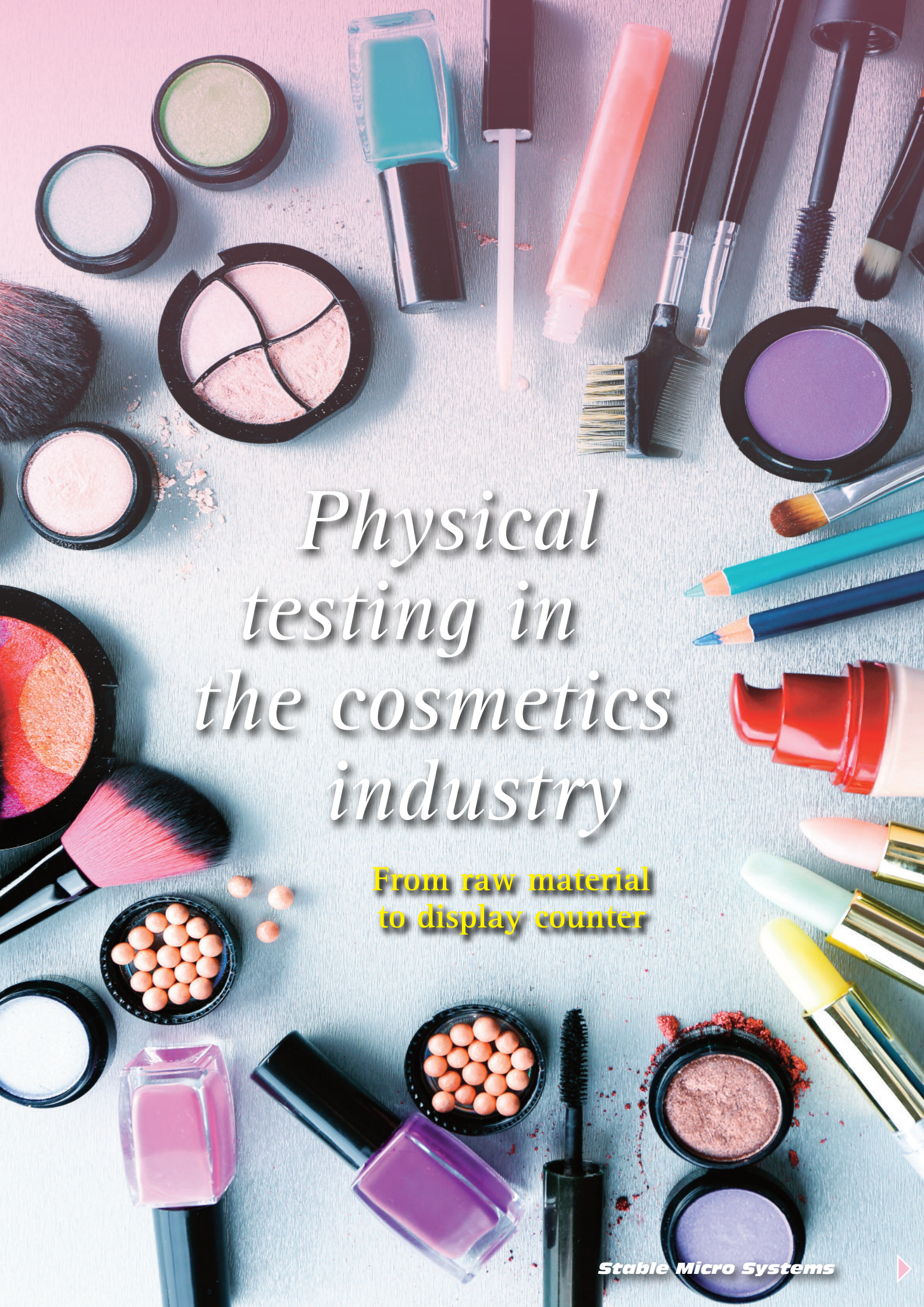


Physical testing in the cosmetics industry

From raw material to display counter

Stable Micro Systems

Physical testing in the cosmetics industry

This article looks at the use of physical testing at every stage of cosmetics manufacture, from the raw ingredients to the fully packaged container on the shop display shelf.

The Stable Micro Systems TA.XT*plus* Texture Analyser

"Machines will give repeatable information that does not vary day to day..."

A customer walks into a pharmacy looking for a new cream to keep their hands soft. They don't have much to go by other than packaging and price, so they choose the same brand they use on their face. They get outside and squeeze some out of the tube with their normal pressure, and their palm fills up with too much thin, watery cream that seems to do nothing to remove the dryness.

