

TEST PRINCIPLE

Evaluation of the consistencies of two types of moisturising creams by back extrusion.

BACKGROUND

Moisturising creams serve to keep the skin feeling smooth, soft, and looking radiant and healthy by retaining moisture or water in the outer most skin layer. Different moisturising creams will be defined by their viscosity or thickness. Formulating moisturising creams therefore largely depends upon the required end product consistency, influencing the choice of material to use.



One desirable factor in product development is having a product that easily squeezes out of a tube and breaks off cleanly after squeezing. With the use of forward and backward extrusion tests these consistencies can be assessed. The **forward extrusion** simulates the force required by the consumer to extrude the sample and the **backward extrusion** an indication of product structure failure and flow properties. The back extrusion accessory is useful for testing viscous products. Samples can also be tested in their containers directly from the production line.

METHOD

EQUIPMENT: CT3 with 4.5kg load cell
Fixture Base Table (TA-BT-KIT)
Dual Extrusion Cell (TA-DEC)

SETTINGS: Test Type: Compression
Pre-Test Speed: 1.0 mm/s
Test Speed: 2.0 mm/s
Post-Test Speed: 2.0 mm/s
Target Type: Distance
Target Value: 25 mm
Trigger Force: 10 g

Note: It is recommended that the pre-test speed be the same as or less than the test speed for accurate trigger detection; for example, 1 mm/s test speed will require ≤ 1 mm/s pre-test speed.

The target distance chosen should be such that the probe does not deform the sample to more than 75% of sample depth, otherwise the base effect may affect results.

PROCEDURE

1. Attach the extrusion disc to the load cell.
2. Fit the back extrusion cell onto the base table; tighten the screws, but still allow some degree of mobility.
3. Align the extrusion container centrally below the extrusion disc (plunger), then tighten the screws of the base table.
4. Remove the sample kept at a specific temperature from storage.
5. Fill the extrusion container with the sample to about 75% full, minimizing air bubbles.
6. Position the probe to a specified starting distance E.g., 10 mm above the top of the container or sample surface.

This will ensure the probe returns to the same position after each test, enabling comparisons of adhesive force which is an indication of the cohesiveness of the sample.

RESULTS

A comparison of consistencies of two types of moisturizing creams by back extrusion.

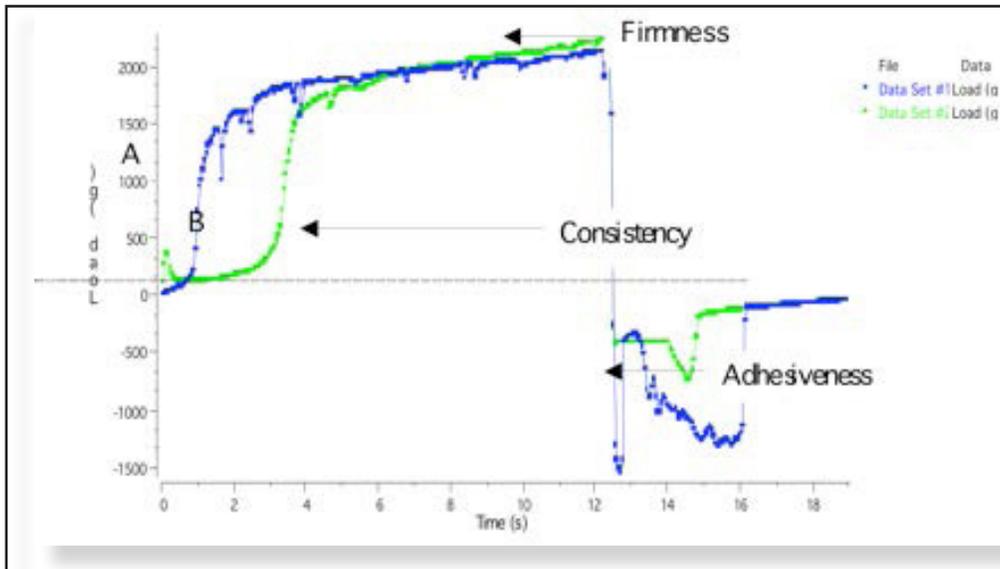


Figure 1

The graph in Figure 1 shows the consistencies of two types of moisturising creams stored and tested at 21°C in a 40 mm diameter back extrusion container.

