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Refractive index matching and clear emulsions

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Synopsis

Refractive index (RI) matching is a unique way of making clear emulsions to meet market trends. However, RI matching has not been sufficiently investigated in terms of physical principles and methodologies. Snell's law ($n_2 \sin r_2 = n_1 \sin r_1$) is applicable to cosmetic emulsions. When oil phase and water phase have equal RI ($n_2 = n_1$) values, light will not bend as it strikes obliquely at the emulsion interface. Instead, light is transmitted through the emulsion without refraction, which produces clarity. Theoretical RI values in solution can be calculated with summation of the product of the weight percentage and refractive index of each ingredient ($RI_{mix} = [W_1 \times n_1 + W_2 \times n_2 + W_3 \times n_3 + \cdots + W_n \times n_n]/W_\tau$). Oil-phase RI values are normally at 1.4 or higher. Glycols are used to adjust the water phase RI, since they typically have larger RI values than water. Noticeable deviations from calculated RI values are seen in experimentally prepared solutions. Three basic deviation types are observed: negative, positive, and slightly negative or positive, which can occur in glycol aqueous solutions at different concentrations. The deviations are attributed to changes in molecular interaction between molecules in solution, which can lead to changes in specific gravity. Negative RI deviation corresponds to a decrease in specific gravity, and positive RI deviation corresponds to an increase in specific gravity. RI values will deviate from calculated values since an increase or decrease in specific gravity leads to a change in optical density.

INTRODUCTION

Product clarity remains an important aspect in cosmetic formulation as it continues to be a mainstay in current consumer preference. To meet this requirement, chemists have developed two ways of making clear emulsions: via microemulsions (1) and refractive index matching (2). Microemulsions have been thoroughly investigated, leading to many microemulsion-conditioning products in the market. The crucial principle behind microemulsion clarity is the size of oil droplets. Microemulsions employ large concentrations of emulsifiers that compete for limited oil (emollient) ingredients. This ensures small oil-drop particle size (smaller than the wavelength of light) so that light can pass through the product without refraction. However, since large quantities of emulsifiers can lead to skin irritation, this technology is somewhat limited to hair care.

Karasssik *et al.* (3) at the Gillette Company disclosed the propriety technology of clear deodorant or antiperspirant gels that contain water-in-silicone oil emulsion. Deodorant

or antiperspirant emulsion gels exhibit reduced staining while retaining excellent aesthetic attributes and efficacy. The water phase comprises about 75% to 90% of the composition and contains a deodorant or antiperspirant effective amount dissolved therein. The oil phase comprises about 10% to 25% of the composition and contains a silicone oil and silicone emulsifiers. Even though it used refractive index matching methodology in the proprietary disclosure, there is no discussion of detailed physical principles, refractive index, or methodology discussion in the patent.

Although refractive index matching is used in the development of cosmetic products, it is mainly a process of trial and error. There is no practical methodology to follow in the discovery of new applications because the physical principle is not well explained.

In our exploration of refractive index matching in the formulation of cosmetics, detailed physical principles are revealed and a practical method is developed leading to a series of unique formulations (4). Refractive index matching enables chemists to make formulas that cannot be achieved by other methods. Refractive index matching should become a common technique for formulation chemists.

EXPERIMENTAL

MATERIALS

Trade names of materials used in this study are as follows: glycereth-7 (Liponic EG-7®, Lipo Chemicals); glycereth-26 (Liponic EG-1®, Lipo Chemicals); PEG-4 (Carbowax PEG 200®, Union Carbide); PEG-6 (Carbowax PEG 300®, Union Carbide); PPG-9 (Polyglycol P-425®, Dow Chemical); PVP/VA copolymer (Luviskol VA 73W®, BASF AG); PVP (Luviskol K30®, BASF AG); cyclomethicone and dimethicone (DC 1501®, Dow Corning); cyclomethicone (Rhodorsil 45V5®, Rhodia); cyclomethicone, phenyltrimethicone, and dimethicone (Gelaid 5565®, Chemsil); cyclomethicone and dimethicone copolyol (DC 5225®, Dow Corning); polyacrylamide, C13-14 isoparaffin, and laureth-7. (Sepigel 305®, Seppic); sodium acrylate/acryloyldimethyl taurate copolymer, isohexadecane, and polysorbate 80 (Simugel EG®, Seppic); hydroxyethylacrylate/sodium acryloyldimethyl taurate copolymer, squalane, and polysorbate 60 (Simugel NS®, Seppic); C13-14 isoparaffin (Isopar M®, Exxon Mobil Chemical); and C11-13 isoparaffin (Isopar L®, Exxon Mobil Chemical).

METHODS

Refractive indices (n) were measured with an Atago hand refractometer at 25°C under florescent light. Specific gravities were measured at 25°C with a stainless pycnometer.

