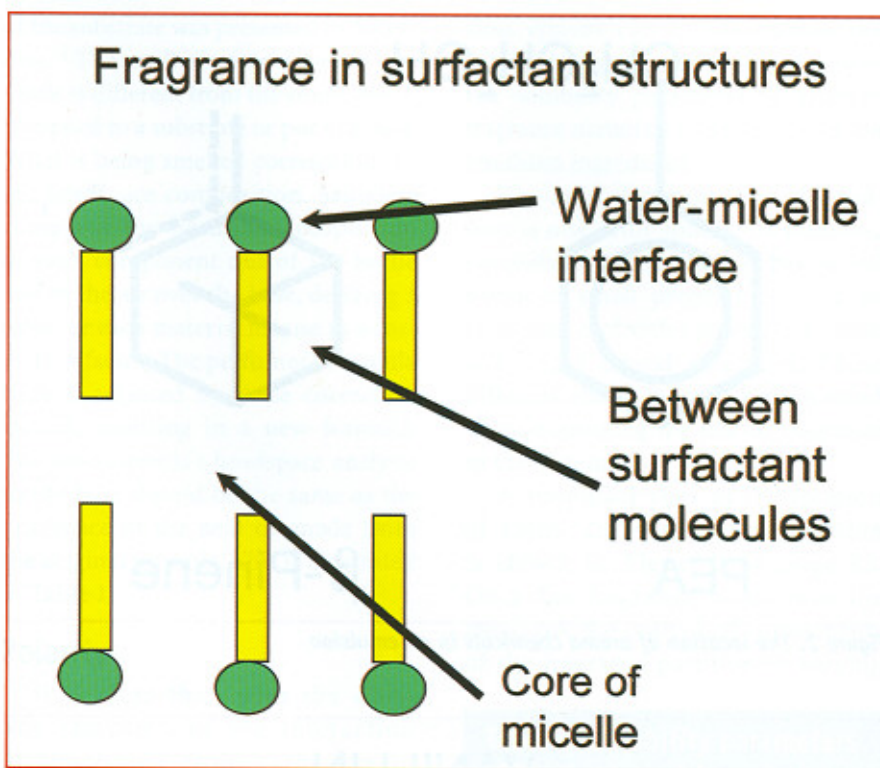


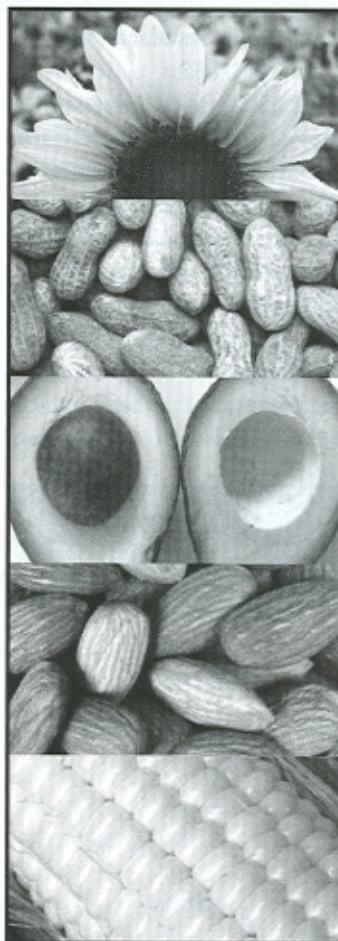
Figure 4. Partitioning of aroma chemicals in a surfactant system

fragrances on viscosity. He used a perfume consisting mainly of phenylethyl alcohol, lillial, citronellol, hexylcin-

amic aldehyde, methyl dihydrojasmonate and methyl cedryl ketone. The surfactant system consisted of 10% sodium lauryl ether sulfate, some minor additives and sodium chloride to adjust viscosity. It was noted that fragrances usually decrease viscosity, but occasionally increase it.

Blakeway's²⁵ study concluded that the odor of a solubilized perfume in the aqueous phase, not the fraction contained in the micelle, determined the odor impact. The greater the number of micelles, the less the odor intensity, and at higher surfactant concentrations the micelles must burst, releasing aroma chemicals into the solution.

