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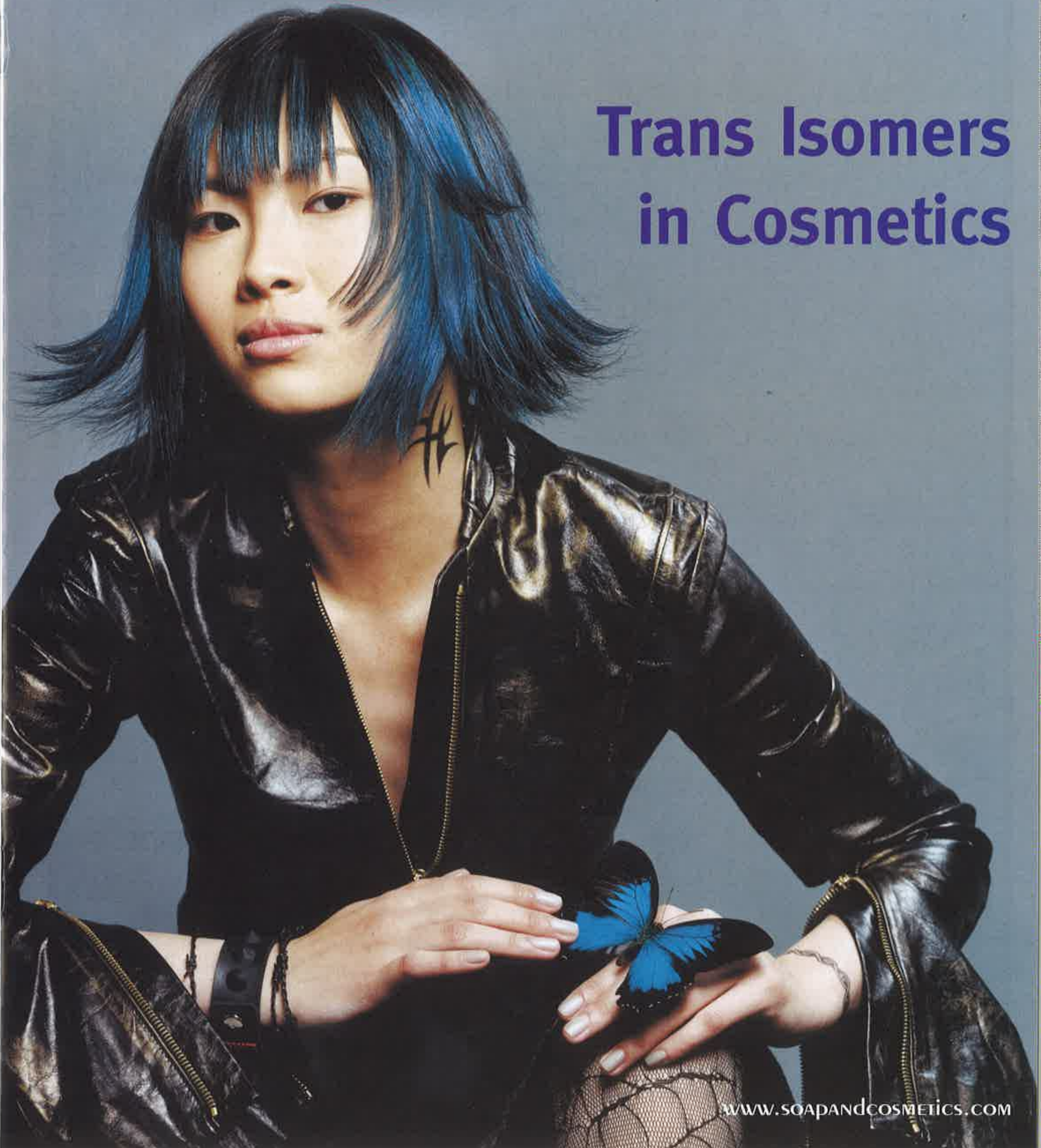
NEW IDEAS FOR SUCCESSFUL PRODUCT DEVELOPMENT



Trans Isomers in Cosmetics

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Trans Isomers in Cosmetics

BY JAMES BROWN AND ROBERT KLEIMAN

Cosmetic chemists may soon be called upon to identify the *trans* isomer content of their brand's formulations and to certify that their products are "*Trans* Free." The impetus behind the movement is in part due to consumer concerns that *trans* fatty acids are not healthful in their diets and will be undesirable to use on their body as well. This intuitive reaction is not without merit. *Trans* fatty acids have been implicated in the inhibition of Δ5 and Δ6 desaturation of polyunsaturated fatty acids; i.e., the metabolic pathway to prostaglandin formation. Prostaglandins are important mediators of skin metabolism. Topical introduction of *trans* fatty acids may disrupt normal prostaglandin formation.