PHYSICAL PRINCIPLES

OPTICAL PROPERTIES

Consider a beam of light (monochromatic) transmitted through air and directed onto the surface of a body of water (Figure 1). Some of the light is reflected at the interface

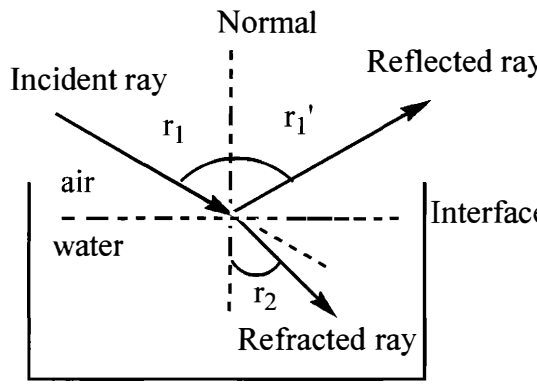

Figure 1. A ray diagram of refraction and reflection showing the angle of incidence, the angle of reflection, and the angle of refraction.

between the air and water; the remainder enters the water and is transmitted through it. The light is represented with an incident ray, a reflected ray, and a refracted ray. Each ray is oriented with respect to a line called the normal, which is perpendicular to the surface at the point of reflection and refraction. The angle of incidence is r_1, the angle of reflection is r_1', and the angle of refraction is r_2, all measured relative to the normal as shown.

Every transparent material has a property called optical density, which is an inverse measure of the speed of light through the material. Because water has a higher optical density than air, the speed of light is reduced as the light enters the water. The beam of light changes direction abruptly as it enters the water because of the change in speed. This bending of the light ray is called optical refraction.

Reflection and refraction are confirmed experimentally to obey two laws: the law of reflection and the law of refraction (5). The law of reflection states that a reflected ray lies in the plane of incidence and has an angle of reflection equal to the angle of incidence. This means that

$$r_1' = r_1$$

The law of refraction states that a refracted ray lies in the plane of incidence and has an angle of refraction related to the angle of incidence by

$$n_2 \sin r_2 = n_1 \sin r_1$$

Each of the symbols n_2 and n_1 is a dimensionless constant called the index of refraction or the refractive index (RI). It is defined as the ratio of the speed of light in a vacuum to its speed in a substance ($n = c/v$, where v is the speed of light in that substance and c is the speed of light in a vacuum). The equation was designated as Snell's Law. The index of refraction of a homogeneous substance is a constant and definite physical property. Consequently, the refractive index can be used to identify a substance, to measure its purity, and to determine the concentration of one substance dissolved in another. Typically, a refractometer is used to determine the refractive index. Some indices of refraction for common cosmetic ingredients are listed in Table I.

Table I
Refractive Indices (n) of Some Selected Cosmetic Ingredients

Ingredient	n
Water, deionized	1.3330
Glycerin	1.4680
Hexylene glycol	1.4276
Butylene glycol	1.4401
Propylene glycol	1.4355
Glycereth-7 (Liponic EG-7, Lipo Chemicals)	1.4720
Glycereth-26 (Liponic EG-1, Lipo Chemicals)	1.4690
PEG-4 (Carbowax PEG 200, Union Carbide)	1.4582
PEG-6 (Carbowax PEG 300, Union Carbide)	1.4615
PPG-9 (Polyglycol P-425, Dow Chemical)	1.4455
PVP/VA copolymer (Luviskol VA 73W, BASF AG)	1.4275
PVP (Luviskol K30, BASF AG)	1.3805
Cyclomethicone and dimethicone (DC 1501, Dow Corning)	1.3972
Cyclomethicone (Rhodorsil 45V5, Rhodia)	1.3960
Cyclomethicone, phenyltrimethicone, and dimethicone (Gelaid 5565, Chemsil)	1.4015
Cyclomethicone and dimethicone copolyol (DC 5225, Dow Corning)	1.3975
Polyacrylamide, C13-14 isoparaffin, and laureth-7 (Sepigel 305, Seppic)	1.4460
Sodium acrylate/acryloyldimethyl taurate copolymer, isohexadecane, and polysorbate 80 (Simugel EG, Seppic)	1.4450
Hydroxyethylacrylate/sodium acryloyldimethyl taurate copolymer, squalane, and polysorbate 60 (Simugel NS, Seppic)	1.4475
C13-14 isoparaffin (Isopar M, Exxon Mobil Chemical)	1.4380
C11-13 isoparaffin (Isopar L, Exxon Mobil Chemical)	1.4255

SNELL'S LAW AND REFRACTIVE INDEX MATCHING

Snell's law states that if n_2 is equal to n_1, then r_2 is equal to r_1. In this case, no refraction takes place and the incident beam continues in an undeflected direction. This case applies to cosmetic emulsions when the RI of the oil phase is equal to the RI of the water phase. The resulting emulsion is clear if the indices have been matched properly. In the formulation of cosmetics, this application is referred to as refractive index matching.