Behan and Perring²⁶ used headspace analysis to examine the vapor phase concentration over a shampoo system. Sodium dodecyl sulfate (SDS) was used at a range of 5–20%, but with 10% as the standard level. Aroma chemicals from 0.5% to 3% were used, with 1% the standard. The aroma chemicals studied were octan-2-one, benzyl acetate, anethole, limonene and heptan-1-ol. Several solvents/solubilizers also were considered. It was found that the solvent could profoundly influence the headspace composition of the volatile components. The perfume partitions in the SDS/water system, and increasing the surfactant



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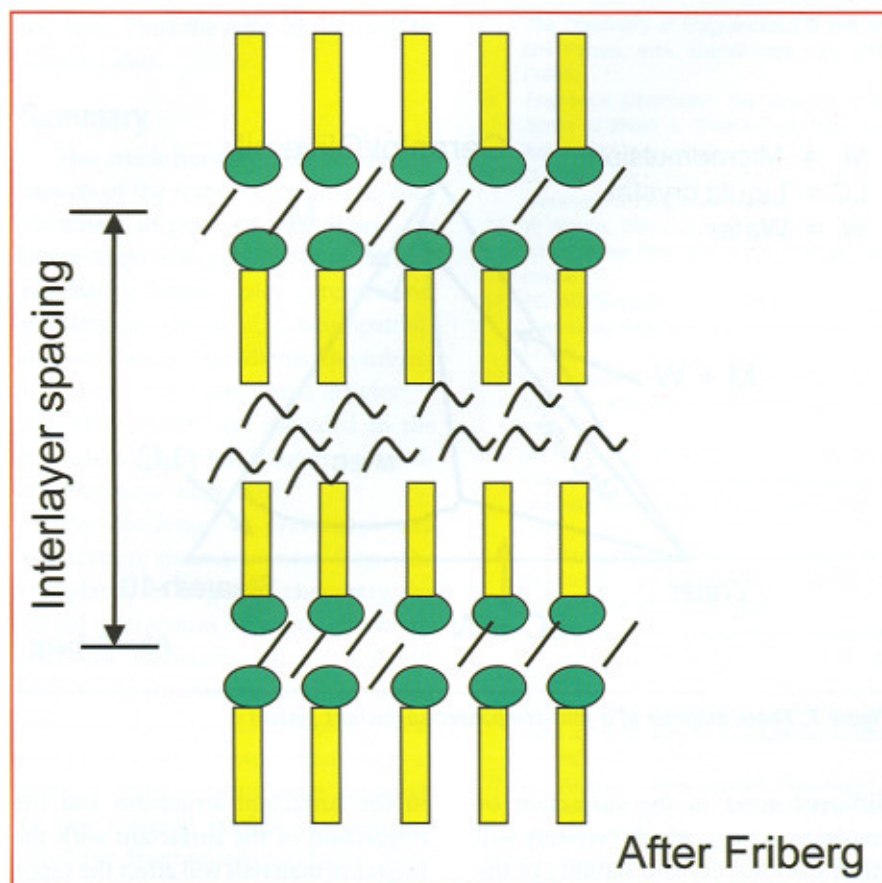