The fire is also being stoked in the USA due to broad new FDA food labeling proposals (*World Food Chemical News*, November 24, 1999, Volume 6, Number 16) that would require the amount of *trans* fatty acids in a product to be listed on the "Nutrition Facts" panel of food labels. This *trans* fatty acid isomer phenomenon is the latest "food borne" trend to be transposed to cosmetics. Marketers will undoubtedly find a receptive audience for claims of "*Trans* Free" cosmetic and personal care products. The good news is that *trans* isomers are easy to detect, there are alternative routes to obtain "*Trans* Free" ingredients and naturally occurring *trans* isomers are rare.

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CIS AND *TRANS* GEOMETRY

The *cis* and *trans* labels refer to the geometric positioning of hydrogen atoms about points of unsaturation (double bonds) found in lipid chains. The *cis* isomer is simplistically represented by both hydrogen atoms of the carbon atoms forming the double bond being located on the same side of the carbon chain (See Figure 1). The *trans* form is depicted with hydrogen atoms appearing on opposite sides of the carbon chain (See Figure 2).

NATURE MAKES *CIS* FATTY ACIDS

Nature creates an overwhelming majority of *cis* fatty acids (and fatty alcohols) as components of lipid materials (emollients) such as vegetable oils, fats, wax esters, etc. *Trans* isomers of lipid materials occur infrequently in nature. Table 1 contains a listing of some of the relatively rare sources of naturally occurring *trans* fatty acid isomers. Non-natural *trans* isomers are usually formed when lipid materials are subjected to various chemical transformations such as partial hydrogenation, oxidation, transisomerization, or certain enzymatic reactions.

TRANS ISOMERS INHIBIT PROSTAGLANDIN SYNTHESIS

In vivo studies[1,2] using human skin cells have shown that some monoene *trans* fatty acids inhibit both Δ5 and Δ6 desaturation of fatty acids. This synthesis pathway in humans to important prostaglandins require that the essential fatty acids, linoleic and linolenic acids, be first Δ6 desaturated to γ-linolenic and stearidonic acids respectively. These two acids are then elongated to their C_{20} isomers. These isomers are then desaturated to their respective Δ5 isomers. These Δ5 fatty acids are used in prostaglandin formation.

CIS AND *TRANS* ISOMERS IN LIPIDS

Shani[3,4,5] studied the *cis* and *trans* isomers of jojoba oil and pointed out some differences in affinity of the two forms to silver ions, the naturally occurring *cis* form having somewhat more affinity. *Trans* isomers of lipid components are thermodynamically more stable and generally higher in melting point than the corresponding *cis* form. Wisniak[6] discusses different routes to achieve *trans* isomerization of jojoba oil and points out that partial hydrogenation and transisomerization of jojoba oil results in the formation of some *trans* fatty acid and fatty alcohol isomers.

Trans forms of unsaturated lipid materials can also be intentionally created through the use of a nitrous or selenium oxide catalyst and have

Figure 1. *cis* Double Bond

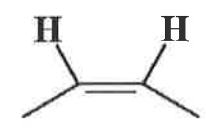
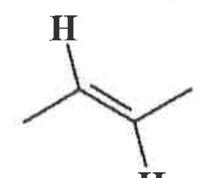


Figure 2. *trans* Double Bond



commercial application as intermediates in various chemical reactions. *Trans* isomers of jojoba oil have been made (Jojoba Butter) by using heat in the presence of acidic bentonite clays[7]. Partial hydrogenation of vegetable oils and other lipid materials is, however, by far the most likely source of *trans* fatty acid isomers that subsequently enter the food and cosmetic chain of distribution.

