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Assessment of styling performance in hair gels and hair sprays by means of a new two-point stiffness test

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Synopsis

A new two-point bending stiffness method on flat hair strands was developed and validated after application of hair styling gels and hair styling sprays. A special mold was used to align single hair fibers after applying the formulations to the hair.

The styling gels used contain different commercially available thickeners and styling polymers, e.g., carbomer, acrylates/beheneth-25 methacrylate copolymer, Polyquaternium-86, PVP, VP/VA copolymers, and VP/methacrylamide/vinylimidazole copolymer. Evaluation of hair sprays was performed after spray application on flat hair strands. Commercially available hair styling resins were used, e.g. acrylates/t-butylacrylamide copolymer, octylacrylamide/acrylates/butylaminoethyl methacrylate copolymer, and VP/VA copolymer (30:70).

The new stiffness test method provided the best correlation with practically relevant sensory assessments on hair strands and a panel test in which styling gels were evaluated. However, we did not observe a correlation between the new stiffness method on flat hair strands and practical assessments in hair spray application. We postulate that different polymer/hair composites are responsible for these discrepancies. Hairs on model heads for half-side testing are spot-welded after spray application, while hairs are seam-welded in the stiffness test after alignment of single hair fibers. This alignment is necessary to achieve reproducible results.

INTRODUCTION

Hair gels and sprays have a high rank among styling products. Gels are favored for short hair styles and are particularly used by men, while sprays are used by women with long hair. However, styling products differ in composition, especially in the polymers used. In order to tailor properties such as fixative power, wash-out, and elasticity, polymers are developed by a smart choice of monomer composition and process technology. A temporary hold is achieved by means of a composition of hair fiber and polymer at the hair surface. For characterization of the polymer film, bending stiffness is the most important parameter. There are several methods to evaluate stiffness: sensorial tests on hair strains, half-side tests on model heads, salon tests, and panel tests. The objective test, first

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evaluated by Frosch and Vogel (1), is the three-point bending stiffness test. Alternatively, the omega-loop method (2) can be used. Depending on the application and type of formulation, the correlation of the objective stiffness test with the subjective consumer relevant tests is limited.

OBJECTIVE OF THE STUDY

The objective test, e.g., the three-point stiffness test on round-shaped hair strains (1), has limited correlation with the afore-mentioned consumer-relevant tests. This is especially a problem for the assessment of the styling performance of hair gels. There is a need for improved objective tests and a closer correlation with practically relevant tests.

Tests on hair gels with combinations of different thickeners and styling polymers will be investigated by means of a new two-point bending stiffness test on flat hair strains. The results will be correlated with the subjective tests on these hair strains and by means of a panel test for selected formulations. In conventional stiffness tests for hair sprays, multiple application procedures are often applied, e.g., by means of a syringe or by dipping the hair strains into the hair spray solution without a propellant (1). Any influence of these factors, such as propellants, solvents, water, and valve cannot be considered in the conventional bending test after dipping. In our new approach we use a two-point bending stiffness test in which flat hair strains are used after practically relevant spray application.

EXPERIMENTAL

MATERIALS

Round- shaped (2.9–3.3 g, 26 cm) and flat (2.9–3.3 g, 20cm) hair strains from Kerling (Caucasian, virgin brown hair, Art- Nr. 826550) were used. Model heads (Caucasian dark brown hair) from Wernesgruen, Germany, were used for half-side tests. All hair strains and model heads were washed twice with a solution of 27–28% sodium laureth sulfate.

FORMULATIONS

Gel and spray formulations tested for bending stiffness are listed in Tables I and II.

