EMULSIONS

What is an Emulsion?

- Oils and water don't mix, oil is less dense than water so will float on top
- We create oil in water emulsions, where oil droplets such as beta carotene are evenly dispersed in a water phase.
- An emulsion requires energy and emulsifiers/surfactants to remain stable:

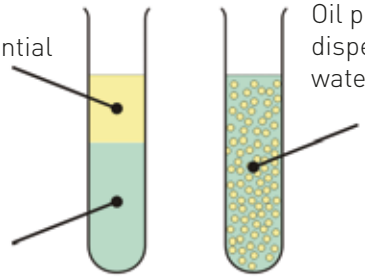
Emulsifiers we use: Gum Acacia, Sucrose Esters, Polysorbates
Energy input: High shear mixers, Homogenisers

Oil Phase

Beta Carotene 30%
Paprika Oleoresin
Black Pepper Essential Oil

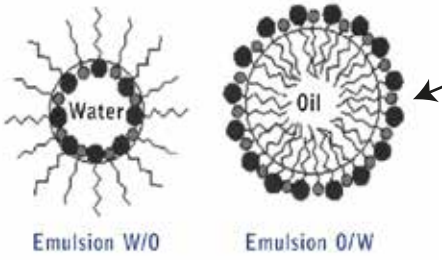
Water Phase

Water
Vegetable Glycerine
MPG
+ Emulsifier
+Antioxidant



Emulsion

Oil phase dispersed in water phase



Emulsifiers surround the tiny oil droplets and prevent them from joining back to together

Particle Size

The size of the oil droplets affects the colour performance and stability of the emulsion.

Performance: a smaller oil droplet (1 – 15 microns) is brighter and more translucent as more light can pass between the droplets. A larger droplet (15 – 50) appears less bright but has a stronger colour and more opacity.

Stability: smaller particles are more stable in beverages, if the particle size is too big the oil will begin to float to the surface. Small particle sizes are required in beverage formulations to avoid separation or ringing from occurring and ensuring stability over product shelf life.

Visual effect of particle size

Left to right: smallest particle size to biggest in beta carotene emulsion (pigment content and dosage are the same throughout)

Making an Emulsion

1. **Prepare water and oil phase -** In separate vessels, involves heating and careful mixing of all the ingredients. Requires precise measurements and careful attention to maintain the right parameters.

2. **Oil phase for Carotene** is heated to 150 celcius and a sample is checked under a microscope to ensure the BC crystals have dissolved into the carrier oil.

3. **Combination of phases** – when the two phases have reached the correct temperatures, the oil phase is slowly poured into the water phase under a high shear mixer. This is a slow process and at this point the two phases are not recoverable, if the emulsion is wrong it must be started again.

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4. Testing – the emulsion is tested for strength using a spectrophotometer. It will also be X-rited for shade checking. A PSA measurement will be carried out to ensure correct particle size and distribution for that specific product.

5. Homogenisation – all beverage emulsions, and some speciality non-beverage emulsions, are then homogenised to specific pressures in order to further reduce the particle size.

6. Further testing – a second round of PSA and X-rite tests to pass QC.

7. Spray drying – in the case of Beta Carotenes, the product will then be spray dried if a powder is required.

Equipment

- 2 x heated vessels
- 2 x High shear mixer
- Homogeniser
- Temperature probes
- Particle Size Analyser and QC equipment
- Sieves, scales, Measuring containers and PPE
- 1 – 2 Production Ops

Ingredients:

- Water Phase mix – mainly water, glycerine, glucose or MPG
- Oil Phase mix – carrier oil (MCT or Rapeseed Oil) plus oil to be emulsified e.g beta carotene.
- Emulsifier – Sucrose Esters, Polysorbates, Gum Acacia
- Acids – citric acid for lowering pH
- Antioxidants – Tocopherols and Ascorbic Acid

Emulsion Stability

- Emulsions must be carefully designed for use in beverage applications.
- If oil droplets are too big then 'neck ringing' can occur, as well as 'creaming' and eventually full separation of the oil from the water. Oil is less dense so floats on water.
- To achieve a stable emulsion the right level of emulsifier must be added with careful processing to ensure an even, and suitably small particle size has been achieved.