Fully hydrogenated lipid materials contain neither *cis* nor *trans* fatty acid isomers. The double bonds of a fully hydrogenated lipid have been eliminated by completely "saturating" the molecule with hydrogen. This full hydrogenation reaction makes the carbon chain less active as a site for further reaction. A fully hydrogenated lipid material has a higher melting point and is more resistant to free radical formation (See Figure 3). Fully hydrogenated forms are also the most oxidatively stable of all possible chemical structures of lipids. Fully hydrogenated jojoba oil (melting point 68-70°C) contains neither *cis* nor *trans* isomers (there are no remaining double bonds in the molecule) and is frequently found as a component of lipsticks, mascara, eye shadow and other "wax matrix" type formulations.

Partial hydrogenation of lipid materials is routinely employed as a commercial means of incrementally increasing the oxidative stability of a lipid, increasing the melting point of the material and also as a means of altering the polarity of the material at the double bond site. "Brush hydrogenation" or "selective hydrogenation" or "selective saturation" are partial hydrogenation reactions often used as a means of "selectively" decreasing the polyunsaturated content of a lipid. These procedures create a higher melting fraction in the lipid, which can then be subsequently removed through a winterization or other fractionation process. "Brush hydrogenation" and "selective" hydrogenation or saturation reactions create unwanted *trans* isomers in lipids.

TRANS FATTY ACID ISOMERS IN COSMETICS

The CTFA List of Japanese Cosmetic Ingredients, 4th Edition (1999) lists seven "Partially Hydrogenated" product categories. These are Horse Oil, Jojoba Oil, Methyl Abietate, Palm Oil Fatty Acid, Perilla Oil, Squalene and Tallow Acid. Where INCI names for these lipids have been assigned in the USA, those INCI names do not include a "Partially Hydrogenated" category as in Japan. Formulators should be aware that "partially hydrogenated" and "selectively hydrogenated/saturated" products containing *trans* isomers are sometimes offered to the trade under the INCI names of their fully hydrogenated analogs.

A search of the CTFA Dictionary for "partially hydrogenated" revealed two products; Pentahydrosqualene and Triethylene glycol hydrogenated rosinate. The CTFA Ingredient Dictionary also includes a listing for "Jojoba Butter," a trans-isomerized form of jojoba oil containing about 50% *trans* isomers.

ALTERNATIVES TO *TRANS* ISOMERS IN COSMETIC PRODUCTS

The challenge of cosmetic ingredient suppliers is therefore to provide manufacturers with oxidatively stable and affordable lipid based emollients that exhibit a broad range of melting points yet which contain no *trans* fatty isomers. Oil phase emollients with these desirable characteristics can be obtained without the formation of *trans* isomers through a process known as "interesterification." Rozenaal[8] discussed use of this interesterification process to modify the melting characteristics of triglycerides using various catalysts including enzymes. Another paper by Haumann[9] points out that the interesterification process may be a means of addressing the growing consumer concern for *trans* fatty acids.

Trans isomers of fatty acids that inhibit prostaglandin formation are rare in nature but can be found in partially (selectively) hydrogenated, brush hydrogenated or transisomerized lipid materials. Consumer awareness of the anti-nutritional effects of *trans* isomers in food products will lead to a heightened awareness of these compounds in cosmetics. *Trans* isomer presence in cosmetic ingredients is easy to detect, although often obscured due to improper, incomplete or misguided labeling by ingredient suppliers. Formulators should request certification from ingredient suppliers that confirm the absence of *trans* isomers.

GLOSSARY OF TERMS

Lipids: Biomolecules insoluble in water that can be extracted from cells by high pressure or by organic solvents of low polarity such as hexane, ether, or chloroform. Examples of lipids are mono, di and triglycerides, wax esters, sebum, lecithin and phospholipids.

Fatty Acids: Generally, straight chain compounds, from three to twenty-four carbon atoms in chain length, having a terminal carboxyl group. These compounds make up the bulk of fats and oils and are important in skin nutrition.

Fatty Alcohols: Generally, straight chain compounds, from three to twenty-four carbon atoms in chain length, having a terminal hydroxyl group.