Data Set #1: Sample A (Premium Moisturising Cream)

Data Set #2: Sample B (Budget Moisturising Cream)

OBSERVATIONS

When a 10 g surface trigger is attained, the plunger proceeds to push onto the sample to a depth of 25 mm. During this time, the sample is deformed and compressed to pack more tightly into the diminishing space available (under the descending plunger) and the force is seen to increase steadily. When the sample becomes more compact with limited air pockets, the force increases sharply and the extrusion commences. When the force increases to a maximum point, a plateau is observed indicating the force required to continue extrusion. This is the final load.

The maximum force on the graph measures firmness; the higher the value, the firmer the sample. The area under the positive part of the graph indicates sample consistency (work done to hardness¹); the higher the value, the thicker and the higher the consistency of the sample.

As the probe returns to its starting position, the initial lifting of the weight of the sample on the upper surface of the disc produces the negative part of the graph resulting from back extrusion. This gives an indication of the adhesiveness/cohesiveness and resistance of the sample to separate (flow off) from the disc.

The maximum negative force on the graph indicates sample adhesive force; the more negative the value, the more “sticky” the sample. The area under the negative part of the graph is known as the adhesiveness (the energy required to break probe sample contact) and can give an indication of the cohesive forces of the molecules within the sample. The higher the value, the more energy required to break the probe sample contact as the probe withdraws from the sample.

From Figure 1, sample A and B have similar maximum peak (firmness) values but Sample A has a higher consistency, seen by the larger area under the positive curve than sample B. Sample B shows a variation in consistency as seen by the initial steady increase in force over the first few seconds followed by a more rapid increase in force indicating the possibility of sedimentation in the sample and formation of two layers. Sample A has a higher adhesive component than sample B with a higher negative peak value.

The test can be used as a quality control tool to ensure consistency in production and also in R&D and Product Development in monitoring effect of formulation or process control changes. Tests obtained from 3 moisturizing creams of each formulation give the following mean hardness and work done values below:

Table I

Sample	Hardness (g)	Hardness Work Done (mJ)
A	2053.0 ± 23.7	398.8 ± 41.54
B	2262.8 ± 13.8	377.65 ± 16.32

TEST PRINCIPLE

Evaluation of the consistencies of two types of moisturising creams by penetration using a hemispherical probe.

BACKGROUND

Moisturising creams serve to keep the skin feeling smooth, soft, and looking radiant and healthy by retaining moisture or water in the outer most skin layer. Different moisturising creams will be defined by their consistency. Formulating moisturising creams therefore largely depends upon the required end product consistency, influencing the choice of material to use.



The penetration test, using a hemispherical probe, is an imitative test simulating the ease by which a human finger will deform the sample during application of the cream. This test allows the consistencies of creams to be assessed. Samples can also be tested in their containers directly from the production line.

METHOD

EQUIPMENT: CT3 with 4.5kg load cell
Fixture Base Table (TA-BT-KIT)
Hemispherical probe (TA-43)

SETTINGS: Test Type: Compression
 Pre-Test Speed: 1.0 mm/s
 Test Speed: 2.0 mm/s
 Post-Test Speed: 2.0 mm/s
 Target Type: Distance
 Target Value: 25 mm
 Trigger Force: 10 g

Note: It is recommended that the pre-test speed be the same as or less than the test speed for accurate trigger detection; for example, 1 mm/s test speed will require ≤ 1 mm/s pre-test speed.

The target distance chosen should be such that the probe does not deform the sample to more than 75% of sample depth, otherwise the base effect may affect results.

PROCEDURE

1. Attach the hemispherical probe to the load cell.
2. Align the sample centrally under the probe.
3. Start the test.
4. When the probe is pulling out of the sample and returning to the start position, firmly hold the sample down to prevent it from lifting.
5. Ensure thorough wiping of the probe using a clean, dry cloth to remove all traces of adhering soap before proceeding onto the next test.

Note: When optimising test settings, the hardest sample would be better tested first in order to anticipate the maximum testing range required. This will ensure that the force capacity covers the range for other future samples.

The penetration test can also indicate whether there are trapped air bubbles or a grainy texture, seen as fluctuations in force, as the probe comes into contact with air bubbles or hard particles.

RESULTS

The following is a typical Texture Analysis penetration plot of two different types of moisturizing cream.

