



3 STEPS CHARACTERIZATION OF DISPERSION

DISPERSIBILITY | SHELF-LIFE | REDISPERSION

What is formulating?

Formulating is the science of mixing different components to achieve desired properties or specifications. The ingredients are often not compatible, and they must be mixed following a specific strict procedure to obtain a uniform and durable structure. This task is even more challenging when formulating with natural ingredients. The constant need to create new formulations or reformulate existing formulations requires thorough monitoring of formulation properties.

What is the science of formulating?

Formulation composition can be summarized as follows:

- **Liquid continuous phase**, water is the most commonly used solvent but not limited to.
- **Dispersed phase**: liquid, solid, or air. The particularity of this phase is that it is non-compatible with the continuous phase. The particle/droplet size can vary from a few nm up to visible particles and the concentration ranges from ppm up to 95%. This phase is the one that provides the desired end-user properties to the formulation (moisturizer, coating properties, drugs, nutrition...).
- **Stabilizers** are one of the key components of the formulation. They are essential to control the dispersion, achieve the desired

particle size, and maintain a “stable” formulation. Nowadays, the number of possibilities is very large and depends on the application. Surfactants have been and remain the most commonly used stabilizers. They either come from chemical synthesis or natural, renewable resources. Recent studies push towards using new polymers and biopolymers, proteins, and even other particles as stabilizers.

- **Additives**: are added to adjust side properties, nevertheless essential for the user appreciation (viscosity, scent, color, texture...). This additive can have an affinity with either the continuous phase, the dispersed phase, or the interface.

Formulations can contain over 50 different ingredients and the fine adjustment of the composition is the key to a high-quality formulation. During the development phase, it is of high importance to also consider the process, packaging and dispensing, storage conditions, and usage conditions. The formula needs to be adapted for all these external parameters.

FORMULATION CHARACTERIZATION STEPS

Whether you are formulating paints, electronic slurries, cosmetics, food, or pharmaceutical suspensions, there are 3 crucial steps, linked to the formulation lifetime, to be considered for a complete formulation characterization.



Dispersibility: Foundation stone of the formulation

Dispersibility is the ability to "suspend particles" (either solid, liquid, or gas) in the continuous phase, in a way that allows them to be equally distributed in the whole volume and preserve their original size. It can be considered as the foundation stone of the formulation process, and it is affected by:

- Solvent affinity with the particles
- Interactions between the continuous phase & stabilizer
- Interactions between the stabilizers & particles
- Stabilizer coverage of the interface
- Preparation process: mixing tool used, time and speed of mixing

Poor dispersibility will have a significant impact on final product specification as the particle/droplet size and homogeneity will affect overall stability and so durability in time of colors for inks, polishing efficiency for CMP slurries, taste for flavored food emulsions...

Monitoring the dispersibility will save time in formulation development and decision-making. The dispersibility ratio (Dr) is the most appropriate parameter to evaluate if the particle size in suspension corresponds to the so-called "primary particle" size.



Stability and Shelf Life

Well-dispersed particles, within the required size range, must remain in that state for the time of use of the formulation. Thus, stability is the next important parameter to consider. Indeed, stability is the capacity of the formulation to maintain the dispersion state under conditions of storage and use, with predefined pass criteria and over a given time. Most, if not all, dispersions are thermodynamically unstable, and with time destabilization is to be expected. There are various destabilization phenomena with different origins that can be classified into two main categories

- Size growth

Agglomeration-aggregation, coalescence, Ostwald ripening

- Particle/droplet migration

Sedimentation, creaming, phase separation, demixion...

Thermodynamically speaking, a "stable" formulation does not exist (except for microemulsions). However, a certain degree of destabilization (usually not visually observed) over a given period is acceptable. The acceptable variation and "time period" are defined depending on the final application and can vary from minutes (inhaler, vaccines...) up to several years (cosmetics or paints...). Hence the need to constantly monitor and quantify the extent of occurring phenomena.

This can be done using a Turbiscan Stability Index, which allows quantifying and ranking formulations at a given time based on global destabilization.



Resuspension: "Shake well" strategy

For some applications, the inevitable destabilizations are reversible. Paints, injectables drugs, and dairy drinks are just a few examples of such formulations and are required to be shaken before use.

- *Would the manual shaking be enough to well re-disperse the formulation?*
- *How long should it be shaken for?*
- *What method to use: stirring, shaking, mixing, ultrasound bath?*
- *Can the formulation retrieve its initial dispersion state?*

All these questions must be considered, and the recommendations made to the user. Once the sample redispersion is achieved, the formulation is renewed and parameters like size, stability, and re-dispersion can be studied again and again. It is of great interest to understand if the formulation can retrieve its original state and properties. How many of these "life cycles" the formulation can support before destabilization is irreversible (and upon what storage conditions)?

TURBISCAN: THE GLOBAL APPROACH

TURBISCAN technology is the most complete solution for formulation characterization: from dispersibility, average particle size, stability quantifying and monitoring, to redispersion studies.

Dispersibility and particle size are measured with a single measurement (30 seconds and online modules), without dilution or sample preparation.

Stability is characterized up to 100 times faster than the naked eye and provides a full understanding of the destabilization mechanisms, stability ranking, and comparative stability prediction.

Resuspending capabilities can be studied, and the impact of the mixing energy and time can be evaluated and optimized to offer the best quality before use.

Conclusion

TURBISCAN is a unique tool that provides a full characterization of your formulations with an extremely wide range of applications



ELECTRONICS



POLYMERS



RAW MATERIALS



PHARMACEUTICAL



HOME & PERSONAL CARE



FOOD & BEVERAGES



AGROCHEMICALS



PAINT & INKS

TURBISCAN RANGE



**THE WORLD LEADER
IN SHELF-LIFE & STABILITY ANALYSIS**