Trans Fatty Acids (Alcohols): Unsaturated fatty acids (alcohols) in which the hydrogens of the double bonds are on opposite sides of the molecule chain.

Cis Fatty Acids (Alcohols): Unsaturated fatty acids (alcohols) in which the hydrogens of the double bonds are on the same side of the molecule chain.

Hydrogenation: The addition of hydrogen atoms or molecules to unsaturated carbon chains (carbon chains with double bonds).

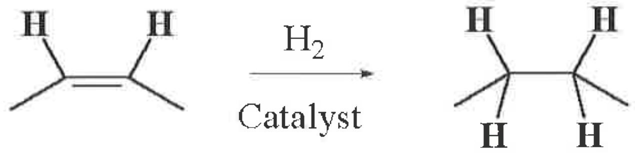
Saturation: Adding the maximum number of hydrogen atoms to an unsaturated bond. In the case of a double bond it is the addition of two hydrogen atoms, i.e., H_2.

Partial Hydrogenation: Incomplete addition of hydrogen to an unsaturated material leaving some double bonds not saturated, also known as "selective saturation" or "selective hydrogenation" or "brush hydrogenation." Partial, brush or selective hydrogenates (saturates) contain unwanted *trans* isomers.

Interesterification: The catalyzed chemical reaction between two or more esters in which the acids and alcohols present are recombined in a random manner, also known as transesterification. This reaction does not alter the double bond geometry of unsaturated compounds, i.e., no *trans* isomers are formed as a result of interesterification reactions.

Transisomerization: The catalyzed chemical reaction converting natural *cis* double bonds to *trans* isomers. Transisomerization creates unwanted *trans* isomers.

Figure 3. Hydrogenation of Double Bonds



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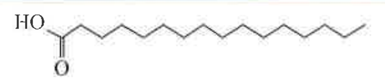
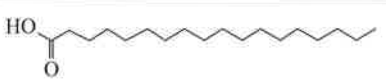
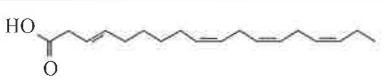
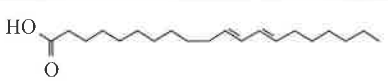
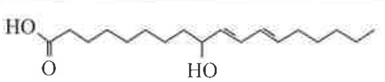
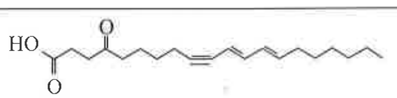
Acid	Structure	Source
trans-3-Hexadecenoic		Aster sp. Hopkins et. al, Lipids (1966)
trans-3-Octadecenoic		Grindelia oxylepis Kleiman et. al, Lipids (1966)
trans-3,cis-9,cis-12,cis-15-Octadecatetraenoic		Tecoma stans, Hopkins et. al, J. Chem. Soc. (1965)
cis-9,trans-11,trans-13-Octadecatrienoic (a-Eleostearic)		Aleuites fordii (tung oil), Chrombie et. al, J. Chem. Soc (1957).
9-hydroxy,trans-10,trans-12-Octadecadienoic (Dimorphecolic)		Dimorphetheca sinuata, Smith et. al, J. Am Chem. Soc. (1960)
4-keto,cis-9,trans-11,trans-13-Octadecatrienoic (Licanic)		Licania rigida (oiticica oil), Brown, Biochem. J. (1935)
9-yne,trans-11-octadecenynoic (Ximenynic)		Ximenia sp., Ligthelm et. al, J. Chem. Soc. (1952)

Table 1. Sources of Naturally Occurring *Trans* Fatty Acid Isomers

Trans Isomers in Cosmetics

Part 2

BY JAMES BROWN AND ROBERT KLEIMAN

In Part 1 of this series[1] the authors point out the growing consumer awareness of *trans* fatty acids due to their antinutritional effect in food products. New FDA labeling law proposals[2] for foods containing these lipid forms will draw further attention to *trans* isomers. Earlier research[3,4] has revealed that some monoene *trans* fatty acids inhibit the Δ5 and Δ6 desaturation of polyunsaturated fatty acids; i.e., the metabolic pathway to important prostaglandin formation. Cosmetic industry marketers will undoubtedly find a receptive audience for "*Trans* free" product claims in the future.