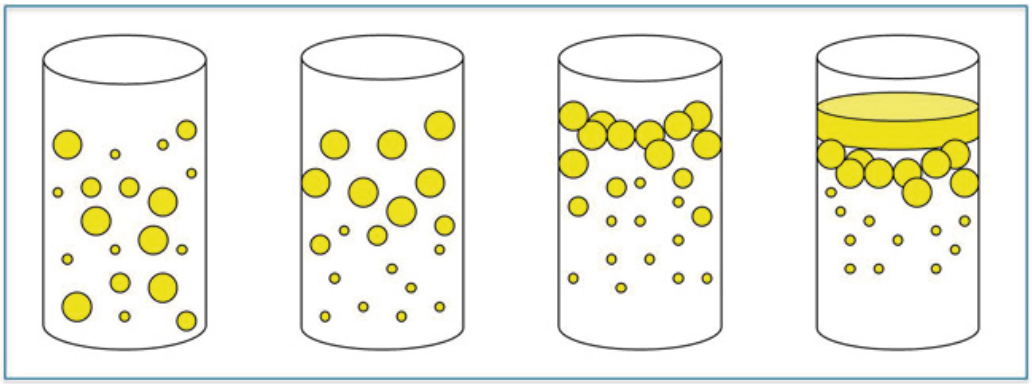

FIG. 2. Emulsions offer different stability, and applications demand varying levels of emulsification stability. From left to right: a stable emulsion; the emulsion has begun to separate; the emulsion is creaming (thick white layer on top of the mixture); the emulsion has broken (noticeable oil layer on top of the water phase). Courtesy of The Lubrizol Corp. (Wickliffe, Ohio, USA); reprinted with permission from *Tribology & Lubrication Technology* 69(9):32–39 (2013).

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• Density of the oil phase can be changed using Ester Gum (weighting agent) so that it is the same/similar to water, therefore is less likely to separate.

• Beverage stable emulsions should have very small particles, with an average particle size of 1 – 5 microns.

• We conduct particle size analysis (PSA) to ensure that the particles are within the correct size range.

• The most common way to achieve this particle size is a two step manufacturing process: first the emulsion is high shear mixed and then it is then passed through a homogeniser at high pressure (typically 300 Bar)

• A homogeniser forces the emulsion through a small hole under very high pressure, this further breaks the oil droplets and reduces their size. As the pressure in the homogeniser is increased the droplets are made smaller.

Particle Size

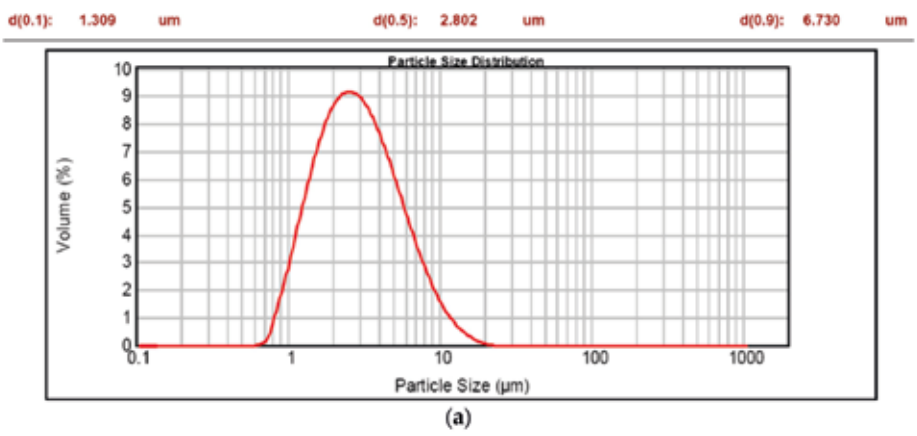
• Oil droplet size can be measured and a graph generated to show the distribution of particles in the liquid. The shape of the graph should be a bell shape.

