WATER-SOLUBLE EMULSION TECHNOLOGY'S IMPACT ON STABILITY

POWERED BY SŌRSE™

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– What Is an Emulsion?

Generally speaking, an emulsion is a combination of two or more immiscible liquids – fluids which will not mix, such as oil and water. Emulsions provide a pathway to mix or blend immiscible fluids, and present them as a uniform mixture. Emulsions play an increasingly active role in our day-to-day lives, including within food and beverages. Uniform dispersion in water platforms and foods is one of the largest challenges relating to cannabinoid product development.

Emulsions can be found in products ranging from mayonnaise, Hollandaise sauce, and salad dressings, to soft drinks, functional beverages, pharmaceuticals, and even baked goods. The introduction of cannabinoids to water-based formulations is a new application in these seasoned industries. Cannabinoid requirements bring an extra level of complication to the emulsion system, primarily uniformity and stability over long periods of time, coupled with managing their extreme bitterness. First, let's look at emulsion constructs and mechanisms.

– What Makes Liquids Immiscible?

Water is a polar hydrophilic molecule (lipophobic, water loving, fat/oil fearing), whereas cannabidiol (CBD), along with most lipids, oils, and fats, are hydrophobic (fats/oil, lipid loving), non-polar molecules. A primary factor to hydrophobicity is the magnitude of symmetry. Polarity is determined by the electronegativity of atoms. A molecule is considered polar if there is a net positive or negative electrical charge (electronegativity). The greater the difference in this charge, the greater the polarity. Polarity can also be derived from the symmetry of molecular structure as with water (H2O). In terms of H2O, there is one oxygen atom with an electronegativity of 3.5 and two hydrogen atoms with an electronegativity of 2, resulting in a net electronegativity of 1.5. The presence of a difference is referred to as an electrical dipole. The electronegativity difference can be used to classify the type of bond and the polarity as shown in the table. While this same conceptual logic can be applied to classifying oils as polar or non-polar, the determination of specific electronegativity is more complicated and outside the scope of this paper. This is due to complex structures and mixtures of oils used.

Nearly all common oils used for foodstuffs and cooking are considered non-polar and hydrophobic. Their electronegativity and hydrophobicity, however, fall on a continuum. This is largely due to their non-polar hydrocarbon bonds which result in low electronegativity differences. Some lipids that have a larger electronegativity difference often contain long non-polar hydrocarbon tails and polar hydrophilic heads. These compounds are referred to as amphiphiles, and include the biologically important phospholipids.

– Emulsions Are Not Created Equally

In many cases, emulsions are used to carry flavors into a beverage system. In the case of cannabinoids, the emulsion provides a variety of functions:

1) isolate the negative sensory aspects of the cannabinoids
2) enhance potential flavor profiles
3) provide a highly uniform/homogenous and stable solution of active cannabinoids
4) create an emulsion that can be diluted and remains stable.

In order to understand all the variables that affect emulsion stability, a basic understanding of emulsions is crucial. Emulsions contain two basic components: a dispersed oil phase, and a continuous aqueous phase (often referred to as the matrix). The dispersed phase can include the active oil to be dispersed, other oil blends, emulsifiers, and weighting agents. The continuous phase can include water, emulsifiers, preservatives, flavors, sweeteners, and other dissolved solids. A simple emulsion is possible with just oil and water if enough mechanical force is applied through a high sheer; however, the emulsion will be unstable and susceptible to a number of failure modes: creaming, flocculation, coalescence, and sedimentation. Next, we will examine these terms.

– Creaming and Sedimentation

Creaming and sedimentation are two results of gravitational forces. Creaming or sedimentation are often the result of a difference in densities between the dispersed phase (oil) and the continuous phase (water). Oils tend to be less dense than water (between 0.91 g/cm3 - 0.93 g/cm3 at room temperature), whereas water has a density of 1.00 g/cm3. Creaming happens when the dispersed oil phase that has a lower density than the continuous phase moves upward and results in a thick, separated layer. Conversely, sedimentation happens if the dispersed oil phase has a higher density than the continuous phase, causing the droplets to move downward (Hu, Ting, Hu, & Hsieh, 2017). An emulsion can appear homogeneous to the naked eye, but creaming may still be occurring. In the case of cannabinoids, emulsion creaming, or sedimentation, is not desirable, as it results in a non-uniform product and uneven distribution of cannabinoids. Prior to complete failure, the emulsion begins to separate into two fractions containing various levels of oil loading. When consistent dosing is part of the purpose of the emulsion, creaming and/or sedimentation can be considered a failure.