THEORETICAL DESIGN OF CLEAR EMULSIONS

Cosmetic chemists are interested in designing products using the principle of refraction. Experimentally, it turns out that if one mixes several miscible ingredients together to form a clear homogeneous liquid phase, the refractive index of the mixture can be calculated from each individual component's refractive index in the composition (4). The calculated value of the refractive index normally is very close to the value measured instrumentally. If W represents the weight of each component and n represents the refractive index of each component, then the RI of the mixture will be determined by equation 1 and 2, which simplify to equation 3.

The calculation equations are:

$$RI_{mix} = [W_1 \times n_1 + W_2 \times n_2 + W_3 \times n_3 + \cdots + W_n \times n_n]/W_T \quad (1)$$

where

$$W_T = W_1 + W_2 + W_3 + \cdots + W_n \quad (2)$$

We can simplify this equation as:

$$RI_{mix} = [\sum(W_i \times n_i)]/W_T \quad (3)$$

By using equation 3, indices of clear water-phase solutions containing several functional cosmetic ingredients, together with clear oil-phase solutions containing several functional cosmetic ingredients, can be calculated. It is possible to manipulate the refractive index of the water phase to be equal to that of the oil phase. Furthermore, it is possible to make a clear or opalescent emulsion by combining the water phase and the oil phase. In order to use equation 3 directly for refractive index matching in an emulsion, equation 3 is modified, as shown in equation 4 for RI_{oil} and equation 5 for RI_{water}:

$$RI_{oil} = [\sum(W_i \times n_i)]/[\sum W_i] \quad (4)$$

where W_i is the weight of each component in the oil phase and n_i is the refractive index for each component in the oil phase.

$$RI_{water} = [\sum(W_i \times n_i)]/[\sum W_i] \quad (5)$$

where W_i is the weight of each component in the water phase and n_i is the refractive index for each component in the water phase. In practice, emulsifiers have to be dissolved in either the oil or the water phase, and so it is necessary that either phase be clear or nearly so.

Some limitations have been found when using equations 4 and 5. First, no chemical reactions should take place between ingredients in either the water or the oil phase. Even neutralization will change the refractive index of some ingredients. Second, ingredients in the oil phase should be physically insoluble in the water phase, and vice versa. In another words, the ingredients chosen for use in the formula should not have dual distribution in both the water phase and the oil phase. An emulsifier (or blended emulsifier ingredients) should stay at the interface of its original phase and cannot be allowed to permeate into the other phase.

Third, it is necessary to produce emulsions at room temperature because RI values are temperature-dependent and normally the oil phase and the water phase differ in their temperature dependency. If a clear emulsion is obtained at an elevated temperature, the emulsion most likely will be cloudy or hazy at room temperature.

The refractive index also varies with the wavelength of light (5). Normally literature listed as n_D^{20} signifies the refractive index using the D-line emission of sodium, measured at 589 nm at 20°C. RI readings from a refactometer are slightly different from literature values since readings are obtained from the visible range of light (either white light or fluorescent light). With index matching in the development of cosmetic emulsions, if the indices of refraction of the water and oil phases are close, but not exact, an opalescent (translucent) appearance will result. This occurs because of light separation caused by a dispersed, drop-like inner phase. The index of refraction n (light bending) encountered by light in any medium except a vacuum depends on the wavelength of the light. The dependence of the refractive index on wavelength implies that when a light beam consists of rays of different wavelengths, the rays will be refracted by a surface at different angles, that is, the light will be spread out by refraction. This spreading of light is called chromatic dispersion. Generally, the index of refraction in a given medium is greater for a shorter wavelength (blue light) than for a longer wavelength (red light). In other words, if a beam consisting of both blue and red light waves is refracted through

a surface, such as from air into quartz or vice versa, the blue component bends more than the red component.

A beam of white light (or fluorescent light) consists of all (or nearly all) colors in the visible spectrum with near uniform intensity. When white light passes through a solid glass prism with a triangular cross section, all colors of the light are separated into a rainbow. The opalescent appearance of cosmetic emulsions originates from the slight mismatch of the RI between the water and oil phases, which leads to light separation. The opalescent effect has been used to create attractive visual effects in emulsion products. In fact, most clear emulsions created from RI matching appear to be opalescent, more or less due to the difficulty in completely matching RI values.

Chemists can utilize equations 4 and 5 to design formulas that meet specific requirements of appearance and performance. After the RI of the two phases are matched (become equal) and mixed together, the emulsion may initially be cloudy. A longer mixing time is needed to guarantee emulsion homogeneity and clarity.

RESULTS AND DISCUSSION

EXAMPLE APPLICATIONS

The following two examples demonstrate refractive index matching. These simplified formulas are intended only to describe the process of formulation design; they are by no means the best performing. The chemist can design a multitude of formulas with many different ingredients by using the principles already described (6). This method can also be used to design general dual-phase products with two colors.