• The peak of the graph shows what size the majority of the particles are.

• The majority of particles (95% – 99%) in a beverage emulsion should be between 1 and 5 microns.

• If the particle size is still too high we have to increase the pressure of the homogeniser.

• Particle size has a massive effect on the colour shade and must be carefully controlled. This is also the case for milled products such as Curcumin LS where instead of homogenising, we use grinding to reduce the particle size.



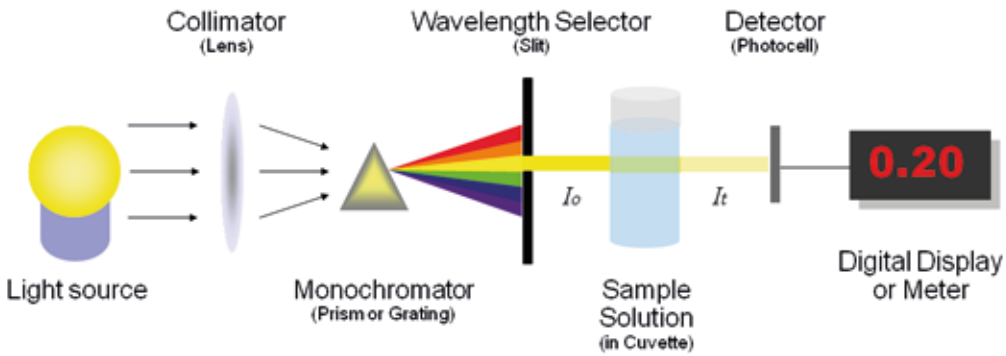
(a)

Costing and QC

- Size of the mix
- Ingredients in stock or ordered in
- Type of product
- Clean down
- Equipment requirements

QC

• Tested for pigment % content using a spectrophotometer. This measures light absorbed by the sample at a given wavelength for that colour and tells us the absorbance (ABS). Using this figure we can then calculate the pigment content.



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• Shade checked using the Xrite – the first time a product is developed we take a picture of it in solution using the Xrite. This is called the standard. Every time we make a new batch we take a picture of it and then overlay it on to top of the standard – this shows if there is any variation in colour and strength.

• The result is a DE figure. If the figure is 1.5 or lower then there is no visible difference between the two images and the product can pass QC.

Carotene Portfolio

Product	Strength Range
Beta Carotene E160a (i) and (iii) Powders and Beadlets	1% to 10%
Beta Carotene E160a (i) and (iii) Hi stability (cloudy)	0.2%, 1% and 2%
Beta Carotene E160a (i) and (iii) Hi stability (clear)	0.2%, 1% and 2%
Mixed Carotenes E160a (ii) Hi stability (cloudy)	1%
Beta Carotene E160a (i) and (iii) (cloudy)	0.5% to 10%
Beta Carotene E160a (i),(ii),(iii),(iv) (Oil Soluble)	1% - 30% depending on source
Carotene Source	**E number**
Synthetic Beta Carotene	E160a (i)
Mixed Carotenes (Excelvite) Alpha, Beta, Cis, Trans....	E160a (ii)
Natural Beta Carotene (Fungal: *Blakeslea trispora*)	160a (iii)
Algal Carotene (Algal: *Dunaliella salina*)	E160a (iv)

Notes
We recommend talking with our technical team prior to finalising an emulsion choice in order that all parameters have been discussed. For example, in some cases, additional anti-oxidants may be required for use in high UV environments to give the colour better protection.

Colour Possibilities:

• Safflower is clear in solution so cloudiness can be added separately (neutral cloud).

• Beta Carotene can give a range of colour shades from yellow to orange and even reds. This is achieved though dosage, pigment content, type of carotene and particle size. For example, a 0.3% Beta Carotene emulsion with a very small particle size will appear more yellow at a dosage of 0.05% but at 0.5% it will be more yellow/orange.