– Uniformity/Homogeneity, Stability and Safety

Getting two immiscible liquids to mix is only part of the problem. Take, for example, a simple, common emulsion – salad dressing. A couple of seconds of shaking will disperse the oil (dispersed phase) into the vinegar/water (continuous phase). The water/vinegar combination act as the emulsifier by coating the oil drops, yet only temporarily. In terms of a vinaigrette, the biggest concern is a salad dressed uniformly with vinegar and oil. For many consumer beverages, homogeneity may be nothing more than a measure of aesthetics and personal preference. This is not the case, however, for beverages containing cannabinoids such as THC and CBD. Uniformity/stability is crucial for containing cannabinoids as well as beverages containing ingredients from sugar and sugar alcohols (for sweetness) to caffeine and other ingredients with a defined limit of exposure. Imagine a product that contains 100mg of caffeine per serving, but there are 10 servings in the container. If the caffeine is not evenly dispersed throughout, it would be possible to unwittingly imbibe all 1000mg in the first sip. It is also important to note that creaming (identified visually with the naked eye or with the analytical tools such as Turbiscan data) is not indicative of a broken emulsion, and agitation may cause the solution to revert to an apparent homogeneous state.

– Creaming and Sedimentation

As mentioned previously, gravity is one of the biggest enemies of emulsions. The force of gravity is a direct cause of both creaming and sedimentation destabilization. Creaming and sedimentation are cases where the emulsified particles remain intact and distinct, yet concentrate at the top or bottom of the liquid due to differences in density and the force of gravity. The forces of gravity can be overcome by manipulating several variables, including: viscosity of the water phase, solute concentration, and increasing the density of the dispersed oils with weighting agents. While increasing the viscosity (thickening) of the liquid is likely the easiest way to increase stability by directly slowing the flow of particles, it is not typically practical because it can negatively impact sensory aspects such as mouth feel. Key characteristics of creaming and sedimentation are that the emulsified particles remain intact, and can be re-dispersed with simple agitation.

Just because an emulsion is stable in a concentrated form does not necessarily mean that the emulsion will continue to be stable once let down/diluted. This is where balancing the density is critical.

EMULSION FAILURE MECHANISMS

– Coalescence and Flocculation

Coalescence describes when the oil droplets recombine and grow in size over time. This typically results in a solid oil layer or "ring" forming at the top of the liquid, and is not recoverable. Flocculation refers to cases when the inter-colloidal forces between the oil droplets are out of balance, resulting in irregular collections of grouped oil droplets, which can lead to a non-uniform product. Use of appropriate and suitable emulsifiers will stabilize the emulsified oil droplets, which, in turn, decreases the rate of coalescence and flocculation. Flocculation typically presents at the top of the beverage, but may occur at the bottom.

– Particle Size and Distribution

Like concentration, the size of the particles and their distribution throughout the medium are equally important to stability, uniformity/homogeneity, and safety. Particle size also has a large impact on the clarity and turbidity of the emulsion. As particle size decreases, the surface area of the oil increases, typically leading to an increase in stability. Emulsions, by definition, are thermodynamically unstable and will eventually break. For simple emulsions (think oil and vinegar salad dressings), separation can occur within seconds. However, for well-formulated emulsions, the process of separation can be slowed to months and years.

– Built to Last

SōRSE has been specifically designed to provide exceptionally long-term uniformity, homogeneity, and stability over time. A number of analytical techniques are utilized by SōRSE to demonstrate and verify stability, uniformity, and cannabinoid assay. It is important to remember it is not just the formula that makes an emulsion successful, but the formulation combined with specific processing to ensure product uniformity and safety.

