Is Stability Testing of Consumer Products Really Necessary?

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Knowledge of formulating any product is successfully acquired only after considerable experience, and a carefully formulated consumer product, cannot be issued with confidence unless controls have been carried out to confirm, that the risk from breakdown during processing and use is low. Stability testing previews the various characteristics of a formulated product and is an important function in any consumer industry.

Testing the stability of a consumer product helps one understand the ability of the formulation to maintain its physical appearance, aesthetic appeal, its odour profile, and it's functional and chemical characteristic. Stability tests are conducted under controlled conditions designed specifically to give an early indication and forewarn the formulator about the problems that may occur during the life of the product.

The stability data, recorded during the stability testing procedures guides the formulator develop and design a superior product that will continue to be as aesthetically appealing to the customer and performing its varied functions as envisioned at the beginning stage of product development. Stability testing does not guarantee, generations of a problem-free formulation, but can definitely help create a strong foundation based on sound scientific facts for evaluation of impending troubles in any product. This is true, particularly on consumer preferences and perception, integrity, and product characteristic that are detrimental to the product.

The main objective to conduct a stability test is to confirm the formulation that is to be marketed has adequate shelf life, under conditions in which it is to be sold. We know that every product produced undergoes continuous change either aesthetically, physically, functionally or chemically over a time and eventually becomes unsatisfactory for consumer use. A shelf life or product life is defined as "that time during which the product complies with the set standards or specifications, when stored under market conditions."

Strictly speaking, exhaustive tests should be carried out on every factor involved in the formulation to avoid problems. The main objective of the tests should be clearly identified in the beginning itself, so that testing is carried out without waste of valuable time, energy, effort and efficiency. However, practically it is near impossible to carry out all analytical tests, on every chemical that may be present in a typical formulation. Stability testing and its limits are optimised and are carried out to assess and evaluate a product. The testing is carried out not only in cases of change in formulation, or raw material, but also in changes of equipment's the manner and method of manufacture or filling.

Stability testing of samples in an industry is a continuing process during all stages of product development. It often helps the formulation development chemist identify problems likely to arise in future and enables one to take corrective action, saving valuable time and development resources. Sometimes even rough evaluation or screening of different formulation can indicate problems (viz., Separation of oil and water phases in an emulsion formulation, changes in colour and odour, formation of turbidity or precipitates) and help identify a formulation with many fewer difficulties.

As the process of product development is continuous, optimisation of formulation also continues to finally yield a refined formula based on experience obtained by rough testing and screening carried out on the products. A detailed study is conducted on the final formulation selected, to evaluate as many potential problems that can occur. At least two pilot batch samples, prepared by use of, different lots of raw material should be put on stability testing.

Product development processes establish a near stable formulation, but testing may be required to approve usage of alternate raw material source. Although the raw material from different supplies may have identical chemical name, their chemical feedstock, and processing parameters may not necessarily be the same. A raw material from one supplier cannot be automatically replaced into the formulation developed and approved by use using an ingredient from a different supply source. The outcome of such a change may seem very minor, and without significance, but confidence in the product is only established by stability testing.

Although stability testing of pilot plant samples is important, a change in the manufacturing procedure, due to change in the scale of manufacture, can also affect stability. After all, working in a pilot plant with kilogram amounts, is entirely different from working in an actual production site with several tons of material. The changes in the order of raw material addition, either to reduce processing time and operator convenience, changes in heating and cooling rates,

due to heat transfer of larger batches and changes in shear rate, due to different mixing conditions can affect stability that may not be evident in pilot plant batches.

The best way to evaluate the impact, of manufacturing changes that occur on a product, is to produce a trial batch and subject the sample so produced in a regular plant to stability test before commercial launch. Usually the sample from the initial batch of a new product is subjected to detailed stability testing so that the impact of actual, production process condition is under study.

Pilot plant samples and production samples at the beginning are necessarily put to stability studies. However, it is also essential to test samples that are in regular production. Stability testing should be an ongoing process, as over a period, unsuspected changes in raw material quality, method of handling, etc., may occur during regular production. To rule out such problems, samples drawn periodically should be put on stability tests at real market conditions and changes in various aspects examined.