• A high % strength carotene will be more orange. E.g NC1605.

• Lutein is very bright yellow at low dosages and gets more yellow/orange at higher dosages and droplet sizes.

• Curcumin can be used for bright yellow but is not light stable so only in cans or opaque packaging.

• CFS blends of Safflower/Pumpkin and Red Beet or Anthocyanin give further shade possibilities in some beverages.

Left to Right in pH 3.5 Carbonated Beverage :
• E161b Lutein WSL @ 0.1%
• E160a Beta Carotene WSL @ 0.1%
• E160a Beta Carotene WSL @ 0.2%
• E120 Carmenic Acid WSL @ 0.1%
• E160a Beta Carotene 10% CWS @ 0.1%

Yellow - Orange Colours

• Yellow and Oranges are usually oil in water emulsions of Carotenes, Paprika or Lutein – pigments from the Carotenoid family. They can provide brighter, bolder colours with a variety of opacities.

• They can also withstand acidic formulations and have good heat and light stability when used in combination with antioxidants.

• Colouring foodstuffs such as Safflower, Carrot Extract and Pumpkin can also be used. These are less stable, with a shorter shelf life and provide more of a pastel hue

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Emulsions in Beverage

When choosing a colour for beverage applications, it is necessary to ask the following questions:

- What is the required packaging and shelf life requirements?
- Country of sale – EU, US
- Labelling expectations?
- Is a cloudy or clear solution required?
- What format is required, powder or liquid?
- What are the processing parameters of the beverage: pH, heat processing, co ingredients and additional flavours
- Additionally, for best results tests should be carried out in the final application by the Plant-Ex lab.

Milled Products

- Milled products work on the same principle as an emulsion – by reducing the particle size.

- Instead of breaking small oil droplets up, the mill grinds insoluble particles into smaller particles.

How it works:

- The head of the mill contains →10,000 small, very hard beads.

- When the mill is switched on, the head containing all the beads begins to rotate at very high speed.

- As liquid passes through the head of the mill it is bombarded by the fast moving beads. This breaks the particles (e.g TiO2 Powder) in the liquid into smaller and smaller pieces – reducing the particle size.

- A number of factors influence final particle size: head rotation speed, time of milling, bead size, temperature, product type and the rheology of the mix itself.

- Current mix size for Curcumin LS – 350 – 400kg with a milling time of approx. 2 days.

- Particle size is measured using PSA and Xrite

Advantages: light stability for Curcumin, increases optical brightness of TiO2, stabilises oil dispersions, darker Carbon Black dispersion

Other Applications

Emulsions can be used in a number of industries:

- For use in meat products, colour and flavour. Paprika emulsions and extract emulsions with essential oils and oleoresins such as Black Pepper.

- In flavouring formulations.

- Oleoresins and essential oils such as Ginger Extracts for use in savoury, confectionery and beverage applications.

- Spray dried for sports nutrition.

- Higher oil loadings in the emulsion are possible for non beverage applications, whereas beverages should use the lowest possible to avoid risk of ringing etc.

- Small particle size not so important for non-beverage applications so lower cost of production.

- Carotene emulsions are commonly used in dairy applications such a ice cream and UHT milk products.

- Some applications are sensitive to certain emulsifiers, particularly aerated products such as ice creams and marshmallow where Gum or Sucrose Esters are preferable to Polysorbates.

Milled Products	E number
Curcumin LS and derivatives	E100
Titanium Dioxide Dispersions	E171
Red Beet OS and other powder/oil dispersions	CFS
Carmine OS	E120
Annatto Bixin OS	E160b

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Encapsulation

A **microcapsule** is a small sphere with a uniform wall around it. The material inside the microcapsule is referred to as the core or internal phase, whereas the wall is sometimes called a shell, coating, or membrane.

Reasons for encapsulating:

• To increase the stability and shelf life of the product being encapsulated.