RI matching in skin-care products: A clear AHA gel. Alpha hydroxy acids (AHAs) have been determined to have antiaging and antiwrinkle effects. AHAs have been widely used in skin-care formulations (7,8). However, AHAs are also skin irritants. By incorporating an AHA complex (glycolic acid and arginine) into a water-in-silicone oil emulsion, it is possible to reduce potential irritation and obtain a clear eye-gel product. The following example shows a preliminary formula with the corresponding RI. The related RI calculation is also given for RI_{oil} (1.3967) and RI_{water} (1.3966) as an example when using equations 4 and 5. The clear eye-moisturizing gel example is shown in Table II.

RI matching in hair-care products: An O/W hair silicone styling gel. Silicone oil is often used to deliver shine, a luxurious feel, anti-frizziness, and manageability to hair-care products (9,10). Hair fixative ingredients (such as PVP/VA or PVP) are commonly used in carbopol gels, giving styling effects to hair. Interestingly, if one can combine silicone oil and hair fixatives into a clear gel-like product, the new product will impart both hold and the aforementioned properties of silicone. As shown in Table III, the principle of RI matching can be used to make a clear silicone styling gel, with a value of 1.4060 calculated for both the RI_{oil} and the RI_{water}.

DEVIATIONS AND PRACTICAL RI ADJUSTMENT

Actual refractive index values often deviate from theoretical values. In theory, calculations in the summation of the refractive index for solutions apply only to ideal solutions or ideally dilute solutions. In ideal solutions, the molecules of various species are so

Table II
Clear Eye-Moisturizing Gel

Ingredient	Weight %	RI value
Silicone oil phase		
Cyclomethicone and dimethicone (DC 1501, Dow Corning)	10.0	1.3971
Cyclomethicone and dimethicone copolyol (DC 5225, Dow Corning)	10.0	1.3975
Cyclomethicone (DC 344, Dow Corning)	5.0	1.3942
AHA water phase		
Water, deionized	36.1	1.333
Glycerin	26.25	1.468
Glydant	0.15	1.425
Glycolic acid and arginine (AHCare G-60, Cognis)	12.5	1.428
	100.0	
RI_{oil} = (10 × 1.397 + 10 × 1.398 + 5 × 1.394)/25 = 1.3967.		
RI_{water} = (35.5 × 1.333 + 27 × 1.468 + 12.5 × 1.428)/75 = 1.3966.		

Table III
Clear Silicone Styling Gel Calculated for Both Oil and Water Phases

Ingredient	Weight %	RI value
Silicone oil phase		
Cyclomethicone (Rhodorsil 45V5, Rhodia)	4.00	1.396
Polyacrylamide, C13-14 isoparaffin, and laureth-7 (Sepigel 305, Seppic)	3.00	1.446
Cyclomethicone, phenyltrimethicone, and dimethicone (Gelaid 5565, Chemsil)	20.00	1.402
PVP/VA water phase		
Water, deionized	26.50	1.333
Glycerin	21.35	1.472
PVP/VA copolymer solution (50% active) (Luviskol VA 73W, BASF)	25.00	1.427
DMDM hydantoin (Glydant, Lonza Group)	0.15	1.425
	100.00	
RI_{oil} = (4 × 1.396 + 3 × 1.446 + 20 × 1.402)/27 = 1.4060		
RI_{water} = (26.5 × 1.333 + 21.35 × 1.472 + 25 × 1.427 + 0.15 × 1.425)/73 = 1.4060		

similar (in terms of structure and spatial filling) to one another that molecules of one component can replace molecules of another component in the solution without changing the solution's energy or spatial structure. In ideally dilute solutions, all solutes are present in very low concentrations and the solvent weight percentage approaches 100%.

In cosmetic formulations, solutions are neither ideal nor ideally dilute because the solvent and solutes cannot be very similar, and the concentrations of solutes cannot be impractically low. They are non-ideal solutions containing both electrolytes and non-electrolytes. More precise calculations would require chemical potential (μ_i), solute activity (a_i), and activity coefficients (γ_i). The calculation becomes very complicated and impractical for cosmetic chemists. Fortunately, a simplified calculation is sufficient to serve the purpose of designing formulas.

In the process of making the oil phase and water phase, for example, the RI will deviate slightly from the calculated value. The deviation in the oil phase is usually very small because all the RI values of the oil-phase ingredients are very close. Consistent RI

readings of oil fluids result from the similarity in both the molecular structure and the interaction between these molecules.

However, there is a noticeable RI deviation in water-phase preparation. In matching RI values of water and oil phases, one uses water (low RI value) and glycols (high RI values). In addition, the molecular interaction between glycol and water is different from the interaction between those molecules by themselves. Therefore, the formula needs further refinement to assure proper index matching.