A disappointed customer, and the very reason the cosmetics company should have used physical analysis on their cream before they released it for sale.

Physical Analysis: Putting Cosmetics to the Test

Physical analysis, often paired with chemical testing, is the measurement of any physical property that could be important to a cosmetic product. It can be used on raw ingredients or to tell the difference between possible formulations of a new release product, or even in quality control once the product is on the market.

Stable Micro Systems have the answers to a multitude of physical analysis problems and strive to solve new puzzles you may have. The use of a texture analyser removes the uncertainty of human testing; sensory panels are invaluable but only when their data can be correlated with quantitative physical measurements. Machines will give repeatable information that does not vary day to day, and it is crucial this data is available.

Skincare brands might find a simple compression test to be priceless to prevent sponges from being too stiff for gentle cleansing, or the *Spreadability Rig* to look at cream consistency, solving the problem outlined above, or even the use of artificial skin samples on the horizontal friction rig to carry out a thorough analysis of the action of cream being rubbed between a pair of hands.

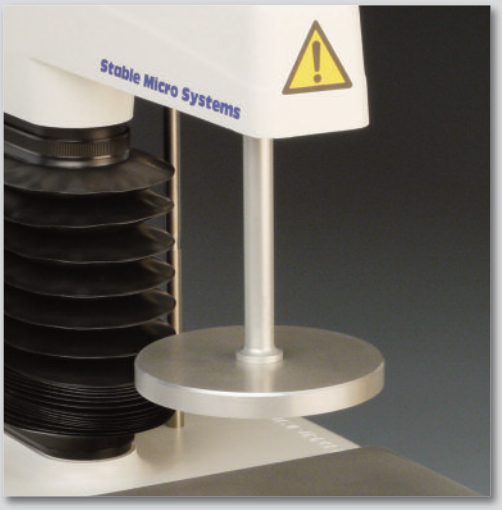
Substantiating cosmetics claims with the aid of texture measurement

An interesting area of this topic is efficacy testing, which tells the manufacturer how well a product has lived up to its intended use. Customers are wary of manufacturers using taglines to tempt them into buying their product – a conditioner that states "hair three

A: TTC Spreadability Rig

Testing consistency of hand cream with the Spreadability Rig

B: Compression Testing



Compression test on sponge

times suppler after first use" will not sell well if customers start using it and find no difference to their tresses. News travels fast these days with thousands of cosmetics review sites and online shops, and products that fail to live up to their claims will be given poor marks. The manufacturers could have performed a simple bend test on hair specimens treated with their conditioner and would have found their mistake before it was too late. The development of methods to measure the effect of cosmetics is driven by increasing pressure on cosmetic companies to provide solid evidence to support product claims.

Claims on cosmetics products need to be substantiated to protect the consumer from false advertising. False claims are not fair to the consumer and lead to scepticism over claims from all cosmetics companies, not just the ones who exaggerated their products' abilities, and so other companies who work hard to ensure efficacy in their products will also be mistrusted. Prior use of particular ingredients or behaviour of formulations is not representative of the characteristics of a new or different formulation, and so reading the literature is not enough.

"... increasing pressure on cosmetic companies to provide solid evidence to support product claims.."

C: Assessment of Firmness by penetration

Cream Firmness testing with a Hemispherical Probe

Instrumental Methods

Instrumental, clinical, sensory analysis and consumer market research are all used to substantiate efficacy claims. The inclusion of all of these methods is very important and will give useful data but instrumental is the least subjective with the least bias on human perception and consumer preference, measuring the nature and magnitude of product effect, and so it should never be skipped. Instrumental methods are precise and sensitive, but even so it can be difficult to measure the whole use of a product, which is why several instrumental methods testing different components of a product are used, and combined with sensory testing or market research.

Additionally, human perception can be correlated with machine data. Efficacy testing using instrumental methods has been of interest for decades, with the first publication of note drawing on a study on the UV absorbance of sunscreen excipients in 1947, in the first *Journal of the Society of Cosmetic Chemists*. Since then efficacy testing has taken off, with subjective studies ahead of the game until the sixties when instrumental methods became more widespread, with the new difficulty of making tests reproducible between laboratories.