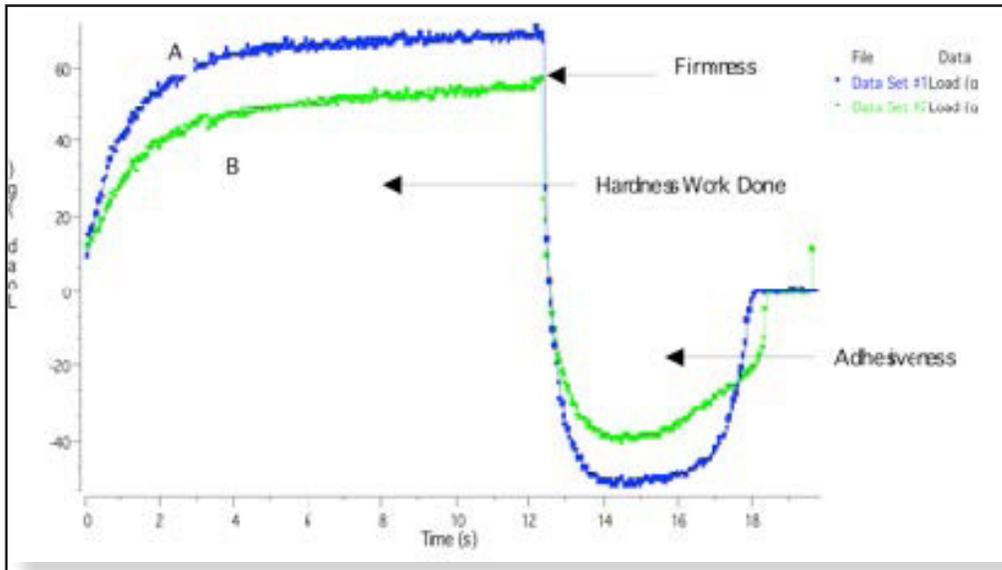


Figure I

Figure I compares the hardness of two different moisturizing cream formulations tested at 21°C.

Data Set #1: Sample A (Premium Moisturising Cream)

Data Set #2: Sample B (Budget Moisturising Cream)

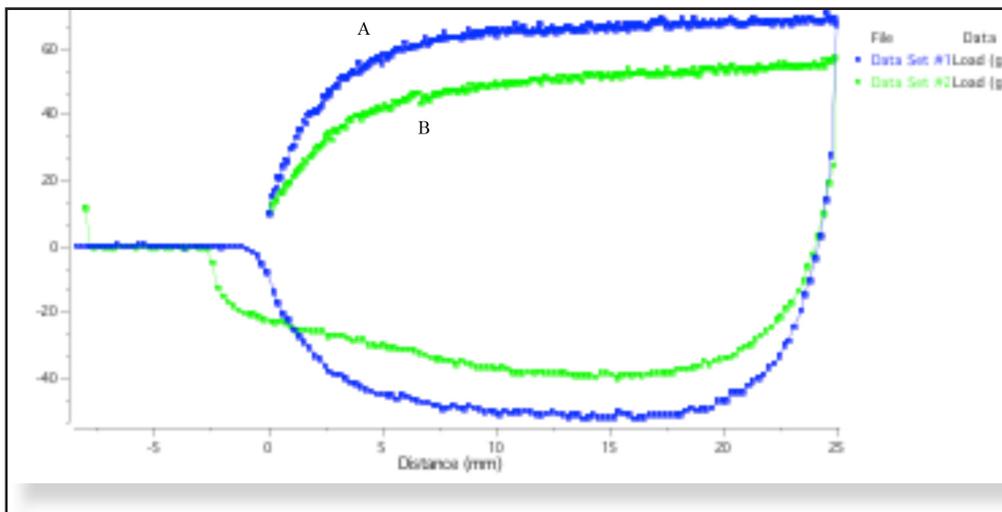


Figure II

Figure II displays a graph of load versus distance comparing the hardness of two different moisturizing cream formulations.

Data Set #1: Sample A (Premium Moisturising Cream)

Data Set #2: Sample B (Budget Moisturising Cream)

OBSERVATIONS

When a trigger force of 10 g is achieved, the probe begins to penetrate the sample to a defined distance (25 mm in this case). The maximum positive force then becomes force value, the firmer the sample. From Figure I, sample A is firmer than sample B and is also more adhesive. The energy required to deform the samples can be quantified by selecting Work Done Hardness 1 in the software. This is the area under the positive curve. As can be seen in the table below, Sample A requires more energy than sample B to deform the sample to a defined seformation distance.