Figure 5. Liquid crystal structures in a surfactant system

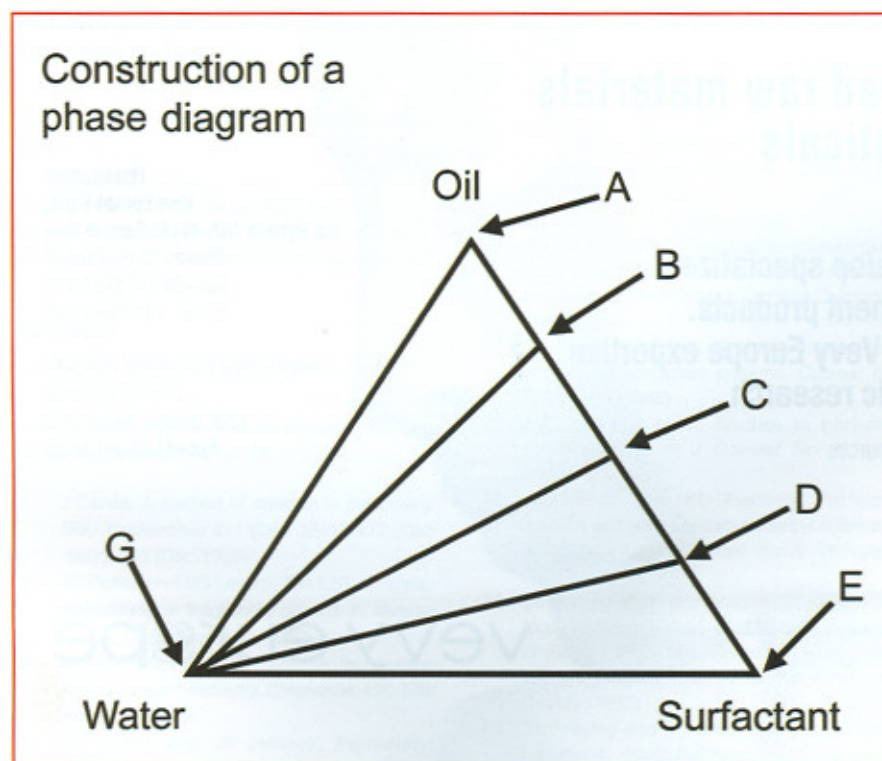


Figure 6. Construction of a phase diagram

concentration reduces the quantity of aroma chemicals in the headspace.

It is known that different perfume components could concentrate in dif-

ferent areas of the micelle. Additionally, some polar materials can be present in the external phase. Materials with some water solubility, such as phenylethyl

alcohol or vanillin, become less soluble if the polarity of the external phase is increased by the addition of salt. Systems that overly rely on salt to build viscosity may be hard to fragrance for two reasons: the low level of surfactant and the excessive polarity of the external phase.

The partitioning of aroma chemicals in a surfactant system is shown in Figure 4 on page 64. Some materials will migrate into the core of the micelle, some will align in the hydrophobic tails, some will be near the micelle surface and a small amount will be in the external aqueous phase. The viscosity is determined by the number, size and geometry of the micelles, in addition to any thickening that has been created in the external phase. The fragrance materials can change any of these parameters, and make the viscosity increase or decrease. The viscosity changes can be extreme and difficult to correct.

Knowledge of the structure of bases and the chemical nature of aroma chemicals allows intelligent conclusions to be drawn concerning the fate of fragrance materials in finished products, but it is not conclusive scientific proof. Research by a number of workers under the general guidance of Friberg²⁷⁻⁴² confirmed the nature of the interaction of fragrance chemicals with larger environments with the *a priori* observations of many formulators

It is impossible to examine every aspect of complex systems in a thorough way: the sheer number of variables make the calculations impractical. Three- or four-component systems of clearly defined composition must be used to provide manageable data. The methods used by Friberg involve titration combined with visual and polarized microscopic examination, and low angle X-ray diffraction. The X-ray diffraction measures the spacing of liquid crystal structures (Figure 5). The titrations yield phase diagrams that map the different structures present in the mixtures.

Changing the initial blend a number of times allows a complete analysis of the system (Figure 6). The line from A to B has no water content. Point A is 100% oil; B is 75% oil, 25% surfactant;

C is 50% oil, 50% surfactant; D is 25% oil, 75% surfactant; and E is 100% surfactant. Water is titrated into the system, providing data along the lines AG, BG, CG, DG, EG, where the point G is 100% water.

To add a fourth variable, a blend of two ingredients is fixed and the titration performed as in the three-phase system. **Figure 7** has a 50:50 mixture of geraniol and olive oil in a system with steareth-10 and water. Areas of lamellar liquid crystals and microemulsions are formed.