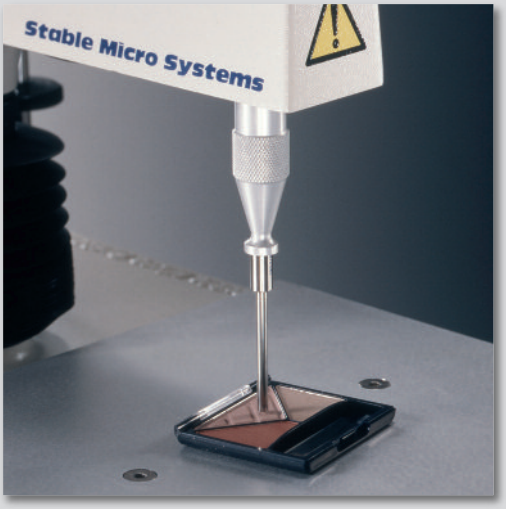
"... the real world conditions must be imitated as closely as possible..."

Improving your Tests

An efficacy test should always be representative of the real-world application. It can be difficult to instrument a complicated process such as rubbing cream into the face, which is why consumer-based testing is useful. The result of any testing should be likely to occur in the real world conditions in which the product is used. For instance, if a hair gel has excellent frizz reduction in the laboratory where temperature and humidity are well-controlled, that does not mean it performs well during real use by a customer (on a windy day, in a hot and humid climate, or in a snowy country with a lot of hot air heating). To make a claim more valid, the real world conditions must be imitated as closely as possible, and a range of conditions should be tested.

The test method should be reliable enough that it can be repeated many times and always give the same result. This is much easier in machine tests than between different sensory panels. The test control also needs to be considered. For example, if a hair conditioner with a new property is being tested, should hair treated with the conditioned sample be compared against hair only washed with shampoo, or hair that has been treated with an ordinary conditioner?

D: Compactability Assessment



Compact breaking strength test using a Cylinder Probe

The drive for cosmetic efficacy testing grows year on year as the pressure increases on cosmetics companies to back up their claims with solid evidence.

Some products face more pressure than others – facial ageing reversal in particular is a sensitive area that can be difficult to evaluate instrumentally. An instrument can only measure one or several mechanical properties, but texture and its associated effects such as moisturisation of skin or softness of hair rely on the consumer's definition of these attributes, as well as their previous experience with other moisturisers, and finally it depends on their particular skin composition and their living conditions.

To overcome these difficulties, the key is to sample a wide range of consumers on a sensory panel, to test all condition extremes the product may face and to keep monitoring the product once it is on the market and the reaction of purchasing consumers.

Hair care manufacturers will find the *Hair Combing Rig* as well as the *Three Point Bend Rig* to be of particular use in efficacy testing. These allow tests to be carried out on real hair samples that may have been treated in any way (hair dye, vigorous brushing, hair serum) to look at the real effects of friction, stiffness or hardness.

Stability Testing

Once these claims have been proven and a perfect product is about to be released for sale, how can you be sure it won't degrade within two weeks of manufacture? The answer is found in stability testing. This is simply the assessment of the lasting power of a cosmetic, during which samples of it are put under different environmental conditions for a set time period, and its change in properties analysed. These conditions vary in light, temperature, pressure and humidity levels and are designed to imitate what the product may be subjected to during its lifetime.

A sample to be stability tested will typically be a new product, an existing product with an altered formula, a new raw material supplier, a new manufacturing procedure, a shift in production to a new manufacturing site or new or altered packaging. A suite of stability tests should precisely target the most important points of testing; physical, chemical and performance characteristics are evaluated at set intervals to see how they have changed.

The qualities of paramount importance are:

- organoleptic (appearance, colour, odour and texture)
- physiochemical (pH, mass, preservatives)
- microbiological (microbial count and preservation efficacy)

"Sachets and tubes have become increasingly popular as plastic qualities improve..."

E: Hair Combing Rig

Conditioner efficacy measured with the Hair Combing Rig

- compatibility between the product and its container

If these changes are small according to the standards set by the manufacturer, the new or altered product has passed stability testing and it can be confidently shipped to consumers. Very few products have to pass international or national standard tests (with the exception of functional products such as sun creams, anti-dandruff products or anti-perspirants).

Accelerated Testing

Once a new cosmetic has begun development it must reach the market as quickly as possible, as each extra day spent on development is another day a turnover cannot be made. Due to this time shortage, it is wise for manufacturers to have a standard set of tests ready for each sample. Additionally, real-time stability testing before a product launch is rarely an option – for instance, a sun cream can remain stable for two to three years. Very few manufacturers have the luxury of testing a product over a three-year period before they release it. Instead, accelerated stability studies take place in which different accelerated storage conditions are used, involving elevated temperature, humidity, light or pressure.