Table I

Sample	Hardness (g)	Hardness Work Done (mJ)
A	70.5	14.91
B	57.1	11.38

The adhesiveness of the samples can also be measured whereby the area under the negative curve is a measure of adhesiveness. From Figure I, Sample A is more adhesive than Sample B.

TEST PRINCIPLE

Evaluate the spreadability of two types of moisturising creams using male and female conical probes.

BACKGROUND

Moisturising creams serve to keep the skin feeling smooth, soft, looking radiant and healthy by retaining moisture or water in the outer most skin layer. Different moisturising creams will be defined by their viscosity or thickness. Formulating moisturising creams therefore largely depends upon the required end product consistency influencing the choice of material to use. For instance, a lipid rich cream will have decreased spreadability values with an increase in viscosity and surface tension (a measure of cohesiveness), making the cream greasy, tacky and difficult to spread. The lower the viscosity of a cream, the lower the surface tension and the more the cream is easily spread and absorbed into the skin.



METHOD

EQUIPMENT: CT3 with 4.5kg load cell
Fixture Base Table (TA-BT-KIT)
Spreadability Fixture (TA-SF)
TexturePro CT Software

SETTINGS: Test Type: Compression
 Pre-Test Speed: 1.0 mm/s
 Test Speed: 2.0 mm/s
 Post-Test Speed: 2.0 mm/s
 Target Type: Distance
 Target Value: 25 mm
 Trigger Force: 10 g

The spreadability accessory quantifies the spreadability of samples. It consists of a set of matched male and female Perspex cones.

SAMPLE PREPARATION

1. Fill the four lower cones (female cones) with the sample using a spatula.
2. Level the surface using a flat knife.

PROCEDURE

1. Position the base table on the base of the machine.
2. Loosely tighten the screws of the base table to enable a degree of mobility.
3. Place the base holder on to the base table and lock into position with the screws.
4. Insert the female cone into the female cone sample holder.

5. Attach the male cone to the Load cell.
6. Precisely align the male and female cones by lowering the male cone and repositioning the female cone such that the male cone fits into the female cone.
7. The screws of the base table can now be tightened.
8. Prior to testing, the male probe must always be placed a few millimeters above the sample.
9. Commence the test.

Note: Precise alignment of the probes is essential in order to avoid instrument overload.

Fluctuations on a smooth curve are a result of the compression of air pockets. It is therefore important to minimize air pockets when filling the female cone.

The base holder should not be removed from the base table between tests as this will necessitate realignment of the male and female probes. The female cone can be removed after the test by loosening the screws of the base holder.

For comparison purposes, the test temperature must always be reported in the results.

RESULTS

The following graph shows the spreadability of two different types of moisturizing cream.

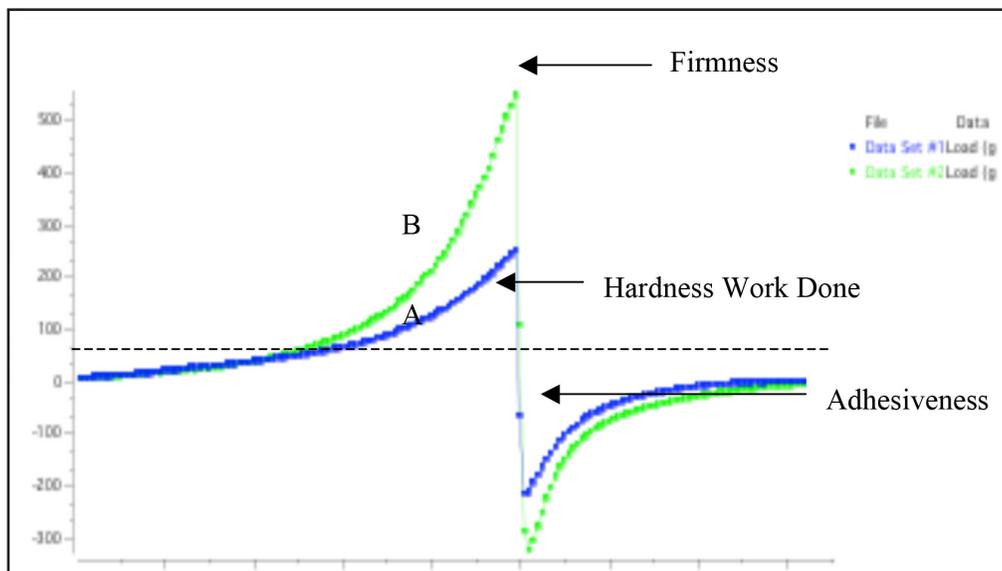


Figure I

Figure I shows the spreadability of two types of moisturizing cream at 21°C using 45° male and female cone probes. For each test, the male cone was positioned 3 mm above the sample surface.