The microemulsion regions occur where the mixture becomes clear. Liquid crystals can be observed by a cross pattern in the micelles viewed in a polarizing microscope. The spacing between lamellar liquid crystals can be measured by low-angle X-ray diffraction, revealing quantitatively the impact of the fragrance molecules on the geometry of the system.

The cumulative work of Friberg's group showed conclusively that different fragrance molecules occupy

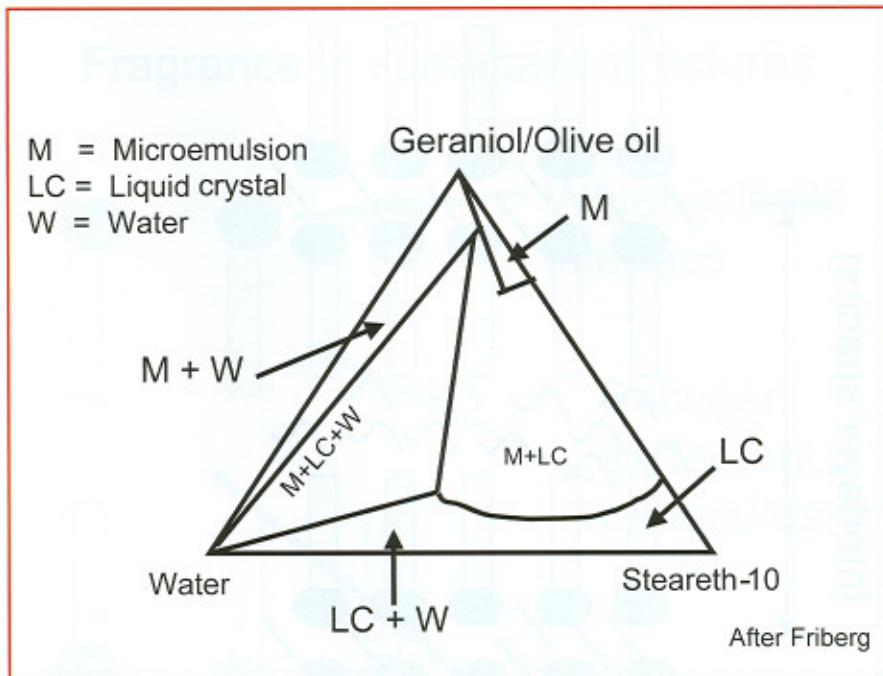


Figure 7. Phase diagram of a four-component surfactant system

different areas in the surfactant or emulsion system, which certainly will affect the viscosity and stability of the system. The location of the fragrance

in the surfactant structures and the interaction of the surfactant with the fragrance materials will affect the vapor pressure of the aroma chemicals in

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the base. Thus the odor impact will be affected also.

Summary

This article has only considered a few aspects of the importance of fragrance chemistry in personal care. There are interactions with packaging; safety and regulatory issues; color interactions; problems at extreme pHs; incompatibilities with active ingredients; the subject is endless. Some additional articles⁴³⁻⁵⁰ and Web sites⁵¹⁻⁵³ are included in the references to help guide interested readers, but many more exist.

The challenge to perfumers and application chemists arises from the complexity of fragrance chemistry, the varied interactions of aroma chemicals with the environment of emulsion and surfactant systems, and the added influence of external factors such as heat and light. Practical experience helps as a guide, and the brute force approach of raw material studies can produce effective fragranced products. True understanding comes with the realization that fragrances are chemicals, that bases create an environment of varied character, and that the core of fragrance chemistry is chemistry.

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Send e-mail to steve@stephen-herman.com.

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Personal Care Ingredients Asia: Innovating Answers

Personal Care Ingredients Asia's (PCIA) showcase of more than 5,000 raw materials and ingredients, married with the information and advice needed to formulate products, was a key focal point at the conference and exhibition, held March 7-9, 2006, in Seoul, Korea. More than 200 international manufacturers and suppliers of raw materials and ingredients participated in this year's exhibition, representing all the key areas of the market and offering innovations to shape the present and the future.