Various glycols are used to raise the refractive index of the aqueous phase to match that of silicone oil (RI ~ 1.4) or isoparaffin (RI ~ 1.43). The refractive index values for several glycols as a function of concentration are shown in Figure 2 for a two-component system. The glycol aqueous concentrations vary from 10% to 80%. Different deviations are seen in the plot. Glycerin shows negative deviation and three glycols show positive deviation. Hexylene glycol shows the largest positive deviation. Glycereth-7 shows very little deviation. All deviations are concentration-dependent.

Deviations are compared with the calculated values shown in Figures 3–5. Negative deviation is observed for glycerin in aqueous solution (Figure 3). The largest negative deviation is seen for glycerin at the concentration of 30%, where there is a −0.51% deviation (1.367–1.37395 = −0.00695) from the calculated value.

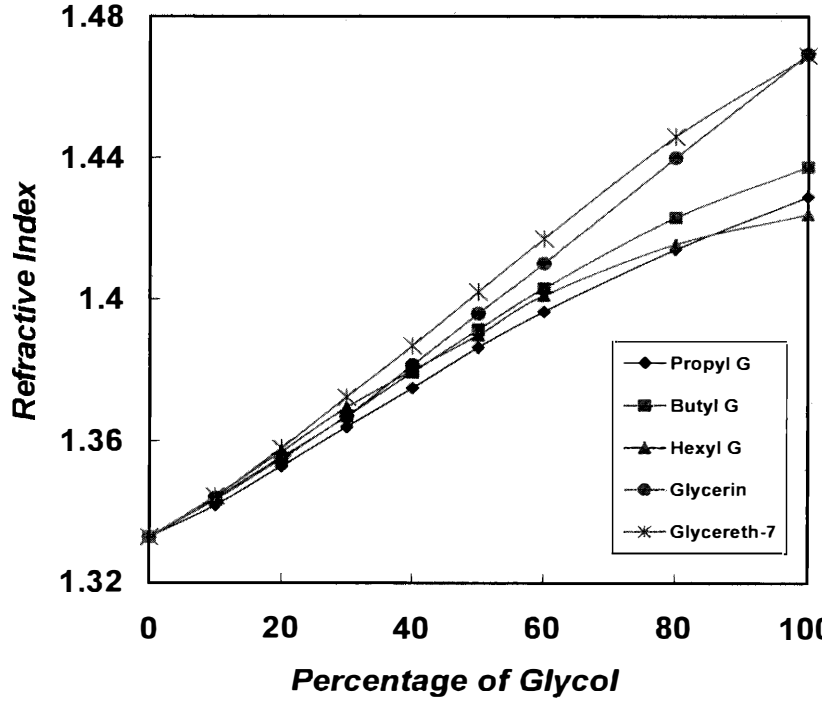

Figure 2. Concentration-dependent RI values of several glycol aqueous solutions.

