Analysis of cosmetic creams – stability tests

OVERVIEW

The purpose of this experiment is to introduce to the use of stability testing procedure. Analysis will be accomplished by using Turbiscan - Multiple Light Scattering equipment.

INTRODUCTION

Traditionally, emulsions have been defined as dispersions of macroscopic droplets of one liquid in another liquid, with a droplet diameter approximately in the range of 0.5-100 µm (P. Becher, Emulsions: Theory and Practice (Reinhold, New York,1965). According to the definition of International Union of Pure and Applied Chemistry (IUPAC) (1971), "In an emulsion liquid droplets and / or liquid crystals are dispersed in a liquid".
Creams are semisolid emulsions intended for external applications. They are often composed of two phases (sometimes of more phases). Oil-in-water (o/w) emulsions are most useful as water-washable light creams and bases, whereas water-in-oil (w/o) emulsions are emollient, creamy emulsions and cleansing agents. The active ingredient is often dissolved in one or both phases, thus creating a three-phase system.
The purpose of the stability testing of cosmetics is to ensure that a new (or modified) product comes across the intended physic-chemical quality standards as well as functionality when kept under appropriate (standard) conditions. Stability testing is simply an experiment in which you create a batch of your cosmetic formula and place samples of it at different environmental conditions for a fixed period of time. These conditions vary in temperature, light levels, humidity and are meant to simulate what will happen to the product during its life cycle (cosmetic life span).
To simplify, at selected intervals evaluation of samples for various physical, chemical and performance characteristics is made in order to see how they have changed. If the deviations are minimal according to company standards (policy), then the formula is said to have "passed" stability testing. This means you can have confident that when the formula is shipped to stores and ultimately customers, it will still be as good as when it was first manufactured.
The general assumption in stability testing is that increasing storage temperature speeds up any aging reactions that will occur. A handy rule of thumb is that a sample stored at 45 ^{o}C for 8 weeks is equivalent to one that is stored at room temperature for one year, whereas stored at 45 ^{o}C for 12 weeks equals to two years shelf life . This is not an precise predictor, but is good enough for the purposes of cosmetic products.
Because of the wide variety of cosmetic products "standard" stability tests cannot be recommended. Thus, specific tests may be developed in order to address new or unusual technologies, or to be adapted to products having extended shelf lives.

When do we perform stability testing?

Since the companies are making hundreds or thousands of prototypes during the process of making new products, it would not be practical to run a stability test on all of them. Here is a short list of some of the most important times to conduct a stability test.

1. New prototypes – Whenever you make a new formula and are satisfied with the way it performs, you will want to do a stability test to ensure that it will stay together(do the ones that work the way you want).

2. New raw materials – Whenever you have to change the fragrance, color, or other raw material in a formula, you will have to do a stability test to make sure there are not unacceptable changes. Also, when you have a new raw material source (or supplier) you will want to run a test.

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3. New manufacturing procedure – Manufacturing is always trying to find faster ways to make formulas. This often means they change some order of addition or shorten mixing time. Whenever changes like these happen, it could affect your formula. Run a stability test to see if the change is acceptable.
4. New packaging – Cosmetic products change their look almost yearly so packaging is constantly being modified. Whenever you get a new package, you'll have to determine if the formula continues to be compatible. Stability testing helps ensure that it is.