– Turbiscan Lab

The Turbiscan is the industry standard and recommended method by the International Standards Organization for monitoring dispersed phase uniformity and stability over time. The Turbiscan cannot only corroborate physically observable characteristics of a beverage such as creaming and sedimentation, but the Turbiscan can make these observations well in advance of what the human eye can detect. Thus, the Turbiscan can also show degree of homogeneity and susceptibility for coalescence and eventual breaking of an emulsion in an accelerated manner.

Unlike simple particle size analysis, which is a single measurement at a single point in time, back scattering compares multiple measurements taken over time. In this manner, it not only provides snaps shots of uniformity, it also yields rate dependent information, such as destabilization kinetics and predictions of long-term stability.

The Turbiscan utilizes static multiple light scattering to measure changes in the emulsion system. The instrument sends an 880nm beam of light and measures back-scattered light and transmitted light. This relationship can tell the user a lot about the product long term stability and has been used to verify that SōRSE is formulated to provide stability in excess of one year.

– How to Interpret Turbiscan Data

Formulaction (the maker of the Turbiscan) has developed what is referred to as the Turbiscan Stability Index (TSI). The TSI is a single value metric that corresponds to a cumulative sum of all of the backscattering and transmission across the entire sample based on the algorithm on the next page. TSI can be used to compare and rank stability objectively across samples. It can also be used to evaluate and identify different phenomena contributing to destabilization. Essentially, the lower the number, the more stable the beverage.

$$TSI(t) = \frac{1}{N_h} \sum_{t_i=1}^{t_{max}} \sum_{z^i=z_{min}}^{z_{max}} |BST(t_i z_i) - BST(t_{i-1,} z_i)|$$

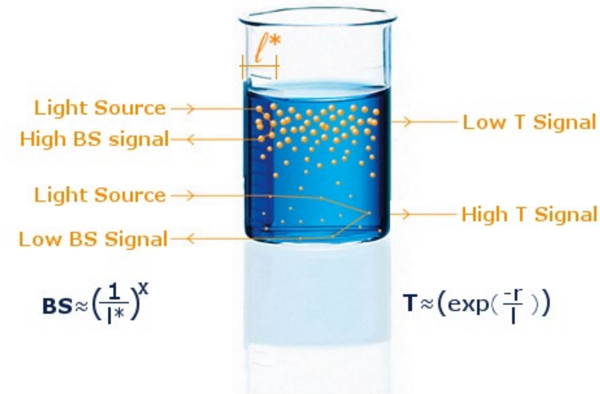


FIGURE 1 BELOW: Example of a stable emulsion, long-term, 197 day Turbiscan result of SōRSE infused beverage. The Horizontal nature of the BS & T curves illustrates little variation over time and a significantly stable beverage emulsion.

