Stability Testing…So What's Adequate?

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Marketing has handed down (often on stone tablets) their product profile with their unreasonable and silly demands and you, being the inventive scientist, have created a product that has met them all! What more could anybody ask for? All that is needed is some safety testing, some micro challenge testing, and maybe a little claim support testing. To the uninitiated, the job of the cosmetic formulation chemist ends here. But most definitely is not the case! There is still one of the most crucial elements to be addressed…the bane of our existence…stability testing! Actually this is the wrong attitude to have. Stability testing is our friend…our very best friend! It will prevent our company from producing, packaging and marketing a product that is unstable. Isn't it far better that we know of the product instability before it gets into the hands of the consumer and the fine reputation of our company is ruined? Absolutely! Let us now design a stability test that will predict long-term shelf life of our products (emulsions).

At What Temperatures Should We Conduct Our Testing?

This is always a major point of contention and debate. We all remember the old adage that the reaction rate doubles for every 10^{o}C rise in temperature. Thus high temperature testing is used as a predictor of long-term stability. Most companies conduct their high temperature testing at 37^{o}C and 45^{o}C. Conventional wisdom says that if a product is stored at 45^{o}C for three months (and exhibits acceptable stability) then is should be stable at room temperature for two years. I have found that this is a good and reasonable prediction. Of course the product must be stored at 25^{o}C for a period of one year. A good control temperature is 4^{o}C (refrigerator). Most products will exhibit excellent stability at this temperature and thus the product can be compared to the high temperature samples to ascertain if any changes in color or odor have taken place. Some people feel that if testing at 45^{o}C is a good predictor then why not use an even higher temperature. I know that a few companies test their products at 50^{o}C and even 55^{o}C for one to as long as three months. As a general rule I disagree with this approach. When the temperature rises to 50^{o}C you are now approaching the melting point of the fatty alcohols (cetyl and stearyl) that are used to stabilize emulsions and the emulsion may exhibit product instability prematurely. In actuality, the emulsion may have a shelf life of two or even three years but "failed" the 50^{o}C test. I would propose that when these temperatures are employed as test stations, the results are not used as decision-makers but are only used to give a higher measure of confidence if the product passed this tough and unfair test. The product should also be subjected to -10^{o}C for three months.

Cycle Testing

The product should pass three cycles of temperature testing from -10^{o}C to 25^{o}C. Place the product at -10^{o}C for 24 hours and place it at room temperature (25^{o}C) for 24 hours. This

completes one cycle. If the product passes three cycles then you can have a good degree of confidence in the stability of the product. Some people suggest five and even ten cycles. If you really want to subject the product to a more rigorous test then use a -10°C to 45°C five-cycle test. I suggest that the product go directly from the oven at 45°C into the freezer at -10°C. This puts the emulsion under a tremendous stress as part of the product is frozen and part is very hot and fluid. The emulsion droplets are deformed and try to break. Additionally, if a lamellar liquid crystal gel network is being used to stabilize the emulsion, then it too will be deformed and emulsion breakdown may be the result.

Centrifuge Testing

As we know emulsions always contain materials of differing specific gravities. Generally speaking the water phase components have a specific gravity of approximately one while the oil phase components have a lower specific gravity (0.8-0.9). Thus the internal/dispersed phase (of an oil-in-water emulsion) has a tendency to separate/agglomerate and rise to the top of the emulsion forming a layer of oil droplets. We give this phenomenon the name creaming. Creaming is one of the first signs of impending emulsion instability and should be taken quite seriously. A good test method to predict creaming is centrifugation. Heat the emulsion to 50°C 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 (titanated mica, bismuth oxychloride, nylon, polymethylmethracylate, etc.). The ability of the emulsion to suspend these powders at high temperature and stress (centrifuge) must be determined if you are to have any degree of confidence in the long-term shelf life of the product.

Package Testing

While the formula may indeed be stable (have an adequate shelf life) if sold in a glass container with a tight fitting cap and a good liner, it may have an unacceptable stability in the actual package that is offered for sale. For this reason all testing should be done in glass and the commercial packaging. In this way you can determine if the cause of product failure is the formula or the package. Weight loss evaluation is one of the most important tests that must be conducted. This testing (performed in the commercial package with the cap torqued to 100% of target torque) is done at room temperature and at 45°C for a period of three months. The weight loss should not exceed 1%/month for the package to be considered acceptable. A variation of this test involves the torque testing of the package. Run the weight loss study with packages adjusted to 75%, 100% and 125% of the target cap torque).

Light Testing

It is well known that when we go outside and are exposed to the sun and its UV output, significant damage to our bodies can and does occur. Thus we rub on sunscreen to absorb this UV energy and protect ourselves. All too often we forget that our formula and package can also be sensitive to the ravages of UV. All products should be placed, in glass and the commercial package, in the window and if its available a light box that has a broad-spectrum output. I like to place another glass jar covered with aluminum foil in the widow 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 designed for this purpose. The Benzophenones seem to work particularly well in this regard, at use levels as low as 0.1%.

What Do You Monitor?
Some of the parameters that we monitor are quite obvious:
pH
viscosity
color
odor

Additionally we should perform assays for preservatives and actives (sunscreens, zinc pyrithione, etc.) if present. Probably one of the most important tests is often the least used: microscopic examination. Photomicrographs of the product should be taken and stored for future comparison. The product should then be examined for particle size (as a function of time and temperature) and particle size uniformity and compared to the initial photo. If any significant change is noted, the product will most probably be unstable and reformulation should begin with all due haste. Very often this examination will predict stability when all other observations have failed to do so. Additionally, microscopic examination may reveal agglomeration of particulates or signs of crystallization of some active or preservative. A final comment regarding measurements. While all measurement should be taken at room temperature, it is also advisable to measure the viscosity at 45^{o}C. If the product has significant viscosity (at least 1000cps) at this elevated temperature then this should give you a high degree of confidence that the product will be quite stable for several years with regard to physical separation (assuming there is no ingredient interaction or degradation).

So now that you have spent the time and performed all of these tedious, but necessary tests, your product is now ready for the marketplace. Well not so fast! Many years ago I developed a facial cleanser cream and subjected to all of the above tests, which it passed with flying colors. I confidently told our marketing brothers (and sisters) and my management that they could go ahead with production and sale of my masterpiece. A few weeks after shipment of the product into the field for consumer testing, some oil bleeding was noted and it was getting worse by the day. Where did I go wrong? What did I overlook? Was it a manufacturing problem? No it wasn't! The answer to this painful experience was that when the product was cooled (it was the winter) and shaken (shipped by truck) synerisis developed due to the high internal oil phase. This could have been predicted if the product was placed onto a pallet shaker and cooled (and heated). For those of you who do not have a pallet shaker I suggest that you drive around with the product in the trunk of your car for a few weeks. Another variant of this test is to ship your product via UPS ground service around the country and then back to yourself for inspection.
Stability testing is often tedious and not very glamorous, but without it you have no product to sell!