MOTHER NATURE CREATES "*CIS*" ISOMERS

The majority of all lipids produced by nature contain only "*cis*" isomers. *Trans* isomers are produced when natural lipids (emollients) are manipulated through partial hydrogenation, selective saturation or transisomerization reactions conducted to alter the physical characteristics of the lipid.

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INTERESTERIFICATION PRODUCES NO *TRANS* ISOMERS

The interesterification reaction used to produce *trans* free emollients is one in which a fully hydrogenated lipid material (no *trans* isomers) is reacted with neat oil containing only *cis* isomers. The resulting product is an isomorphous mixture of partially saturated, saturated and unsaturated esters that contain no *trans* isomers. This route to produce "*Trans* Free" oxidatively stable emollients with a broad range of melting characteristics is considerably less practiced and more expensive than the partial (or selective) hydrogenation, selective saturation or transisomerization reaction, all of which produce unwanted *trans* isomers.

JOJOBA ESTERS: AN INTERESTERIFICATION MODEL FOR "*TRANS* FREE" LIPID EMOLLIENTS

Jojoba Esters are an ideal model to use to demonstrate the *trans* free interesterification reaction and the properties of the resulting components. The CTFA Ingredient Dictionary defines Jojoba Esters as: A complex mixture of esters produced by the transesterification/interesterification of Simmondsia Chinensis (Jojoba) Oil, Hydrogenated Jojoba Oil, or a transesterified mixture of the two. Figure 4 represents the interesterification reaction between (I) jojoba oil (only *cis* isomers) and (II) fully hydrogenated jojoba oil (no *cis* or *trans*isomers).

This interesterification reaction is similar to a game of lipid "musical chairs". In this reaction the ester linkages between the jojoba acid and the jojoba alcohol in both reactants are unbuckled, liberating the saturated and unsaturated jojoba alcohols and acids so that they move about freely (and separately) in the reaction vessel. When the music stops (the reaction is complete) the meandering alcohols must find a free acid "partner". In a completely random manner some old partners find each other again (Randomized Ia with Ib and randomized IIa with IIb). At the same instant some new bonds are formed as saturated alcohols link with unsaturated acids (IIb with Ia), and unsaturated alcohols link with saturated acids (Ib with IIa). The result is a statistically randomized and isomorphous mixture of Jojoba Esters, partially saturated Jojoba Esters and fully saturated Jojoba

Esters. No *trans* isomers are formed during this interesterification reaction. Table 2 illustrates the range of properties achievable through this interesterification process that is used to produce "*Trans* Free" Jojoba Esters.