BENDING STIFFNESS METHOD: THREE-POINT ROUND

Hair strains (2.9–3.3 g, 26-cm round shape) were dipped repeatedly into the diluted gel (50 g of gel and 140 g of water) or in the spray solution without propellant. Excess gel or spray was wiped off and the hair strains were lightly compressed on filter paper. Afterwards the hair strains were formed with fingers until they were round-shaped. The strains were dried overnight at 20°C and 65% relative humidity. The measurement of the bending stiffness was performed on a Karg tensile tester (TT 27025E6). The spacing between

Table I
Gel Formulations Tested for Bending Stiffness

No.	Formula
1	1.00 g Acrylates/beheneth-25 methacrylate copolymer (Rohm & Haas Company); 12.50 g VP/methacrylamide/vinyl imidazole copolymer (BASF SE) (2.50 g polymer content); 100.00 g water
2	1.00 g Acrylates/beheneth-25 methacrylate copolymer (Rohm & Haas Company); 12.50 g PVP 20% solution (BASF SE) (2.50 g polymer content); 100.00 g water
3	1.00 g Polyquaternium-86 (BASF SE); 12.5 g PVP 20% solution (2.50 g polymer content); 100.00 g water.
4	0.50 g Carbomer (Lubrizol Corp.); 15.0 g PVP 20% solution (BASF SE) (3.00 g polymer content); 100.00 g water.
5	0.50 g Carbomer (Lubrizol Corp.); 2.50 g Polyquaternium-11 (BASF SE) (0.50 g polymer content); 12.5 g PVP 20% solution (BASF SE) (2.50% polymer content); 100.00 g water.
6	Market formulations with carbomer (Lubrizol Corp.); VP/VA copolymer and PVP (BASF SE)
7	Market formulations with carbomer (Lubrizol Corp.) and AMP-acrylates/allyl methacrylate copolymer (Lubrizol Corp.)

All formulas contained 0.90 g phenoxyethanol/ethylhexylglycine as a preservative.

Table II
Spray Formulations Tested for Bending Stiffness

No.	Formula
1	3.00 g Octylacrylamide/acrylates/butylaminoethyl methacrylate copolymer (Akzo Nobel Surface Chemistry); 0.53% AMP; 40.00 g DME; 100.00 g ethanol 96% (neutralization grade of polymer: 90%)
2	3.00 Acrylates/t-butylacrylamide copolymer (BASF SE); 0.35 g AMP; 40.00 g DME; 100.00 g ethanol 96% (neutralization grade of polymer: 100%)
3	3.00 g VP/VA/copolymer (BASF SE); 40.00 g DME; 100.00 g ethanol 96%
4	5.00 g Octylacrylamide/acrylates/butylaminoethyl methacrylate copolymer (Akzo Nobel Surface Chemistry); 0.53 g AMP; 40.00 g DME; 100.00 g ethanol 96% (neutralization grade of polymer: 90%)
5	5.00 g Acrylates/t-butylacrylamide copolymer; 0.35 g AMP; 40.00 g DME; 100.00 g ethanol 96% (neutralization grade of polymer: 100%)
6	5.00 g VP/VA copolymer (BASF SE); 40.00 g DME; 100.00 g ethanol 96%
7	3.00 g Octylacrylamide/acrylates/butylaminoethyl methacrylate copolymer (Akzo Nobel Surface Chemistry); 0.53 g AMP; 40.00 g P/B; 100.00 g ethanol 96% (neutralization grade of polymer: 90%)
8	3.00 g Acrylates/t-butylacrylamide copolymer (BASF SE); 0.35 g AMP; 40.00 g P/B; 100.00 g ethanol 96% (neutralization grade of polymer: 100%)

DME: dimethylether; P/B: propane/butane (25%/75%); AMP: 2-amino-2-methyl-1-propanol.

points 1 and 2 (cantilever) as well as between points 2 (cantilever) and 3 was 4.40 cm. The cantilever was moved vertically with a constant velocity while the force was recorded (constant speed of 500 mm/min, traverse 40 mm). The maximum force that is necessary to break the polymer film was recorded and describes the bending stiffness forces. Each sample was tested with at least seven different hair strains to determine the average and standard deviation (1,3,4).