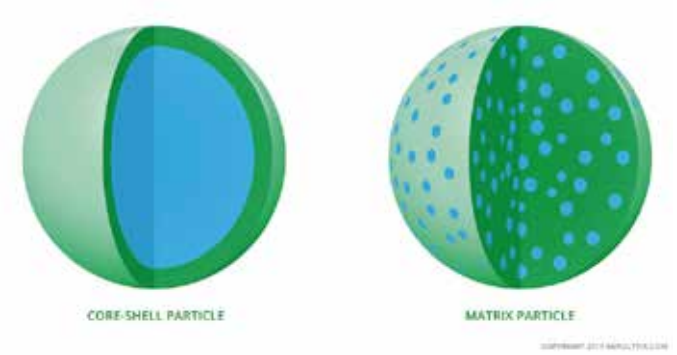
• Control the rate of release of the internal phase and how quickly the powder dissolves.

• In some applications the oil phase must be isolated from its surroundings, to protect the essential oil, extract or flavour aroma from oxidation.

• To prevent the release of volatile ingredients such as Acetic acid in spray dried vinegar powders.
Improving the handling properties of a sticky material such as Honey, Maple Syrup or Molasses.

Techniques we use:

• Spray drying
• Some milled products like Curcumin LS



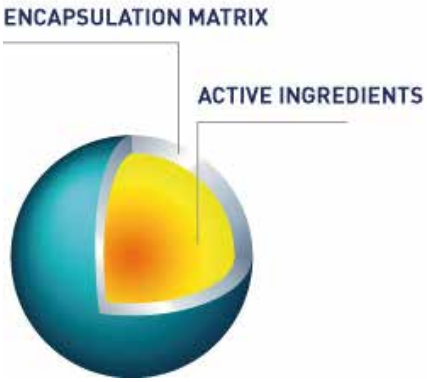


Table 1

COMMONLY USED TEST CONDITIONS FOR EMULSION STABILITY TESTING

Storage Conditions	Storage Period
Ambient temperature	25 °C for 3 years (or projected shelf-life of the product)
Elevated temperature	37 °C for 6 months and 45 °C for 6 months
Refrigerator	Approximately 4 °C for 3 months
Freeze/thaw cycles (5)	Approximately -10 °C to ambient
Cycling chamber	4 °C to 45 °C in 48 hours for 1 month
Light exposure	1 month exposure to north-facing daylight or light cabinet

• A select range of beverage emulsions will be subjected to the testing regime to ensure maximum stability and product performance before launch.

• Upon completion we will launch a new range of

Table 2

EMULSION PROPERTIES TO BE MONITORED DURING STABILITY TESTING

Property	Test Method
pH	pH meter
Viscosity	Rotational viscometer
Flow behavior	Oscillatory shear viscosity with a cone/plate rheometer
Tack/Texture	Extensional and compressional deformation
Color	Visual or colorimeter
Odor	Organoleptic
Specific gravity	Pycnometer
Separation	Creaming value - visual or instrumental
Conductivity	Conductivity meter
Droplet size distribution	Microscopic examination (image analysis) and instrumental
Preservation	Microbial challenge and/or assay
Vibration	Shipping test or shaker table
Active ingredient(s)	Chemical or bio-assay

Dispersions Vs Emulsions

• An emulsion is different to a dispersion.

• A dispersion is where a particle (insoluble) has been evenly spread and held in susupension throughout a liquid. It contains no emulsifier and cannot truly dissolve. For example – Carbon Black, Iron Oxides, Titanium Dioxide, Water soluble powders in oil (Red Beet OS).

• A dispersion can be milled to reduce the particle size, this improves the intensity of the colour dispersion and improves the stability.

• Stability of dispersions can be influenced by: viscosity of the carrier liquid and particle size. If this is not addressed, then eventually the particles will sink to the bottom of the container.

• It is advised to shake before use to prevent sedimentation and re-disperse the product.

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