How you do stability test a cosmetic?
Here are described approaches to predicting how well cosmetics will resist common stresses such as temperature extremes, humidity and light. Typically, manufacturers determine whether to perform such specialized testing based on the vulnerabilities of the particular cosmetic product and its anticipated shipping, storage display and use conditions.
Here is a basic format you can follow for conducting a cosmetic formula stability test.
Step 1 – Make your batch. Calculate how much to make based on the number of samples you'll be using for the test. It's a good idea to make 30-40% more than you think you'll need.
Step 2 – Fill your samples. Ideally, you'll have the correct packaging but don't count on it. When appropriate, fill glass jars with the product along with the finished package. In stability testing, you want to do both glass and packaging if possible. The number of samples depends on how much testing you are doing but at minimum you should have 2 samples for each storage condition.
Step 3 – Take initial readings. Once you have a sample filled test it for all the characteristics you're going to evaluate later. The exact tests depend on the product but minimally you'll want to record notes about the appearance, color and fragrance. You'll also want to take pH and viscosity readings. For aerosol products you will test spray patterns.
Step 4 – Put samples at different conditions. Stability testing requires different temperature and light conditions. Some standard temperatures include 50, 45, 37, 25 (RT), and 4 ºC. You'll also want to conduct a freeze/thaw stability test which involves cycling (three to five cycles) your product through 24 hours of freezing (-10ºC) then 24 hours of thawing (25ºC). This puts emulsions under a tremendous stress and, if it passes the test, indicates that you have a really stable product.Different lighting conditions involve a fluorescent light box and a natural light box (to simulate sunlight). Of course, the product must be stored at 25 ºC for a period of one year. A good control temperature is 4 ºC where most products will exhibit excellent stability. The product should also be subjected to -10 ºC for three months.
Step 5 – Evaluate the product. Samples should be evaluated at the following intervals. 2 weeks, 4 weeks, 8 weeks, 12 weeks, and 52 weeks. Only the RT, 37C and 4C samples will be evaluated after one year. The highest temperature samples and the light exposed samples only need to be evaluated for the first three test intervals. The evaluation tests should be the same ones you conducted when taking your initial readings.
Step 6 – Determine stability. After 8 weeks you can confidently decide whether your formula is stable or not. Nearly all products will exhibit some change so it will be up to you (and your boss) to decide whether the product passed or For all the above mentioned tests you should monitor the color, odor / fragrance, viscosity, pH value, and, if available, particle size uniformity and/or particle agglomeration under the microscope.
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Common Additional Test Procedures

Centrifuge testing: The dispersed phase (of an oil-in-water emulsion) has a tendency to separate and rise to the top of the emulsion forming a layer of oil droplets. This phenomenon is called creaming. Creaming is one of the first signs of impending emulsion instability and should be taken seriously. A good test method to predict creaming is centrifugation. Heat the emulsion to 50 ºC and centrifuge it for thirty minutes at 3000 rpm. Then inspect the resultant product for signs of creaming. This test is an absolute necessity for those products that contain powders of any kind such as liquid/cream make-up.

Light testing: Both formulas and packaging can be sensitive to the UV radiation. All products should be placed, in glass and the actual package, in the window and if its available a light box that has a broad-spectrum output. Place another glass jar completely covered with aluminum foil in the window to serve as a control. All too often we will see significant discoloration of the product and sometimes of the package also. This discoloration may be due to the fragrance or some other sensitive ingredient. Usually all that is needed is the addition of a UV absorber (e.g. 0.1% of benzophenone).

Mechanical shock testing: In order to determine whether or not shipping movements may damage the cosmetic and its packaging mechanical shock testing is often conducted. Vibration testing (e.g. on a pallet shaker) can help to determine whether de-mixing (separation) of powders or granular products is likely to occur.

EXPERIMENT

Cosmetic cream preparation

Please prepare the cream according to the following composition:

Composition	Main components	Quantity [%, ± 0.01]
Creagel® EZ 7	Polyacrylamide Hydrogenated polydecene Polyoxyethylene lauryl ether	9.8
Alphaflow® 20	Hydrogenated polydecene	16.7
Distilled H_2O	Distilled H_2O	73.5

The Creagel EZ 7 as well as Alphaflow 20 should be weighed into a 200 ml beaker. Afterwards, distilled water has to be added. The oil phase has to be dispersed in the aqueous phase by using a magnetic stirrer (100 rev/min, 20 minutes).

Stability test

After preparation of cream please store it in a climate chamber at 50ºC, 75% humidity, light 7500 lux/ 1.1 W/m^2 (UVA) for 2 h. The instructor will give you a quick run-down of this instrument before you get started.

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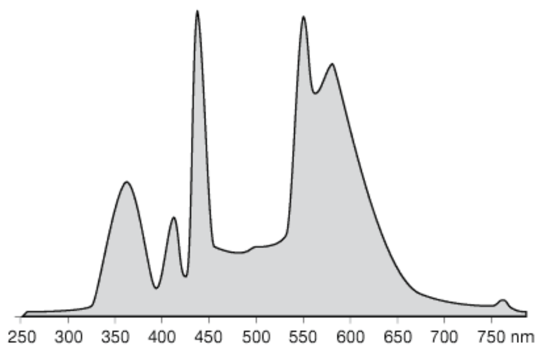
BINDER constant climate chamber KBF LQC 240



BINDER Light Quantum Control (LQC)   Light spectrum

/http://www.binder-world.com/en/products/constant-climate-chambers/kbf-lqc-series/kbf-lqc-240/