Data Set #1: Sample A (Premium Moisturising Cream)

Data Set #2: Sample B (Budget Moisturising Cream)

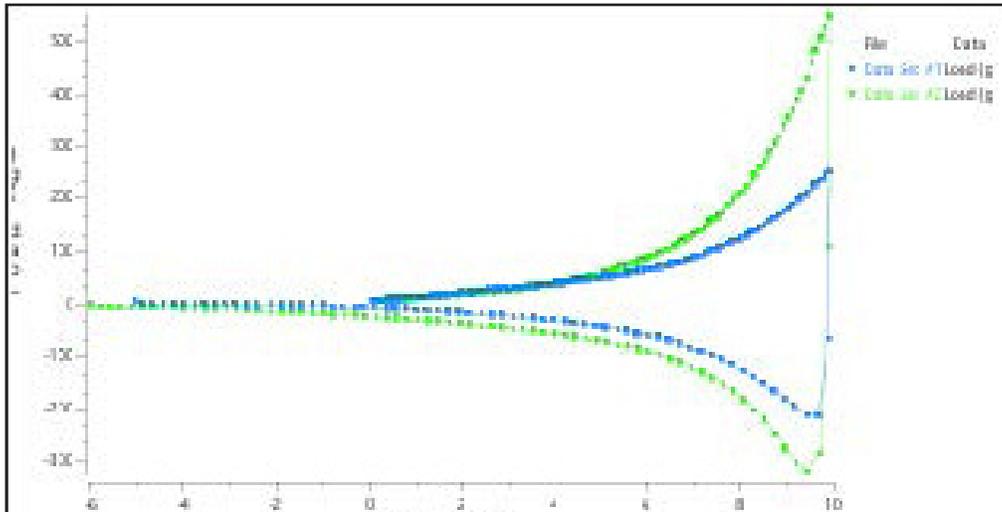


Figure II

Figure II is a load versus distance graph for the spreadability of two types moisturizing creams.

Data Set #1: Sample A (Premium Moisturising Cream)

Data Set #2: Sample B (Budget Moisturising Cream)

OBSERVATIONS

When a trigger force of 10 g has been achieved, the probe proceeds to penetrate the sample at a test speed of 2 mm/s to a depth of 25 mm. During this time, the force to penetrate the sample increases. When the specified penetration distance has been reached, the probe withdraws from the sample at the post-test speed of 2 mm/s. The maximum force value on the graph is a measure of the firmness of the sample at the specified depth. The area under the positive curve is a measure of the energy required to deform the sample to the defined distance (Hardness Work Done). Research has shown that the firmness and energy required to deform a sample to a defined depth grades samples in order of spreadability. A higher peak load (firmness) and hardness work done value indicate a less spreadable sample. Conversely, a lower peak load (firmness) value coupled with a lower hardness work done value indicates a more spreadable sample. From Figure 1, Sample B is significantly much firmer than Sample A and has a higher hardness work done (area under the positive curve). This indicates that A is more spreadable than sample B.

As the probe returns to its starting position, the initial lifting of the weight of the sample on the upper surface of the disc produces the negative part of the graph. This gives an indication of the cohesiveness and resistance of the sample to separate (flow off) from the disc. The maximum negative force on the graph indicates sample adhesive force; the more negative the value the more “sticky” the sample. The area under the negative part of the graph is known as the adhesiveness (the energy required to break probe sample contact) and can give an indication of the cohesive forces of the molecules within the sample. Sample B is more adhesive or “sticky” and therefore more cohesive than Sample A. Tests obtained from 4 samples of each moisturising cream formulation give the following mean hardness and work done values below:

Sample	Hardness (g)	Hardness Work Done (mJ)
A	250.5 ± 57.5	6.94 ± 0.94
B	542.5 ± 48.6	8.58 ± 2.29