BENDING STIFFNESS METHOD: TWO-POINT FLAT

Gel application. The gel was diluted with water (50 g of gel and 140 g of water). The flat strains were dipped into the diluted gel solution and compressed on filter paper. Afterwards the hair strains were pulled through a preparation device (see Figure 1). The preparation device is a tool that aligns the single hair fibers parallel to each other. The reproducibility in preparing the hair tresses is increased, and therefore the error limit of the stiffness test is significantly reduced. The alignment of the hairs is achieved by pulling the flat hair tresses through the mold of the preparation device using a comb at one side.

Spray application. The hair strains were mounted vertically and fixed with braces. Ten grams of hair spray was applied at a distance of approximately 20 cm between the valve and the hair. The hair strains were then pulled through a preparation device (see Figure 1).

Afterwards the strains, treated with gel and spray, were dried overnight at 20°C/65% relative humidity. Each formulation was tested on ten (gel) and seven (spray) hair strains to determine the average, standard deviation, and confidence interval.

The bending stiffness was measured on hair strains that were fixed between two glass plates. The hair strains between the glass plates were fixed in a horizontal position. The bending force of the hair/polymer composite was measured in a tensile tester, Texture AnalyzerTA.XTPlus (see Figure 2). The spacing between the two points (edge of the glass plates that fix the hair tress and the cantilever of the Texture Analyzer) was 2.00 cm. The cantilever was moved vertically with a constant velocity while the force was recorded (starting speed of 500 mm/min, traverse 55 mm, and force at starting point of 20 g).

For gel application, the strains were broken at four different positions (A to D). For spray application, the strains are broken only at position D (see Figure 3). The maximum force that is necessary to break the polymer film was recorded and describes the bending stiffness forces. Figure 4 depicts a typical force displacement curve.

HAND GRADING

Round- and flat-shaped hair strains were treated in the same way as used for the bending stiffness tests. After drying overnight at 20°C and 65% relative humidity, the strains were broken by hand and their stiffnesses were rated subjectively (1 = highest, 4 = lowest). A rating of 1- is closer to 1, but rated lower. A rating of 1- is significantly different from 2+, i.e., closer to 2.

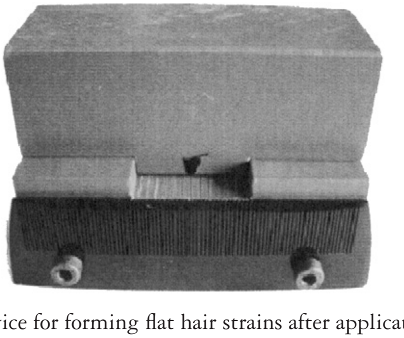

Figure 1. Preparation device for forming flat hair strains after application of styling products.

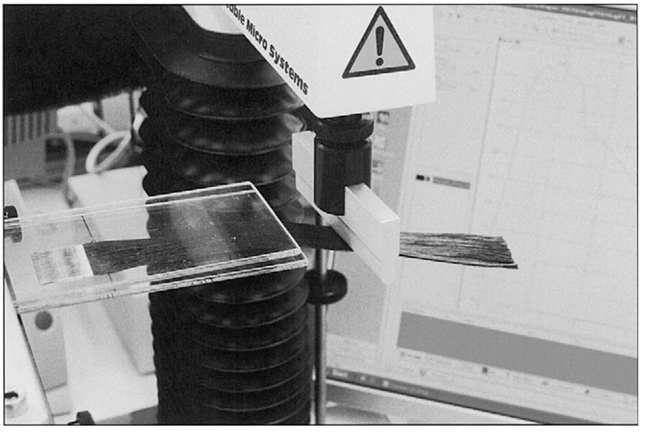

Figure 2. Measuring device: Texture Analyzer TA.XTPlus.