These accelerated tests help to predict the product's stability behaviour and are often used alongside post-release "real-time" tests. After a sample is launched it is stored under ambient conditions and monitored to help refine the accelerated testing method. These post-release tests can also be used to further improve the product.

All tests, real-time and accelerated, include an evaluation of the physical properties of products after storage, transport and use, and the compatibility between the products and the container. These can be performed using texture analysers and rheometers. Additionally, the stability of a product after opening its container can be a useful measure.

Predicting Cosmetic Shelf Life

Shelf life prediction using accelerated methods is not an area that has been widely generalised for scientific publication and so there is a lack of public information on the subject. Part of the problem lies in the wide complexity and variety of both the cosmetic products and their packaging as well as the large number of changes that need to be tested (be they physical, aesthetic, functional, chemical or microbial). Additionally, the cosmetics industry is a guarded place, with both products and test methods under high protection by patents and trademarks.

However, there are some general guidelines that can be followed. For accelerated testing,

"Until recently, only subjective methods were available for assessing 'force to pull'..."

F: Back Extrusion Rig

Consistency testing with the Back Extrusion Rig

the temperature and time components are chosen according to the product type and the company's prior experience. Conditions do not have to be limited to a single time and temperature test – a range of temperatures spanning 30 to 45°C is often used, over a time period of a few months. These conditions depend on the product type and place of storage. The fundamental assumption in stability testing is that an increase in storage temperature will speed up ageing reactions. A useful rule of thumb is that a sample stored at 45˚C for two months is equivalent to one that is stored at room temperature for one year.

The combination of results from accelerated testing and the company's prior knowledge help to give a good prediction of the product's shelf life.

Some Points to Consider

Small scale testing – if only small quantities of a product are available in its early stages of development, small samples from laboratory batches can be tested for an early prediction of stability behaviour. Once development has moved along and the product is being manufactured using full scale equipment, the information on stability behaviour will be more accurate.

Colour and fragrance – various additives are used to achieve the colour and fragrance variations of a product that will be brought to market, and these are likely to affect stability behaviour. Different combinations can react adversely with each other, with the product itself or with the container. If there will be a low number of variations released, it is advisable to test them all. If there are tens of different shades, however, then the evaluation of a representative selection will suffice, as long as testing continues past the product release.

Packaging - the containers used in stability testing should be as similar to the final product container as possible (using the same materials, shape and size). As with colour and shade, if there will be a range of package sizes or types, then they should all be tested if possible. A control container made of glass or another inert material can be used, and if testing is to be very thorough then different orientations can also be tested, with the container upside down or on its side.

Extreme Conditions

Some extreme testing will be performed on any given product, and the tests carried out depend on its likely weaknesses as well as the conditions it will face during shipping, storage, display and use.

Manufacturers decide whether to perform such specialised testing based on the weaknesses of any particular cosmetic product and its anticipated shipping, storage, display and use. For example, an emulsion will not typically survive being frozen and re-thawed, a suspension may become cloudy, packaging may distort and its label come loose, or corrosion may occur on the inside of an aluminium container that has been coated in a lacquer.

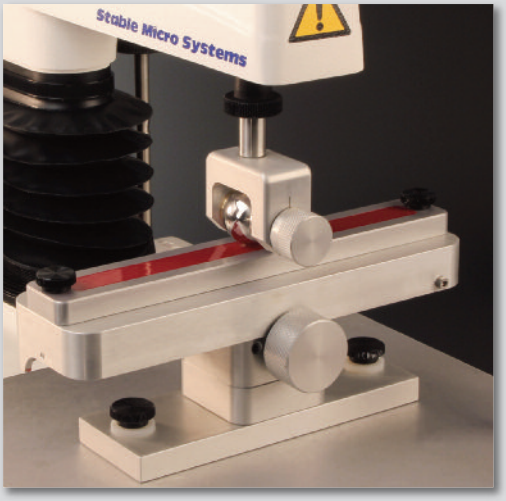
Finally, mechanical shock testing is performed to check for vulnerabilities during shipping, and vibration testing used to check for the likelihood of separation of components in a granular product, whereas light stability testing is used to mimic the light intensity that a product will be exposed to if stored in a clear container.

"Some extreme testing will be performed on any given product..."