Color cosmetic formulations incorporating actives for beyond-color effects were launched by Sederma. Actives stirred the PCIA scene as several companies offered antiaging answers. A novel active from prickly pear was presented by Mibelle to hydrate and protect the skin from the environment. Cognis

highlighted its *Vigna aconitifolia* seed botanical active with effectiveness reportedly similar to retinol. Additionally, Vincience rolled out numerous peptide, botanical, biotech and marine ingredients for slimming, anti-stress/antiaging, skin lightening, anti-itch/anti-irritant and other skin care effects.

In skin care, "soft and mild" was the focus of Desert Whale Jojoba Company's natural exfoliants, based on jojoba esters "spheres" technology. Wacker showcased novel cyclodextrin complexes and silicone elastomer gels for skin care, and alkyl-modified silicone waxes for the formulation of shampoos and conditioners. Cognis

also added a multifunctional wax dispersion to the conditioning hair care offerings, among others.

Beyond education on the exhibition show floor, PCIA offered attendees a scientific program on key industry areas: hair care, active ingredients, antiaging, skin whitening and protection, delivery systems and novel formulating.



PCIA welcomed attendees with more than 200 exhibits and a three-day scientific conference of more than 30 paper presentations.

Locating Aroma Chemicals in a W/O Emulsion

Editor's note: Following is a corrected version of Figure 2 appearing on page 63 from Steve Herman's article in the April issue. C&T regrets the error.

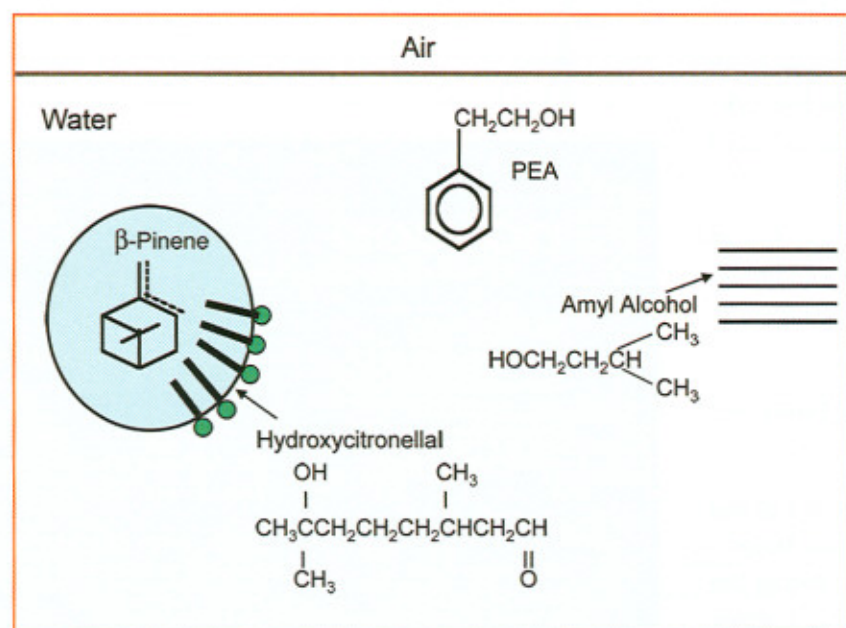


Figure 2. The location of aroma chemicals in an emulsion

A simplified view of the location of aroma chemicals in an emulsion is shown in **Figure 2**. Once the fragrance mixes into the emulsion, the individual components of the fragrance partition in varying degrees into the different structures of the product. Pinene, with an SP under 9 and no oxygen groups, will migrate into the internal oil phase. Hydroxycitronellal, with its surface activity, can concentrate on the micelle surface; there it can displace some emulsifier, lower the viscosity or break the emulsion. Phenylethyl alcohol and vanillin, with SP greater than 11, can have substantial presence in the external aqueous phase. The amyl alcohol, with a fatty chain and hydroxyl group, can partition into the liquid crystal structure. The partitioning takes time, accounting for the aging necessary before one can accurately evaluate the effect of the base on the fragrance's olfactory performance.