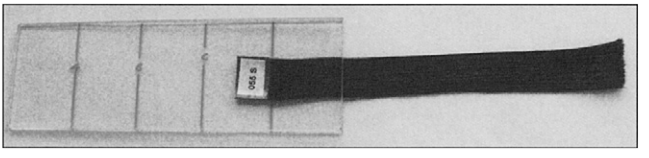

Figure 3. Prepared flat hair strain between two glass plates at a defined position.

PANEL TEST

The panel test was performed in 2007 in Ludwigshafen, Germany as a double-blind test. Twenty-eight responses were counted. The questionnaire asked for the direct comparision of the two gels with three ranks: better, similar, or worse (4).

HALF-SIDE TEST

The preparation of the hair and the evaluation were performed in a climate room at 20°C/65% relative humidity. *Ca*. 3.8 g of spray was applied on each side of the model head. In direct comparision, two different products were evaluated by three trained lab technicians. Focus was on estimation of the stiffness of the hair style after drying of the hair spray overnight. The hair setting was rated by numbers 4 (no setting), 3 (poor setting), 2 (medium setting), and 1 (high setting).

RESULTS AND DISCUSSION

The results of the standard three-point stiffness test on round-shaped hair strands after application of styling gel formulations (see Table I) are depicted in Figure 5. Discrepances between hand grading and stiffness are obvious. Formulations 1 and 2 are characterized by a low stiffness (< 150 cN) but with a quite good hand grading of formulation 1. The lowest correlation between the objective bending test and hand grading occurs with

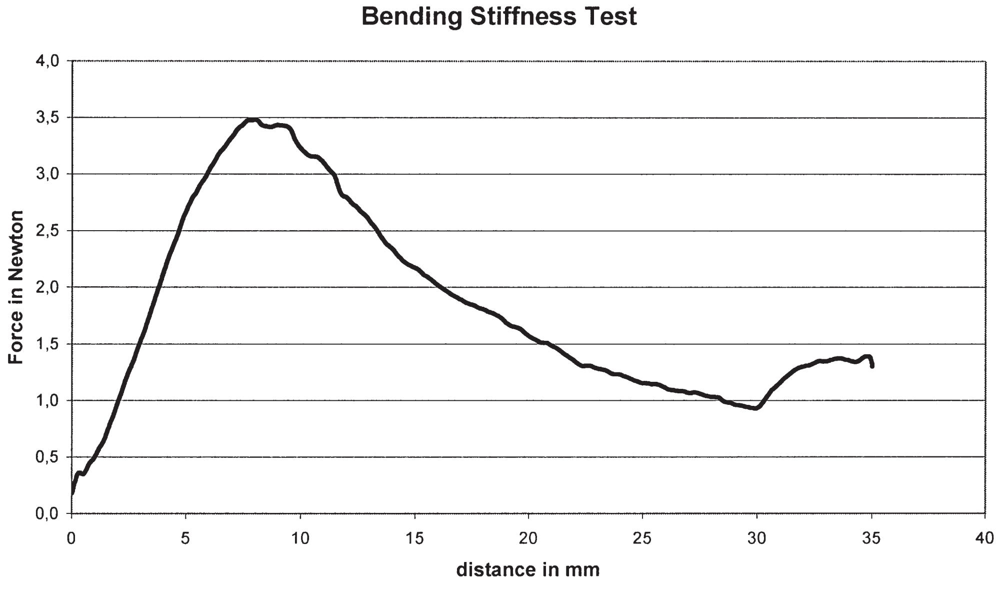

Figure 4. Force displacement curve of the bending stiffness test.