G: Lipstick Bend Rig

Measuring lipstick strength with the Lipstick Bend Rig

H: Nail Polish Adhesion Rig



Nail polish drying characteristics can easily be determined

"All the tests are archived with date and time information..."

Where does a Texture Analyser fit into all of this?

Many of the tests mentioned involve physical properties. The team developing a product will have spent a long time assessing its ideal assets. These might be the consistency of a cream, the extrusion force of a toothpaste from its tube, the cohesion behaviour of a powder, the cake strength of a compact, the shear strength of a lipstick, the drying time of a nail polish, the adhesive strength of depilatory wax... the list goes on. All of these products can be reliably tested using one of the Stable Micro Systems' Texture Analysers to give reliable, repeatable results.

If time is an important parameter, there are methods to help increase sample throughput such as the *ALIS* – an automated indexing system that enables multiple tests to be performed automatically. Additionally, the *Exponent* software is designed to make the testing process fast and simple, with flawless comparison of samples within a batch or between batches themselves, and automated macros to analyse all the experimental data at the press of a button.

All the tests are archived with date and time information to help compare real-time tests after the release of the product with the accelerated stability tests that were performed pre-release, with the ability to constantly update the accelerated testing procedure and help it to be as representative of real storage as possible.

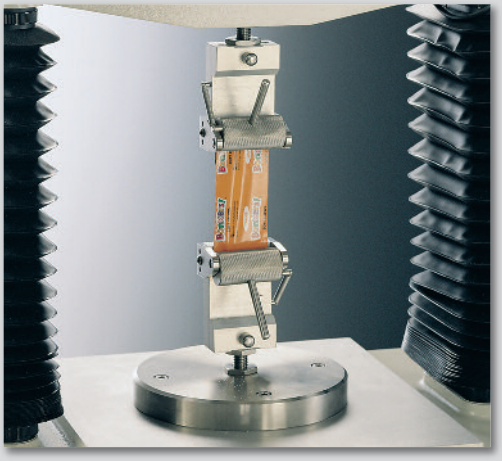
Cosmetics Packaging

The development of cosmetics for release into a competitive market is a high cost endeavour, so it would be inefficient for these high stakes products to be shipped in low quality packaging, or for the container to degrade during its shelf life. Packaging is the first thing the customer sees in the shop so from this point of view the graphics and physical design are important to make it stand out amongst other similar products; the appearance of packages can directly affect marketing. However, the main purpose of packaging is to ensure the product arrives in a customer's hands in perfect condition and to prevent any losses caused by shipping, handling or storage.

Protecting the Contents of your Package

This is the first question to be answered during this series of tests. The majority of obstacles faced by a product can be imitated using physical testing with a texture analyser. Any given company will have a standard set of tests for new products or for spot checking the quality of products already on the market. Physical testing measures the strength of each

J: Tensile test measurements



Elongation and tensile strength measurement

Tensile test assessing seal strength

component and joint, assessing whether or not the container will continue protecting its contents after shipping or a long storage time.

Shipping can be imitated using force cycle tests to represent the repeated loading the product will face during its journey, whereas storage testing may involve a timed compression with a large platen to mimic the action of stacking many products on a shelf.

Bottle tops are widely-used and the strength of the cap itself is an important measure, as this influences the transportation restrictions. Points for consideration include the worst case scenario a package may face, and what the cumulative effects will be of days of shipping or months of storage amongst other packages, or even in a cosmetics drawer in the consumer's home.

The absolute material strength of a product is an important part of this testing process. For instance, adhesives are widely used in cosmetics packaging, particularly in a seam at the edge of a sachet. These are assessed by performing a T-peel test on a small section of the sachet joint. The customers' safety must also be considered, and this is relevant when testing a property such as the puncture strength of an aerosol deodorant can.

Packaging tests will cover either the whole assembly (a whole lipstick tube), one part of the packaging (the lid of a hairspray) or a sample of the material itself (a dogbone specimen of a plastic used for a talc bottle).

Post-Test Aesthetics and Functionality

A customer might have found a lipstick they adore and have carried it around in their coat pocket for months, but the lipstick is nearly out and the plastic has started cracking at the fragile lid opening, causing the brand to appear to have skimped on packaging costs. In this case the fragile lid should have raised a red flag during testing and been reinforced with an alternative material.

Many cosmetics are integrated with their packaging, so the packaging also acts as a

"Shipping can be imitated using force cycle tests..."