When the formulation and the method of manufacture are kept constant, then any changes in the packing material can also cause stability problems. Not all packing is the same. Glass packing, plastic packing, paper, and hardboard behave differently with different products. Even among them, glass, plastic, and paper have different properties regarding their colourfastness, oxygen permeability, resistance to heat, light and water vapour. A change in the existing packing option or even change in the supplier of the packing material demands stability testing and evaluation, to confirm that the product will remain stable in the new pack.

BASIC PRINCIPLES OF STABILITY TESTING

All stability-testing procedures are so designed, that the data, obtained is useful to alert one to potential formulation problems in the shortest possible time. This is possible only when samples of the formulation / product are placed under experimentally induced conditions, that accelerate changes, occurring under normal storage conditions. However it is to be remembered that greater the degree of acceleration, the greater is the risk of happening of changes, that will never be observed in actual use of the product. A properly optimised test condition is all the more necessary, so that only those changes are observed that can actually happen during the shelf life of the product.

The usual and widely followed procedure for stability studies requires storage of samples at elevated temperatures, freezing temperatures, at elevated humidity levels and exposing the samples to varying types of light source that can come across during the life of the product. Some products may require vibration tests conducted for some fixed amount of time with different frequencies and amplitude of vibration to determine changes liable to occur (for example, to assess the stability of emulsions or foams). Storing a product at elevated temperature is very critical, as it is believed that a 10°C rise in temperature approximately doubles the rate of chemical reaction.

This helps one to note down problems earlier than they would occur at normal room temperature. However the main drawback of this method is that higher temperature of storage can trigger off chemical reaction that normally does not occur during the life of the product at lower room temperature. Nevertheless, this method is very useful to indicate the stability of the product, as a product that is stable at higher temperature is certain to be chemically stable at normal room temperature.

Samples are stored at room temperature conditions of 20°C/25°C ambient humidity levels as standard samples. It is preferable to store samples at constant temperature — 20 or 25°C — rather than at uncontrolled room temperature, as otherwise the temperature and humidity levels are subject to wide fluctuations due to seasonal and geographical variations. Moreover, the standard samples stored at constant room temperature would also represent stability results more accurately when compared to samples stored at elevated temperatures.

The most common storage conditions include the following: 20°C, 25°C, 35°C/37°C, 45°C, 50°C or 55°C ambient humidity. It is however important that samples are stored at a minimum of three different temperatures, spaced mildly from each other to render the results more accurate. Storage studies at higher temperature of 60°C, 70°C or 80°C are conducted only for specific samples to provide evidence of stability and for short period of a few days or weeks. In normal temperature stability studies, the relative humidity maintained is usually 80%. At higher humidity, there is always a danger of mould and fungal growth, which is a rare occurrence in actual high-humid conditions.

Tests at higher humidity are conducted for testing the stability of the packing materials rather than the product content. It helps one to observe the barrier properties of the container or the pack. Products stored in a container or packing may be affected adversely by the humid atmosphere, indicating the deficiencies of the packing used to protect the product packed from the environmental humidity. In some case high humidity may retard the changes that occur normally by reducing loss of water occurring otherwise at normal storage conditions.

Samples stored at standard conditions provide valuable data and information to determine the stability and compatibility of the product at normal market conditions. However, in a market with both high temperature and high humidity the prediction of shelf life is much more difficult. It is always preferred to store samples under actual market condition of temperature and humidity in such cases. It may be also necessary to carry out separate testing, at several set conditions of temperature and humidity depending on the climatic condition of the places where the product is to be marketed. It is also possible that certain products tested for stability will exhibit different shelf life in different market environment.

Freezing tests at low temperature (at sub-zero temperature) is carried out usually in semi-solid, liquid, emulsions, creams, etc. It provides data on the stability of the emulsions, the tendency to solidify or crystallise, clouding and turbidity. The data on the physical changes observed on the test sample at room temperature, when compared to a control product at standard normal ambient temperature helps the development chemists optimise the formulation.

Light exposure can cause changes in product quality. Daylight varies with location, season, and climatic conditions. Exposure to direct sunlight is not advisable as products in a real life situation are rarely exposed to such intensity of light. A cabinet replication of normal daylight can however be used for such purposes. Test samples are compared with similar light fast, stable control samples and assessed for any change in colour, appearance, and other quality parameters. A practical way to test a sample for light stability is by storing control samples of satisfactory light stability along with the test or experimental sample under market storage conditions and comparing the results or changes observed in either of them.