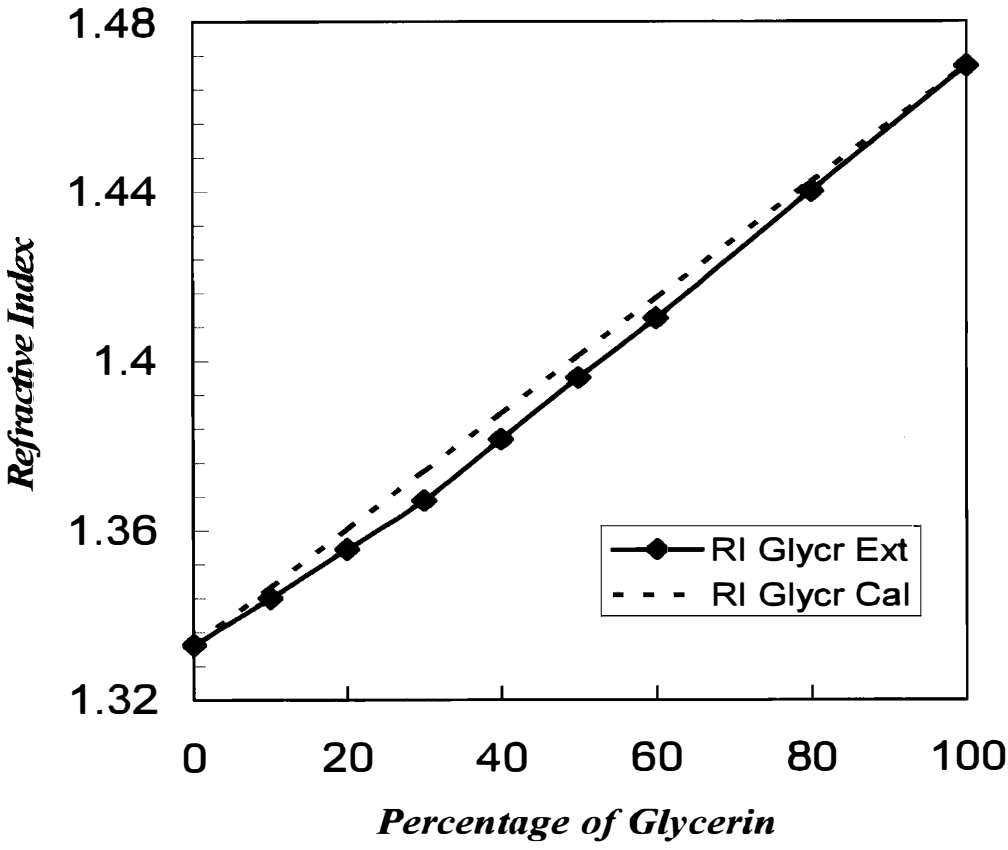

Figure 3. Theoretical and experimental concentration-dependent RI values of glycerin aqueous solution. Solid line is experimental and dashed line is theoretical.

Positive deviation is seen for hexylene glycol aqueous solution in Figure 4. It is the largest positive deviation among all the studied glycols (Figure 2). The experimental RI value of hexylene glycol is even larger than the value of glycerin solution at concentrations in the range of 10–35%. The largest deviation of the refractive index for hexylene glycol is at the concentration of 60%, where it deviates 0.97% from the calculated value.

Figure 5 demonstrates slightly negative deviation at lower concentration and slightly positive deviation at higher concentration for glycereth-7 aqueous solution. Glycereth-7 is a unique case since deviations run from slightly negative at low concentration (below 40%) to slightly positive at high concentration (above 50%). Negative deviation at lower concentration is comparable to what is observed in aqueous glycerin, and positive deviation at higher concentration is comparable to that of hexylene glycol.

As a way of explaining deviation, the specific gravities of aqueous solutions of glycerin and three glycols (propylene glycol, butylene glycol, and hexylene glycol) at 50% by weight were measured and are listed in Table IV. The specific gravity of glycerin solution is lower than the calculated value (average of two components), which indicates a slight increase in volume after glycerin and water are mixed. The volume increase might be related to the difference in intermolecular interactions between water mol-

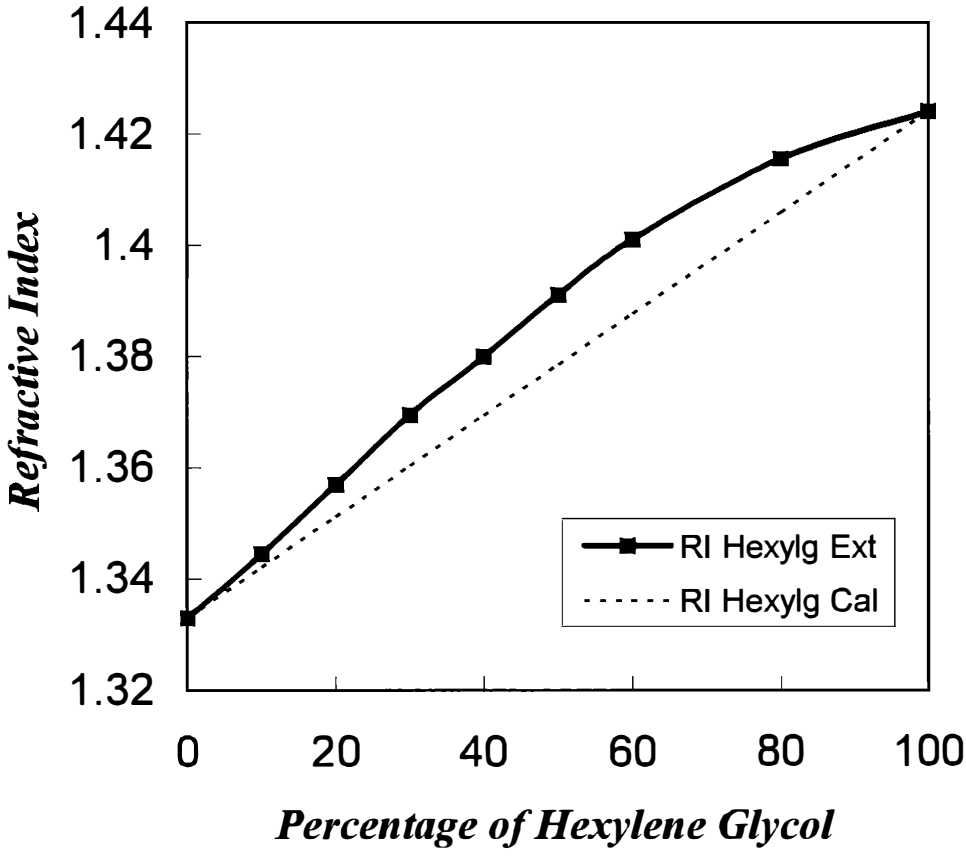

Figure 4. Theoretical and experimental concentration-dependent RI values of hexylene glycol aqueous solution. Solid line is experimental and dashed line is theoretical.

ecules, between glycerin molecules, and also between water and glycerin molecules. The interaction between water molecules and between glycerin molecules is possibly stronger than the interaction between water and glycerin molecules. A decrease in specific gravity results in a decrease in optical density, which is observed as a negative deviation in refractive index measurements.