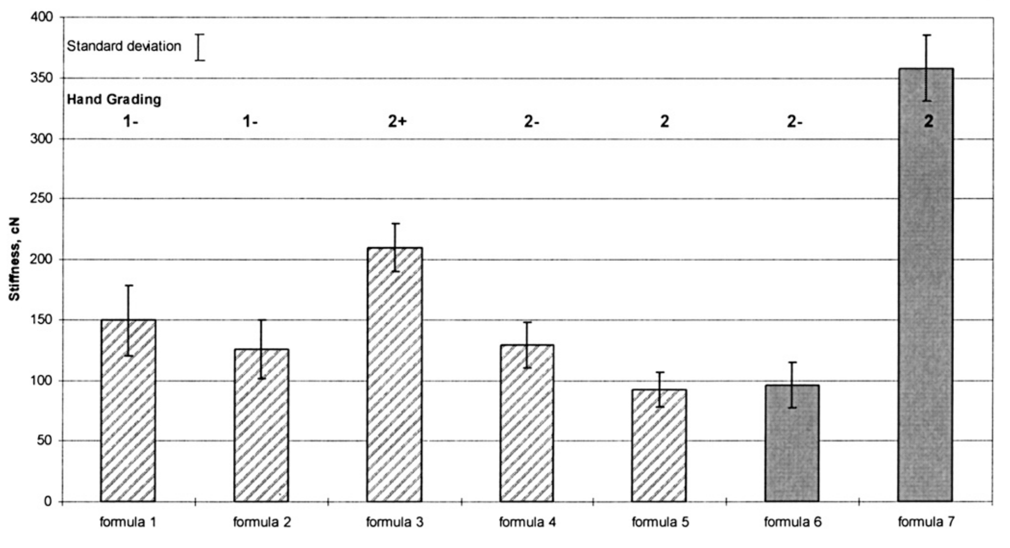

Figure 5. Result of the three-point stiffness test after gel application (striped-grey data set: BASF formulations; full-grey data set: market products). Hand grading: 1 = highest, 4 = lowest. Breaking of the strains by hand. For formulas, see Table I.

formulation 7. The high stiffness of > 350 cN could not confirmed by the hand-rating panel. Hand grading gave only a score of 2. This low degree of correlation is still not fully understood. Obviously there is strong evidence that the round-shaped hair geometry is not practically useful to assess the setting effect on hair.

The new two-point stiffness test on flat hair strands after application of styling gels (see Table I) was developed to overcome those discrepancies between hand grading and the stiffness test (see Figure 6). The two-point stiffness test on flat hair strands gives lower stiffness values than the three-point measurement. Especially for formula 7 the difference is obvious.

The stiffness measured on flat hair strands shows a much better correlation with hand grading. Now the gel with the highest stiffness values (formula 1, 160 cN) was rated to have the highest setting effect by hand grading (-1). Gels with lower stiffness values (formula 3–7) were consequently rated lower by hand grading (2).

The accuracy of the new two-point method is very good with standard deviations less than 20%. A confidence interval of > 95 % could be achieved by measuring each hair strain four times at different positions (see Figure 3). Significant differences between the stiffness values at these different positions could not be detected. Therefore, it was possible to calculate confidence intervals with data of $4 \times 10 = 40$ single measurements.

Gel formulas 3 (1.0% cationic thickener Polyquaternium-86, 2.5% polyvinylpyrrolidone) and 4 (0.5% anionic thickener carbomer, 3.0% polyvinylpyrrolidone) were further investigated in a panel test (4). Apart from stiffness (setting), further parameters were characterized. The panel test results are depicted in Figure 7. Fifty percent of the panelists could confirm a higher setting effect with formula 3 compared to formula 4. This correlates with the two-point and three-point stiffness results in Figures 4 and 5.