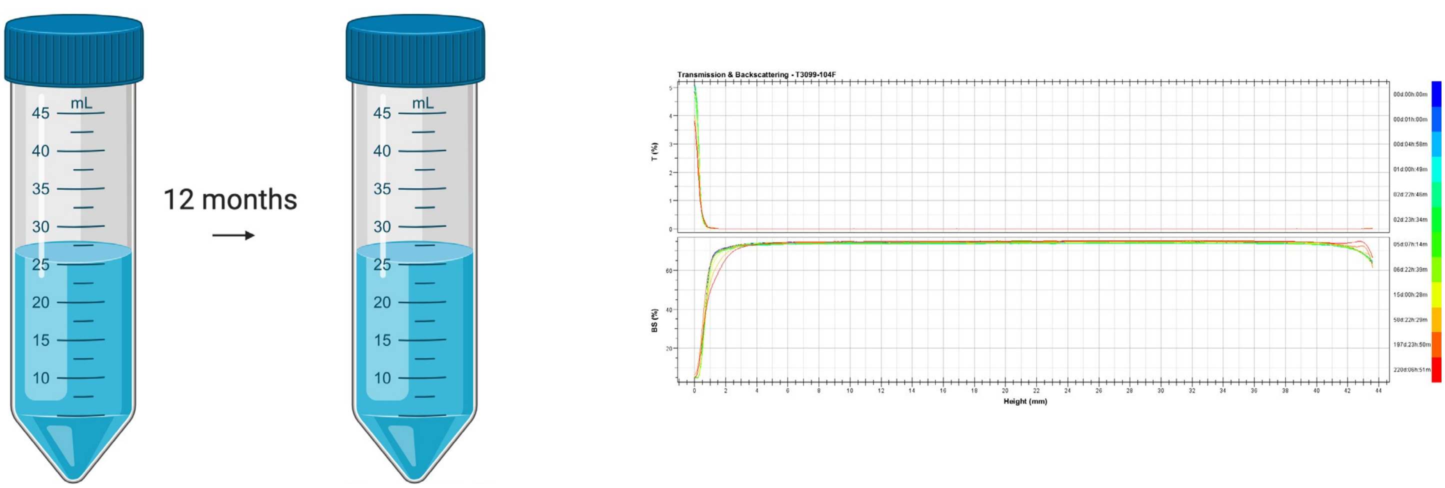


FIGURE 2 BELOW: Example of an unstable emulsion (not SōRSE) undergoing sedimentation: A decrease in backscattering (BS) at the right side of the plot is suggestive of clarification, whereas an increase in BS on the left of the chart is indicative of sedimentation. Both of these destabilization mechanisms occur simultaneously.

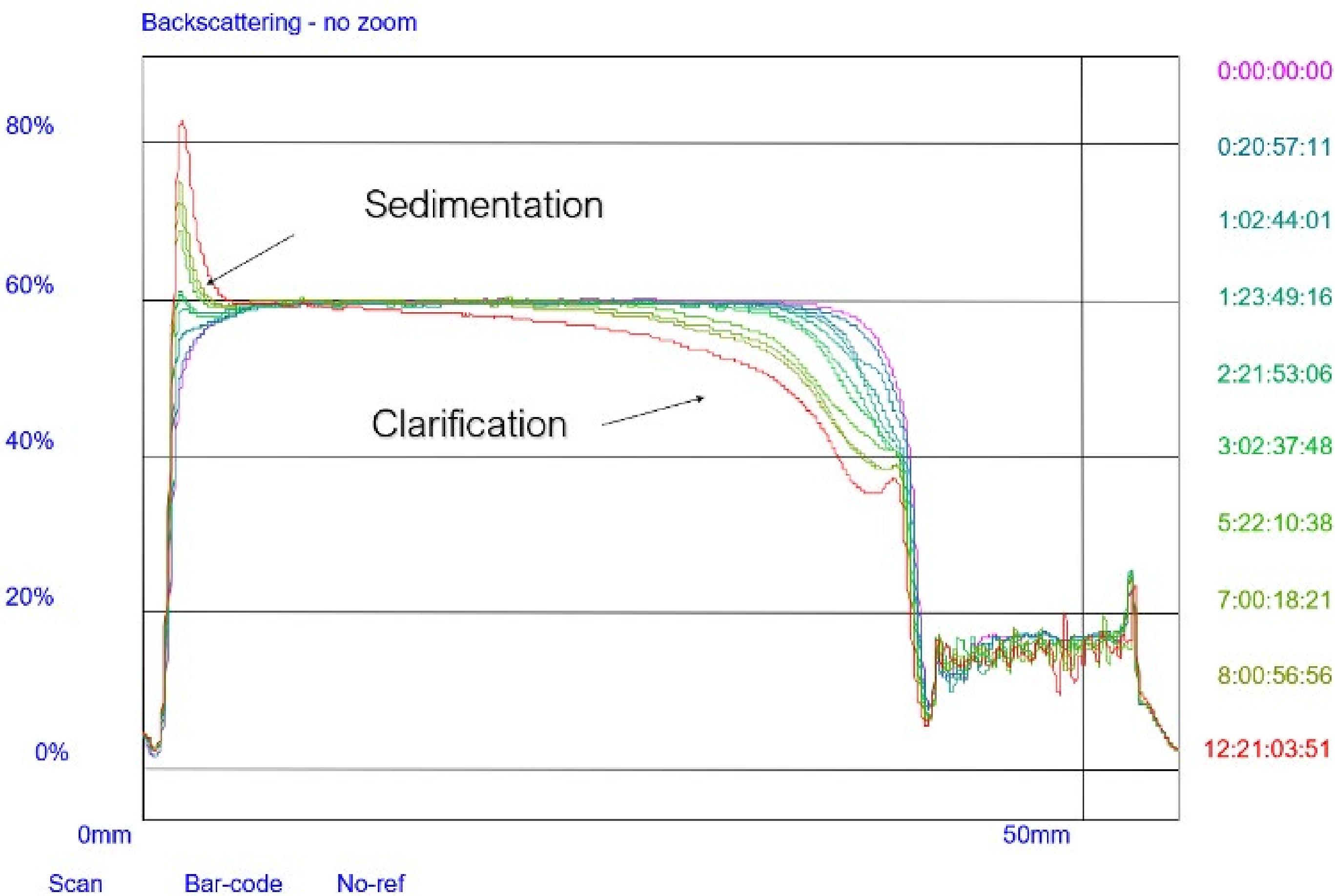
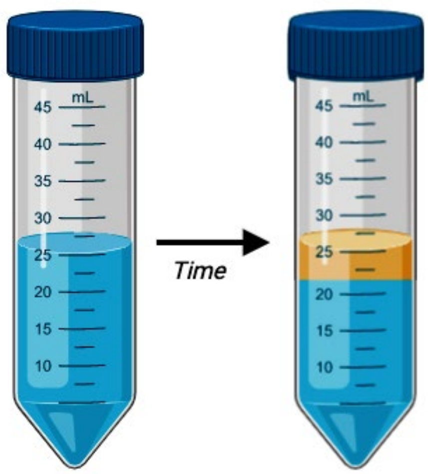


