High-Resolution, Comprehensive, and Quantitative Physical Stability Analysis of Cannabis Beverage Emulsions: Homogeneity, Formulation, and Shelf Life

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Food Formulations and Stability Concerns

Both liquid and solid formulations in the food & beverage field require some type of stability testing over time.

This is mainly practiced in order to maintain the physical integrity of the materials in terms of customer appreciation as well as the performance of the formulation when consumed.

This shelf life testing can be done visually and can take many weeks, months, or even years to provide accurate results.
Food Formulations and Stability Concerns

In the case of cannabis-based edibles and beverages, care must be taken to ensure consistency throughout a material in an effort to avoid a heterogeneous distribution of the active substance.

It follows that the stability profile of these materials should employ pharmaceutical-like testing to ensure that the potency of the formulas are consistent throughout the ageing period of the formula.
Formulation Stability – Industry Examples

Food beverage emulsions – testing/prediction of shelf life based upon phase separation and redispersion kinetics.

Pharmaceuticals – redispersion techniques that can re-disperse flocs, particle size distribution analysis, overall homogeneity. Sedimentation in adjuvants is an important aspect to quantify.
Monitoring Physical Stability – Static Multiple Light Scattering

Static Multiple Light Scattering can be used to monitor particle concentration and size changes inside of concentrated formulations (Turbiscan, Formulaction, FR).

Backscattering (BS) is a function of:
- $d$: particle size
- $\Phi$: particle concentration

Repetition of the measurement provides:
- $\Delta d$: change in particle size
- $\Delta \Phi$: change in particle concentration

Allows for quantification and prediction of destabilization kinetics such as creaming, flocculation, particle size, and volume fraction change.

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Emulsion Concentrate Stability Data

Even after 200+ days, sample is still opaque and homogeneous to the naked eye!

Negative evolution of BS signal at the bottom of the sample: a clarification

Positive evolution of BS signal at the top of the sample: a creaming
Emulsion Concentrate Stability Data

By averaging the light scattered in the bottom clarification region, kinetics of this zone can be obtained.

Increasing phase separation
Decreasing volume fraction

<table>
<thead>
<tr>
<th>Sample</th>
<th>BS% (initial)</th>
<th>BS% (50 d)</th>
<th>BS% (100 d)</th>
<th>BS% (190 d)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RT</td>
<td>71.02</td>
<td>69.29</td>
<td>66.34</td>
<td>61.60</td>
</tr>
<tr>
<td>38F</td>
<td>72.20</td>
<td>70.50</td>
<td>68.65</td>
<td>67.00</td>
</tr>
<tr>
<td>104F</td>
<td>71.37</td>
<td>68.03</td>
<td>66.87</td>
<td>65.33</td>
</tr>
</tbody>
</table>
Emulsion Concentrate Stability Data

The device software allows for mean particle size analysis or volume fraction calculations. By inputting particle size and refractive index data, the concentration of the bottom clarification layer can be calculated.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Φ (initial)</th>
<th>Φ (50 d)</th>
<th>Φ (100 d)</th>
<th>Φ (190 d)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RT</td>
<td>9.86</td>
<td>9.40</td>
<td>8.43</td>
<td>6.97</td>
</tr>
<tr>
<td>38F</td>
<td>10.40</td>
<td>9.85</td>
<td>9.24</td>
<td>8.62</td>
</tr>
<tr>
<td>104F</td>
<td>10.00</td>
<td>8.94</td>
<td>8.59</td>
<td>8.07</td>
</tr>
</tbody>
</table>

Particle size analysis (Malvern) shows a starting D50 of 1.33 µm. Refractive index values of the THC/CBD oil blend is 1.45. Volume of oil loading is 10% by volume.
Diluted beverages can be subjected to the same protocol to monitor shelf stability and dosage within regions of the sample.

This provides an assurance that the product is stable and consistent for 7 months, with a projection of well over 1 year.

<table>
<thead>
<tr>
<th>BS, % (initial)</th>
<th>BS, % (210 d)</th>
<th>Φ, % (initial)</th>
<th>Φ, % (210 d)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6.98</td>
<td>6.61</td>
<td>0.43</td>
<td>0.41</td>
</tr>
</tbody>
</table>
Conclusions

• Emulsions and beverages made with cannabinoid extracts can be monitored for their physical stability, quantifying destabilization and shelf life kinetics.

• The long term shelf stability of cannabis oil-based emulsions can be quantified with a non-invasive light scattering technique.

• Local volume fraction calculations can be easily performed in an effort to quantify the potency variations at various points in the sample, allowing for a standard QC evaluation of the cannabis oil concentration without the risk of sample modification due to aliquot sampling.

• Applicable to multiple types of formulations (drinks, concentrates, gels, oils) and types (flavors, dyes, emulsifiers, binders).
Future Work and Considerations

- Application to multiple types of formulations (drinks, concentrates, gels, oils).

- Use for different additive types (flavors, dyes, emulsifiers, binders, oil identities).

- Redispersion studies: quantifying the effects of physically shaking/disturbing a sample to return to its homogeneous state.

- Work to produce the same results in an accelerated manner (analysis time of days or weeks rather than weeks or months).

- Particle size studies: monitoring the mean particle size evolution for flocculating samples.
Acknowledgements

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