Stability test by Multiple Light Scattering

The stability measurements should be performed immediately after the preparation of emulsion and after the stability test. The stability was examined at constant temperature (25°C) by multiple light scattering using Formulaction Turbiscan™ LAB. Turbiscan is easy to operate because no dilution is required (no sample preparation) . Various forms of instability such as creaming, sedimentation, flocculation and coalescence could be correctly identified, which significantly enables to shorten the aging tests and development process of new products. The measurement principles based mainly on multiple scattering of light. A pulsed near infrared light source (at a wavelength of 880 nm) was used. The light which goes through the sample is received by the transmission detector and the light scattered backward by the sample goes to the back scattering detector. The emission intensity depends mainly on three parameters: the particle diameter, volume fraction of dispersed difference of the refractive index of the continuous phase and dispersed phase.

The obtained formulations should be placed into cylindrical glass tubes and submitted to Turbiscan Lab® Expert stability analysis. The instructor will give you a quick run-down of this instrument before you get started. The analysis of stability will be carried out as a variation of back-scattering (BS) profiles.

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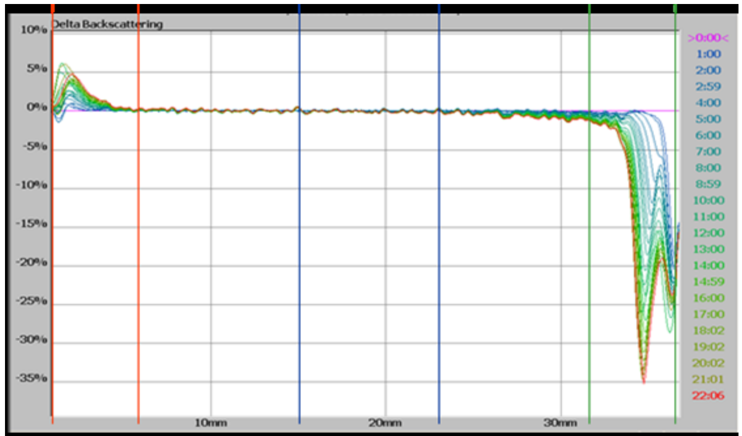
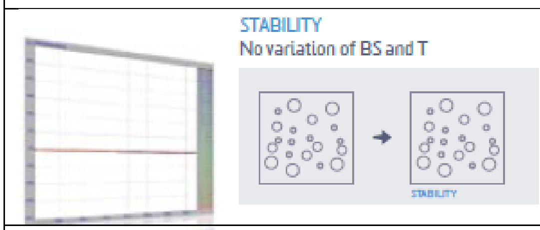
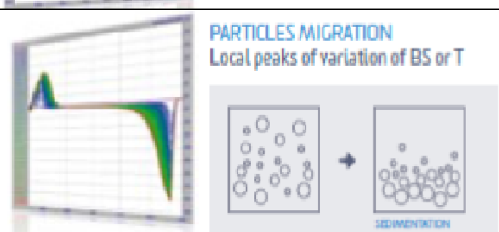
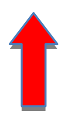
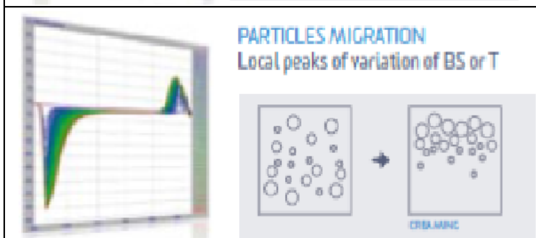
Examples of pictures that can be recorded for destabilization processes (creaming, sedimentation, flocculation, coalescence):



	Bottom part	**Middle part**	**Top part**	**Stabilty**
	**No changes**	**No changes**	**No changes**	**stable**
		-		sedimention
		-		creaming

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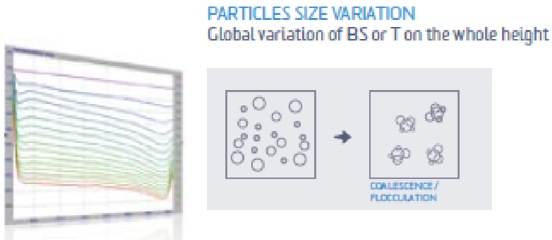	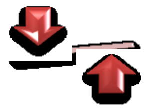			coalescence/ floculation

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International Federation of Societies of Cosmetic Chemists, IFSCC Monograph, Number 2*: The Fundamentals of Stability Testing*, Michelle Press, 1992 (http://www.ifscc.org/pubs.htm)

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