LABELING INCONSISTENCIES

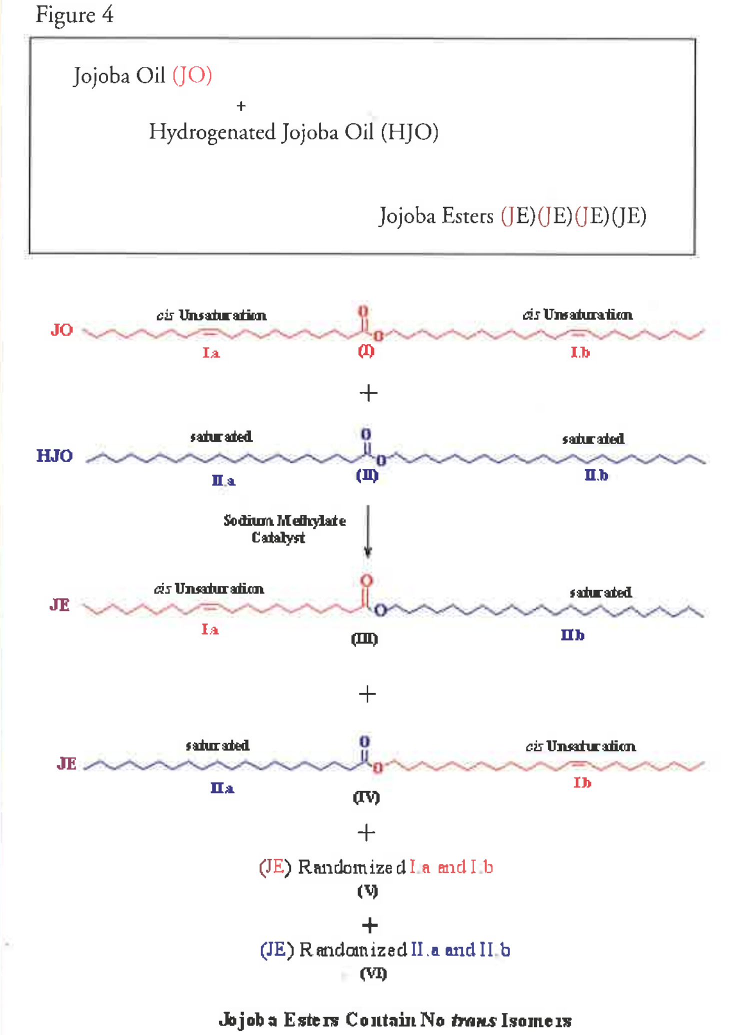
"Jojoba Esters" have been registered for use in Japan under the existing Japanese Cosmetic Ingredient Codex (JCIC) category of "Partially Hydrogenated Jojoba Oil" even though these Jojoba Esters contain no *trans* isomers. Conversely, some products are offered in US, Asian and European markets containing *trans* isomers while bearing the INCI name "Hydrogenated Jojoba Oil". These mislabeled products are in fact Partially Hydrogenated (or Selectively Saturated) jojoba oil. Other suppliers offer simple mixtures of Hydrogenated jojoba oil and jojoba oil and unknowingly mislabel these blends as "Jojoba Esters". Jojoba Butter containing about 50% *trans* isomers is offered to the trade under the "Jojoba Esters" INCI designation although the product is said to be produced by "transisomerization" and does not conform to the INCI definition for Jojoba Esters. *The message to formulators is to either conduct simple tests to determine the absence of trans isomers and/or request appropriate certification from suppliers.*

TRANS ISOMER ANALYSIS

It is possible to determine if a lipid material contains *trans* isomers through a number of different analytical methods.

The fact that *trans* isomers absorb infrared light at 900 to 1000 µM is routinely used to quantify the presence (or absence) of these isomers. Partially hydrogenated jojoba oil can be shown to have *trans* isomers as evidenced by the IR Spectrum absorption peak highlighted in figure 5. The same IR Spectrum was determined for Jojoba Esters 60 and the lack of an absorption peak in figure 6 at the 900 to 1000 µM range is proof that *trans* isomers are not present.

The Modified Chatillon Device measures the slip index.



"FEEL" AND "SLIP" OF JOJOBA DERIVATIVES

The tactile properties of partially hydrogenated jojoba oil (with *trans*-isomers) differ considerably from those of Jojoba Esters (*cis* only isomers), both having the same melting point. The *trans* containing partially hydrogenated isomers result in a more "grainy" and "rough" texture when applied to the skin as compared to the isomorphous and more elegant "dry emollient" characteristic of randomized and *trans* free Jojoba Esters. To demonstrate this difference in skin feel an expert panel of thirteen individuals were asked to simultaneously apply and rub in single 0.25 gram quantities of Jojoba Esters and Partially Hydrogenated Jojoba Oil on the volar forearm area. The materials tested both exhibited melting points of 59°C. Panelists were asked to complete a questionnaire that required the panelist to select a rating for the "skin feel" of the sample during a) application; b) rub in; and c) five minutes after rub in. The rating scale was 1-5, with the higher rating indicating a "smoother" skin feel. The results of the testing indicated that an overwhelming majority of all panelists considered Jojoba Esters to be "smoother" when applied and rubbed into the skin as compared to Partially Hydrogenated Jojoba Oil (containing *trans*-isomers). The panelists felt that the Partially Hydrogenated Jojoba Oil had a much "greasier" feel than the corresponding "*trans* free" Jojoba Ester.