It turns out that glycol solutions behave differently compared to glycerin solutions, as is illustrated in Table IV. The observed increase in specific gravity corresponds to a decrease in volume after glycols dissolve in water. A plausible explanation could relate to spatial filling, since the three glycol molecules are structurally larger than water molecules. Also, water molecules can fill voids between glycol molecules in glycol solution. An increase in solution specific gravity leads to an increase in optical density, which results in positive deviation in refractive index measurements.

Propylene glycol, butylene glycol, and hexylene glycol all show positive deviation (Figure 2). However, propylene glycol demonstrates the least and hexylene glycol the most. The nature of deviation is related to molecular interaction between water molecules, between glycol molecules, and between water and glycol molecules. These three glycols have the same diol (dihydroxy) functional group on two carbon atoms (illustrated in

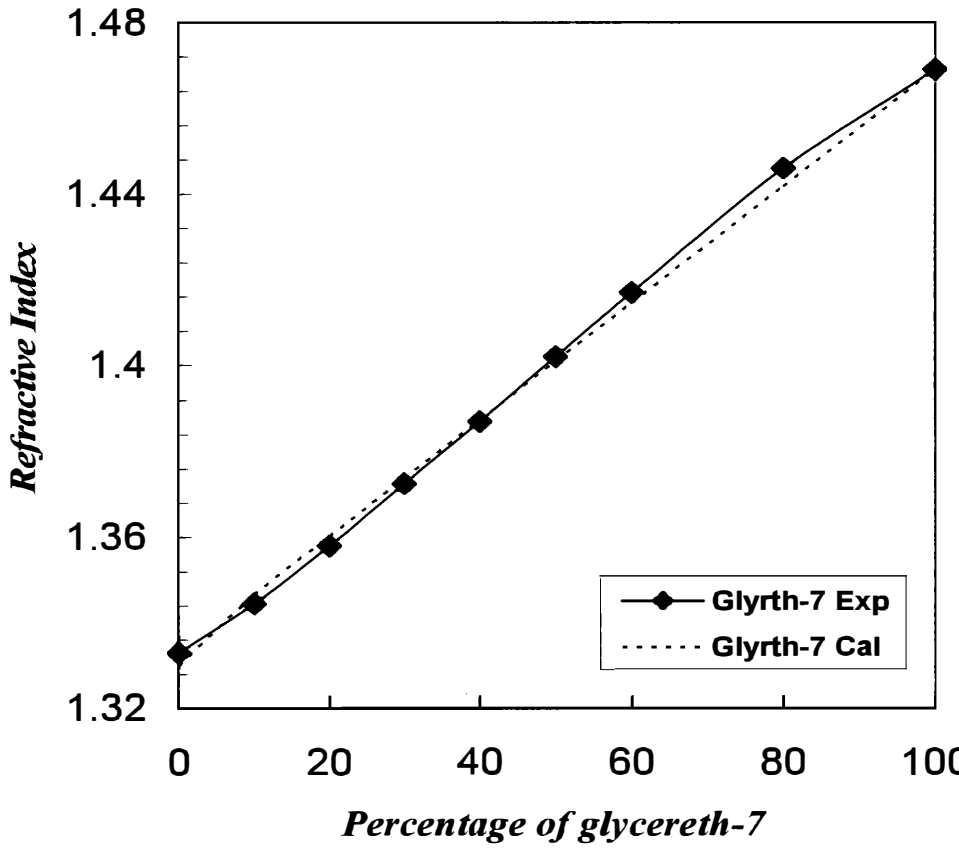

Figure 5. Theoretical and experimental concentration-dependent RI values of glycereth-7 aqueous solution. Solid line is experimental and dashed line is theoretical.

Table IV
Specific Gravity of Glycols in 50% Aqueous Solution

Glycol	SPG Exp	SPG Cal	ΔSPG
Glycerin	1.1268	1.1304	−0.0036
Hexylene glycol	0.9870	0.9613	+0.0257
Butylene glycol	1.0251	1.0085	+0.0166
Propylene glycol	1.0354	1.0184	+0.0170

Figure 6). The more carbon contained within the molecular structure, the bulkier the glycol molecule. The more carbon the molecule has, the greater the difference will be in molecular interaction between glycol and water molecules. The greater this difference, the more positive is the observed deviation (Figure 2). Hexylene glycol shows the greatest increase in specific gravity and a concomitant positive deviation in the refractive index.