The important part of stability testing is examination of samples stored to make observation and thereby evaluate the product. Most of the tests during the process of evaluation may physically affect the sample. Moreover, sample tests may have to be repeated when unexpected results are observed. Sometimes special problems may arise which may require one to carry out tests not envisaged at the start. When samples are removed for observation, the product remaining in the container is to be destroyed and a new container opened for any subsequent tests later. To take care of all such eventualities sufficient number of samples, more than the calculated amount should be put on storage. This would ensure that at the end of the test period enough samples are left for evaluation.

Control products should always be put to stability tests along with experimental sample. In stability tests on completely new products, that is when a control product is not available, a related or a competitive product, known to exhibit satisfactory stability provides valuable data in the evaluation. In testing packing material, samples packed in inert impermeable container are used, as control sample. On products that have a modified formula / process, or in tests with new packaging material the original product or package is used as a control sample.

The samples put on stability testing and storage is examined at set time intervals. The duration of the testing time entirely depends on the condition and on the latest findings of the tests. Although the time is set at the beginning of the stability testing, it is modified according to the test progress. If the product is less stable than envisaged, the time interval between subsequent examination may be shortened. Similarly, if samples stored at higher temperatures show no change, the frequency of examination may be reduced, in case of samples stored at lower temperatures. Typically, the duration set for testing is 1, 1½, 3, 4½, 6, 9, 12 or 24 months at the maximum.

Room temperature	Expected product shelf life (6, 9, 12 or 24 months)
04°C	Expected product shelf life (6, 9, 12 or 24 months)
37°C	3 to 6 months
45°C	1 to 3 months
37°C/80% humidity	1 to 3 months
Sub-zero temperature	Duration, specific to product tested

At each checkpoint, the critical properties are observed and assessed subjectively. These include appearance, colour, odour, texture, viscosity, weight-loss, density, etc. Changes if recorded on a scale of 1 to 5 are much better than that of recording purely subjectively. Chemical tests that are conducted include pH, microbial challenge tests, percentage of actives, functional attributes, etc.

When the product formulated contains an active ingredient then the degradation/ decomposition of the active or even the interaction of the active with the other product present or the packing should be investigated. The analytical methods selected for use should be indicative to confirm the stability of the ingredient used. Preservative challenge tests are to be carried out to ensure an effective preservative system used, to rule out degradation.

Samples containing an antimicrobial, as an active ingredient, should be tested for antimicrobial efficiency of the product. The product formulated with the antimicrobial, must continue to show adequate antimicrobial activity, at the end

of the product shelf life. Packing subjected to storage studies should be examined for stress-cracks, blistering or detachment of inner lining or lacquer on cans and tubes, any blockage on the spray valves, label attachment, etc. Sometimes the ingredient in the product is absorbed by plastic and this should be tested. Changes observed in the product as well as the containers are to be noted. It is to be emphasised that sufficient amount of samples, should be put on storage to ensure that at the end of the test period enough samples are left for evaluation.

Packaging stored at higher temperature and humidity may exhibit effects like loosening/rupture of cap/wads, exfoliation of laminates and improper adhesion of labels stuck on the outside. If the barrier property of the package is faulty, then the effect would be demonstrated by the difference between experimental sample and control samples stored in impermeable container. Similarly adverse effects of moisture atmospheric oxygen, loss of perfume/flavour or volatile constituent of the product, leaching of constituents of the container, corrosion of metal tubes, etc., can also be noted and compared with that of the control.

The interpretation of the observation that are essentially a comparison between test sample and control is relatively simple. The observation should not only be limited to the formulation, but also changes that may occur due to the formulation, or on interaction of the packaging with the product. Any weight-loss, change in packing plastic colour, odour profile, and package related observations are also equally important.

The main objective of the observation is to get as much information as practically possible about product performance, to establish a product specification/characteristic, etc., over a period. The stability testing procedure although not cent percent correct has a very good probability of success. Nevertheless, the success factor increases, as the condition used for stability testing, is similar to the actual market conditions and lowest when they are based on shorter version of test under higher temperature acceleration.

To conclude it can be said that stability testing helps one understand the functional and chemical characteristic of a formulated product, to be issued with confidence without any risk of breakdown during processing and use. It can be emphasised that these subjective stability tests, although not absolute, is essential, if one has to produce a product that remains stable long enough for complete consumer satisfaction.