TABLE 2. CHEMICAL AND PHYSICAL PROPERTIES OF JOJOBA ESTERS

Jojoba Ester	Melting Point °C	Iodine Value g I_2/100g	Oxidative Stability Value, Hours	Lubricity Degrees	Penetration, Mm	Monoene, Content, 0%
15	10-15	78-85	70	19.0	—	0
20	42-48	64-70	130	20.4	—	25-35
30	47-51	57-61	165	18.9	26.4	36-46
60	56-61	40-44	225	17.9	9.3	42-52
70	66-70	0-2	680	15.9	0.3	0

A further indication of the tactile differences in a lipid material containing either cis or *trans* isomers was quantified by measuring the "slip" of Jojoba Esters vs. Partially Hydrogenated Jojoba Oil according to the method of Cadicamo[5]. The procedure was modified by substitution of filter paper for the "membrane" used by Cadicamo and by use of a modified lipstick breakage meter (Chatillon DFM-10) to gradually increase the incline of the plane upon which the "slip" was being determined.

TABLE 4. PHYSICAL PROPERTIES OF INTERESTERIFIED JOJOBA ESTERS IN COMPARISON WITH LIKE MELTING PARTIAL HYDROGENATES

Sample	Melting Point °C	Iodine Value g I_2/100g of Sample	% *Trans*	Oxidative Stability Index, Hours	Index of Refraction μ_D@80°C	Penetrometer, Mm	Slip, Cadicamo, Degress (1%)	Slip, IFT, Degress (100%)
Jojoba Esters 30	49	58	0	165	1.4418	26.4	18.9	23
Jojoba oil Partial Hydrogenate	49	69	11	27	1.4435	—	19.3	>25
Jojoba Esters 60	59	42	0	225	1.4403	9.3	17.9	14.1
Jojaba Oil Partial Hydrogenate	59	49	37	108	1.4408	5.2	18.1	15.4

In the test Jojoba Esters 30 and 60 (no *trans*) and two Partially Hydrogenated Jojoba Oil (with *trans*) samples with identical melting points were examined.

To perform a slip test on a material in question using the Cadicamo method, a mixture of 99% lubricity base (10% myristyl lactate, 30% isopropyl myristate and 60% oleyl alcohol) and 1% test material is used. A 41 mm disc made from Fisher P2 filter paper is soaked in the test solution for at least 5 minutes. After the soak, the disc is removed from the solution and the excess solution is allowed to drip off until only a thin layer of test material remains on the disc. The glass incline plane is set to a very low angle and the soaked disc is placed on the top of the incline plane. The 200g weight is placed on top of the soaked disc and allowed to sit in this position for at least one minute. The instrument is set so that the end of the incline plane will increase in height at a constant rate in each trial. The movement of the incline plane is stopped at the exact moment that the weight and disc begin to slide on the inclined glass plane and the angle of the plane is reported. The Floratech slip test method is very similar to the Cadicamo method. The only difference is that in the method, the lubricity base is not used. Every material is tested at 100% concentration. In the case of room temperature solid materials, the solid is melted and the filter paper disc is soaked in the melted material. Remove the paper and let the excess material drip off. The

(Continued on page 50)

TABLE 3. AOCS METHODS USED TO DETERMINE *TRANS* UNSATURATION

AOCS Official Method	Title
Ce 1c-89	*trans* and *cis,cis* Isomers by GLC
Cd 14b-93	*trans* Composition of Partially Hydrogenated Oils by GLC-IR
Cd 14-61	*trans* Isomers by Infrared
Cd 17-85	*trans* Unsaturation in Margarine
Cd 14c-94	*trans* Isomers by Capillary GLC