Negative RI deviation corresponds to a decrease in the specific gravity of glycerin solution. Positive RI deviation corresponds to an increase in the specific gravity of glycol solution. Based on these observations, it is reasonable to generalize that any

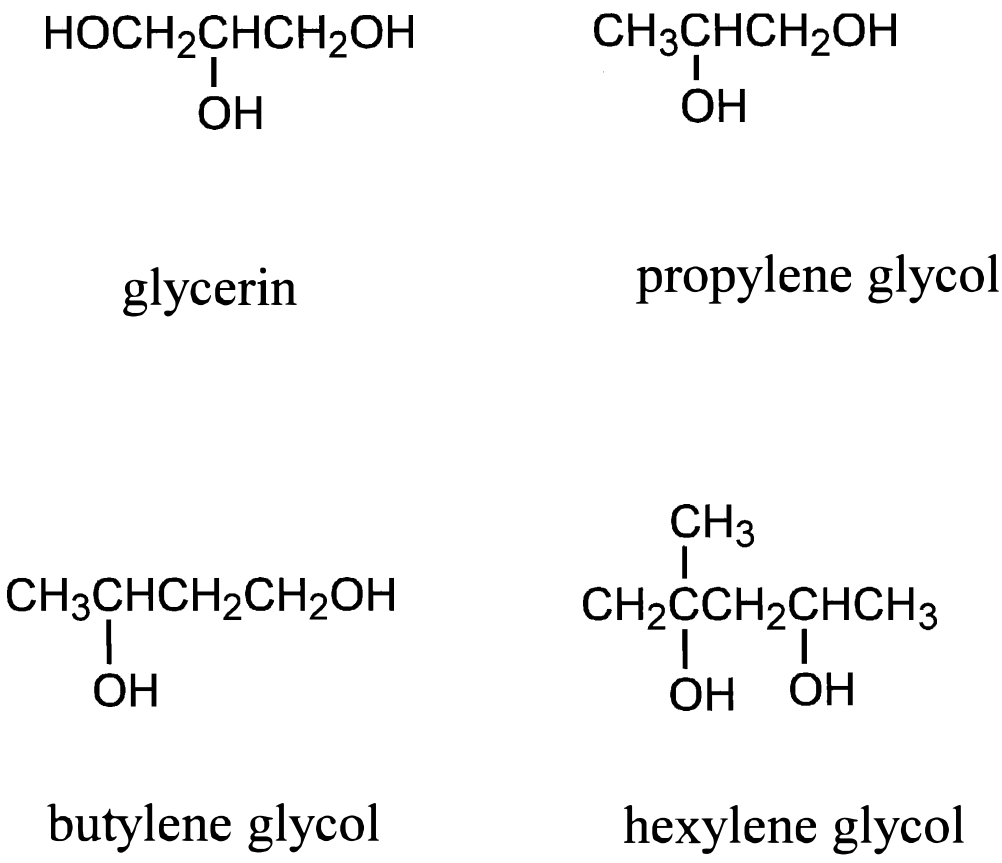

Figure 6. Molecular structures of glycerin, propylene glycol, butylene glycol, and hexylene glycol.

increase or decrease in specific gravity leads to an increase or decrease in optical density, which results in RI values that deviate positively or negatively from calculated values, respectively.

The dominant interaction forces between molecules of water and glycols or glycerin are hydrogen bonds since all of them are polar molecules with hydroxyl groups. Hydrogen bonds play important roles for the decrease in specific gravity in glycerin solution. The reason for volume increase (specific gravity decrease) when forming glycerin aqueous solution is that there is stronger hydrogen bonding between glycerin molecules than the hydrogen bonding between molecules of water and glycerin. That the process accompanies a significant heat release when hydration takes place also indicates the different strength of hydrogen bonding. Glycols, however, are hydrated differently. That there is a volume decrease (specific gravity increase) when forming glycol solutions indicates that there is weaker hydrogen bonding between glycol molecules than the hydrogen bonding between molecules of water and glycols.

It is apparent that the most efficient way to raise the refractive index of the aqueous phase is to use a combination of hexylene glycol in the range of 20–30% with either glycerin, glycereth-7, or butylene glycol. Propylene glycol is less efficient at raising the RI of the water phase because it has the least positive deviation and the lowest refractive

index value in the group. Glycereth-7 is also the easiest one to use in calculation since it has the least deviation from calculation. In the formulation process one has to consider, among other factors, the formulation's performance, ingredient cost, and ease of operation.

CONCLUSIONS

By using Snell's law in cosmetic formulation, a simple calculation scheme has been developed for designing clear emulsion formulas by matching the refractive indices of the water phase and the oil phase. The RI value of the water phase was adjusted by varying the ratio of water and glycols. Positive deviation and negative deviation were observed for water-glycol two-component systems. The optical density changes in glycol aqueous solutions result in RI deviation. The most effective glycols are the glycereth-7 and glycereth-26 type, which have the least deviation from calculation. The use of index calculation and deviation charts leads to more precise formulation design.

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