FIGURE 3 BELOW: Example of an emulsion (not SōRSE) with clarification/creaming. An increase of backscattering to the right of the plot indicates creaming, whereas a decrease on the left side indicates clarification. This is the most typical failure mechanism in beverage emulsions because oils are typically lighter than water.



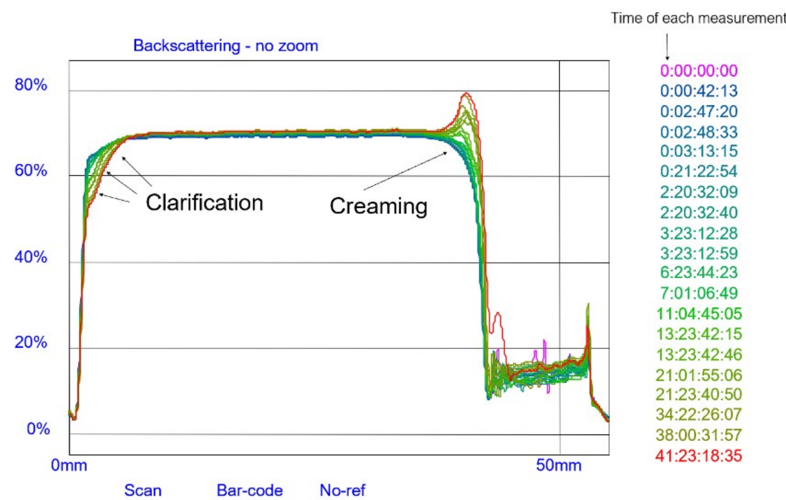
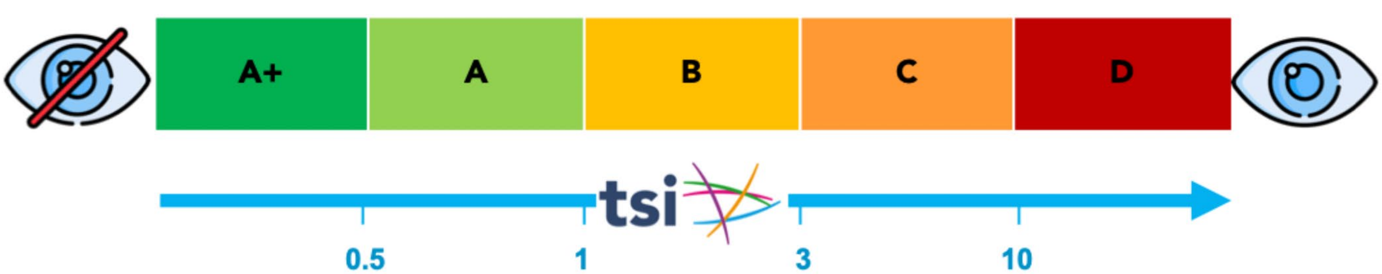