Figure 5 IR of partially hydrogenated jojoba oil with *trans* absorption peak

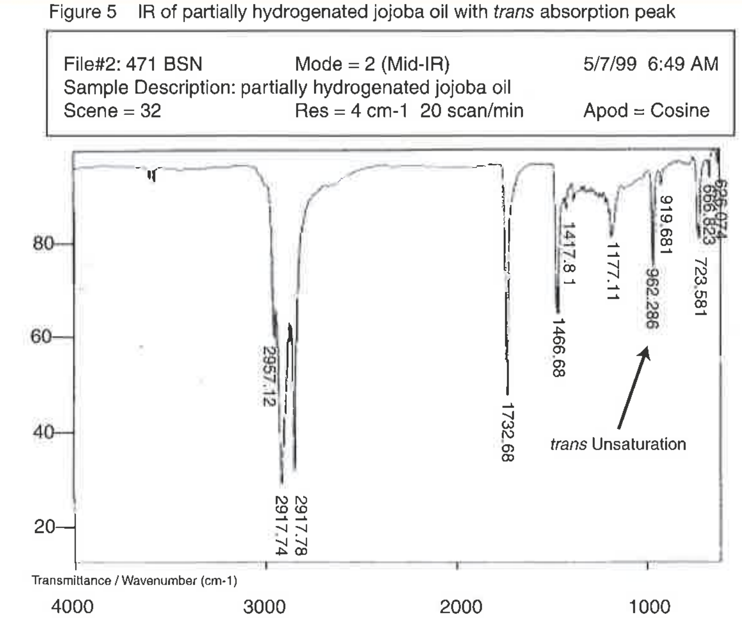
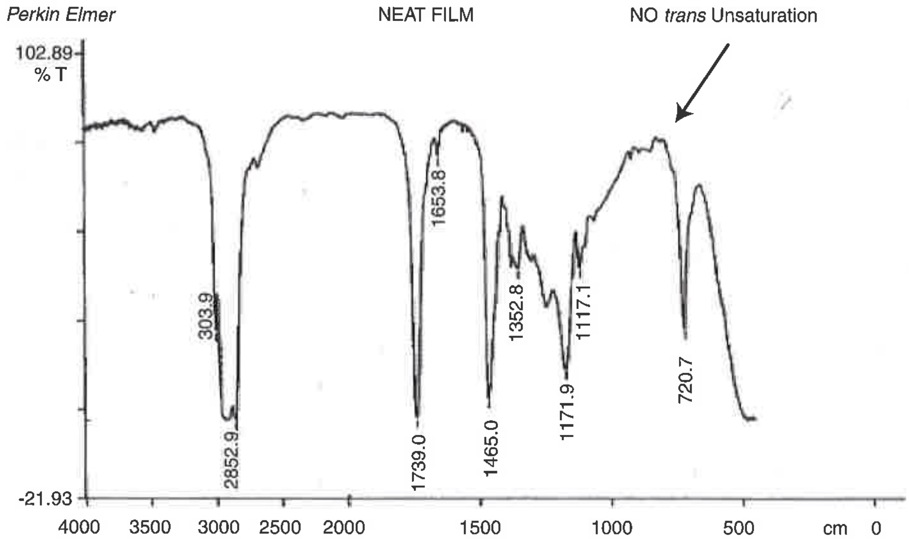


Figure 6



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remaining material will solidify on the disc in a uniform thin layer. The disc may then be tested under the same procedure shown above. However, a new disc must be used each time since the material 'permanently' coats the discs.

The Cadicamo slip test method was used to compare Jojoba Esters 60 to its partially hydrogenated jojoba oil analog, and Jojoba Esters 30 to its partially hydrogenated jojoba oil analog (Table 4). In each case the Jojoba Ester had better lubricating properties (smaller slip angle). Therefore, it can be concluded that, in general, Jojoba Ester samples have better slip properties than partially hydrogenated jojoba oil samples.

CHEMICAL PROPERTIES

Iodine Value (IV) and Refractive Index (RI) of lipid materials are two measures commonly used to follow the progress of commercial hydrogenation reactions. Fully Hydrogenated Jojoba Oil exhibits an IV of less than 2 and an RI of 1.4368 @ 80°C. Iodine Values for Hydrogenated Jojoba Oil in this range indicate that the double bond positions of the molecule comprising jojoba oil have been completely filled or "saturated" with hydrogen. The melting point of this Fully Hydrogenated Jojoba Oil is 68-70°C and it contains neither *cis* nor *trans* isomers.

In addition to the differences in skin feel and slip, the *trans* free Jojoba Ester will exhibit a lower IV than its *trans* analog when both are at the same melting point. This phenomenon is due to the *trans* isomer being thermodynamically more stable and therefore requiring a higher temperature to melt than the *cis* form of a Jojoba Ester with an identical IV. This phenomenon and other chemical and physical properties are further demonstrated in Table 4 where Jojoba Esters are compared to partially hydrogenated jojoba derivatives exhibiting identical melting points.

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