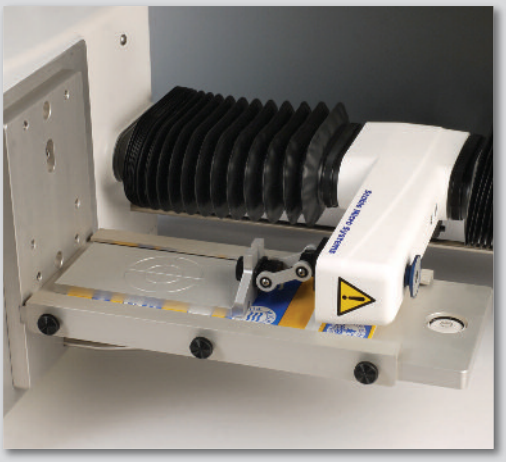
K: Actuation Force measurement

Measuring force to operate cream dispenser...

... and aerosol

L: Different methods of assessing packaging properties

Ball probe assessing adhesion



Horizontal Friction System assessing film surface properties

"Adhesives are widely used in cosmetic packaging."

dispenser or applicator. For example, the applicator brush in a mascara tube is both a component of the lid and a crucial part of the application process, and the nozzle on a hairspray can is responsible for dispensing the product. For these products, their functionality may be tested after the ageing or shipping tests have taken place.

Towards the end of its useful life, what experience will the consumer have of the packaging? Testing can help to predict whether it will still be functional or if it will start falling apart, and this quality assurance goes a long way towards helping the product designers tweak the design.

Ease of Use

The forces involved in using a product must be within a suitable range for any customer ability in the intended market. For any given action (such as pulling, snapping or pressing) an acceptable range of forces can be determined by referring to tabulated results of the forces a child, healthy adult, elderly or infirm customer is capable of. The product container can then be tested in a quantitative way on a texture analyser, and the forces verified. For example, if the lid of a tub requires a high force to remove, it may be too difficult for some customers to use that product without assistance.

Adhesives are widely used in cosmetic packaging. A pressure sensitive adhesive that may be found on a film lid to seal a face cream could either be tested alone using a ball probe compression-tension cycle, or a peel test could measure the real force required to remove the lid at a 90 or 180° angle.

Another commonly-used packaging method is the polymer-based sachet. In this case the ease of opening is determined by the tearing force, strength of seal (from the hot press process) and friction of the material surface. When a consumer is opening the package, if the friction does not match with the tearing strength, they cannot get enough grip on the package for a high tear force and it will slip in their hands. In this case all of these properties should be tested individually by machine and verified using a sensory panel.

Container-Contents Compatibility

This question is answered using packaging compatibility tests. These tests are carried out on everything that is destined to go into production, simultaneously with stability tests. If a formula is not appropriate for the intended container type, if the combination of container and closure is inadequate or if the packaging is poorly made, the compatibility test will catch it out. Occasionally a test as simple as having the formula in containers

M: Sachet/Tube Extrusion Rig

Measurement of tube or sachet content expulsion force

made of different materials will give very different results of the contents' stability.

Weathering and ageing tests are often used to speed up the degradation process, especially when the product may be stored in extreme temperature environments. Heated cabinets with temperature control are the standard piece of equipment used in this case. A container is filled with its formula and heated to a specific temperature for a given time period. Once this ageing is over, the container is taken back to the texture analyser and its physical properties are tested once again, along with an assessment of decolouration, weight loss of the contents and stress cracking.

Summary

The minor improvements that are so important to cosmetic product performance require carefully conducted experiments in order to provide definite information. This article has covered the use of physical testing for high quality cosmetics, methods to help with substantiating claims, stability testing and shelf life prediction, and finally tests to ensure packaging is a high standard.

Exponent is extremely valuable to this type of testing as standard projects can be saved to enable each set of test settings to load at the press of a button, along with automatic data analysis and report generation. A wide variety of international standards can also be built into the software. With data appearing graphically on-screen in real time, the suite of texture analysers along with their expert software is ideal for use in quality control.

To find out how we can help you improve customer satisfaction and loss reduction with a custom set of cosmetics products and packaging tests, talk to Stable Micro Systems today.

"Weathering and ageing tests are often used to speed up the degradation process..."

Parts of this article were originally published in Laboratory News

Stable Micro Systems

Vienna Court, Lammas Road, Godalming, Surrey GU7 1YL, UK

Tel: *44 1483 427345

Fax: *44 1483 427600

Email: sales@stablemicrosystems.com
app.support@stablemicrosystems.com
tech.support@stablemicrosystems.com

Web: www.stablemicrosystems.com