FIGURE 4 BELOW: The scale below is Formulactions' (maker of the Turbiscan) Quantifiable Index that can be used to describe the destabilization kinetics of the emulsion, and to predict several of the emulsion's properties such as shelf life, stability, and uniformity within and between products. On the left end the scale, 0/A+ indicates that no significant destabilization has been observed with the Turbiscan® and the sample remains visually stable. A+ ranking is the best stability mark. On the right end of the scale, 10/D indicates a visual fail in the emulsion. Extreme and important variation and the destabilization is most likely visible, corresponding to large sedimentation or creaming, phase separation, and wide change in the particle size or color.



– High Performance Liquid Chromatography (HPLC)

Chromatography is a technique that separates a solution into its individual components based on their molecular weight and their ability to flow through a material. Different compounds/molecules travel at different rates, through a medium such as a packed column, which separates the mixture into its components. Chromatography techniques can vary, from the simpler paper chromatography or thin layer chromatography (TLC) to more complex and informative methods such as High-Performance Liquid Chromatography. Chromatography cannot only be used to make qualitative observations but quantitative determinations as well.

– Particle Size Analysis

Distribution of particle size is an important aspect of SōRSE stability. For stable emulsions, smaller particles are good. Long term emulsion stability avoids coalescence and gravitational separation in part due to particle size management. Typically, smaller is better (<1μm). Consistent particle size with a narrow range is most beneficial for long term stability.

For particle size determination, SōRSE labs utilizes the Horiba LA-960 high performance laser diffraction analyzer. This equipment utilizes laser diffraction theory (Mie Theory) to determine particle size similar to the principles used with the Turbiscan but with many more sensors. The LA-960 uses these sensors to detect wavelength, relative refractive index, and scattering angle to determine particle size assuming spherical shape and give the user a histogram of size variation. Formulae and computer programs are available for calculating/predicting shape. The distribution range given by the histogram allow further prediction/indication of stability. The wider the distribution of particle size, the more likely coalescence of these particles will occur.

– Discussion

Cannabinoids are relatively new to the food space. As they are introduced into food and beverages, the primary drivers for cannabinoid ingredients will always be based in consumer safety. These are followed by sensory (or consumer liking) coupled with efficacy and ease of use at a manufacturing level.

Cannabinoids bring with them some specific challenges which have been outlined above and include: uniformity in product (safety), stability over time of that uniformity in product (safety), achieving desirable sensory (consumer preference), achieving rapid and repeatable onset with predictable experience durations (safety and consumer preference), and ease of use in manufacturing environments (practical considerations).

The data above confirms that SōRSE encompasses all these factors critical for success. Long term Turbiscan results confirm complete uniformity/homogeneity over long periods of time of both the emulsion concentrate and let down beverages. Particle size analysis confirms the underlying stability of the emulsified particles. The pharma-kinetics work clearly demonstrates the rapid absorption coupled with a defined and managed experience duration. As for sensory, you simply need to have a taste. A glass of water with SōRSE emulsion added (50 mg of cannabinoids) still tastes like water.

With ease of manufacturing in mind, SōRSE's water-based emulsion easily dilutes in either a tank or in an individual bottle environment. The inherent uniformity makes let down easy and ensures perfect dosing. The two-powder forms, including an agglomerated powder, are designed for either dry mixing, tablet compression, or easily reconstituted beverages.

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