The new two-point bending stiffness test was also implemented to spray application. The focus was on fixative polymers in European water-free spray formulas based on dimethylether (DME) rsp propane/butane (P/B), a liquid pressure gas, as propellant (see Table II). Figure 8 depicts the results of the stiffness test and the hand grading after spray application. The striped-grey and full-grey data sets (formulas 1 to 6 in Figure 8) are formulations

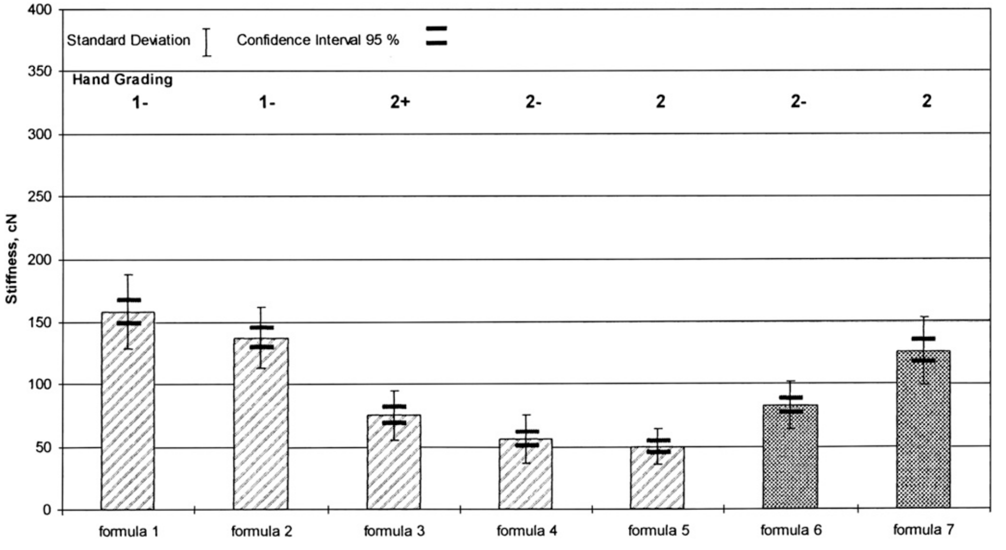

Figure 6. Result of the two-point stiffness test after gel application (striped-grey-data set: BASF formulations; full-grey data set: market products). Hand grading: 1 = highest, 4 = lowest. Breaking of the strains by hand. For formulas, see Table I.

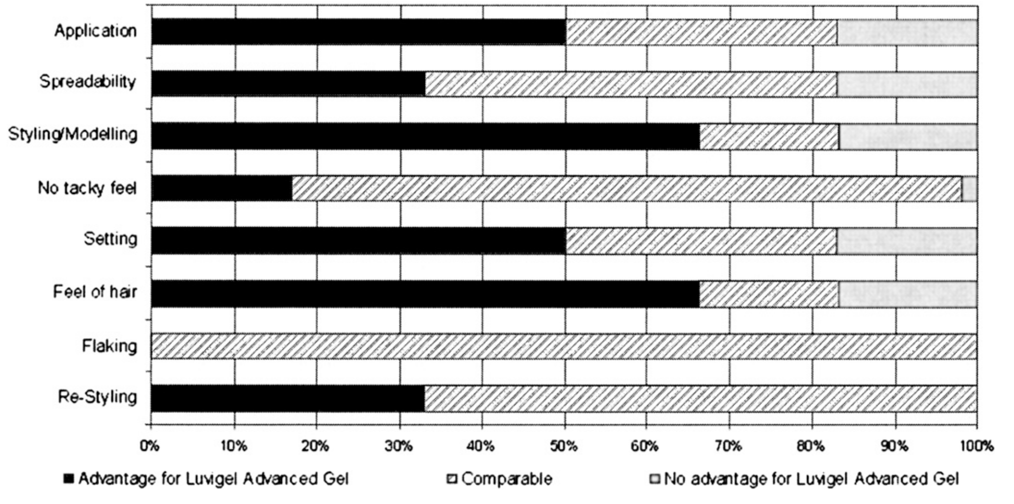

Figure 7. Result of panel test: Comparison of gels based of Polyquaternium-86 with polyvinylpyrrolidone and carbomer with polyvinylpyrrolidone; 28 answers.

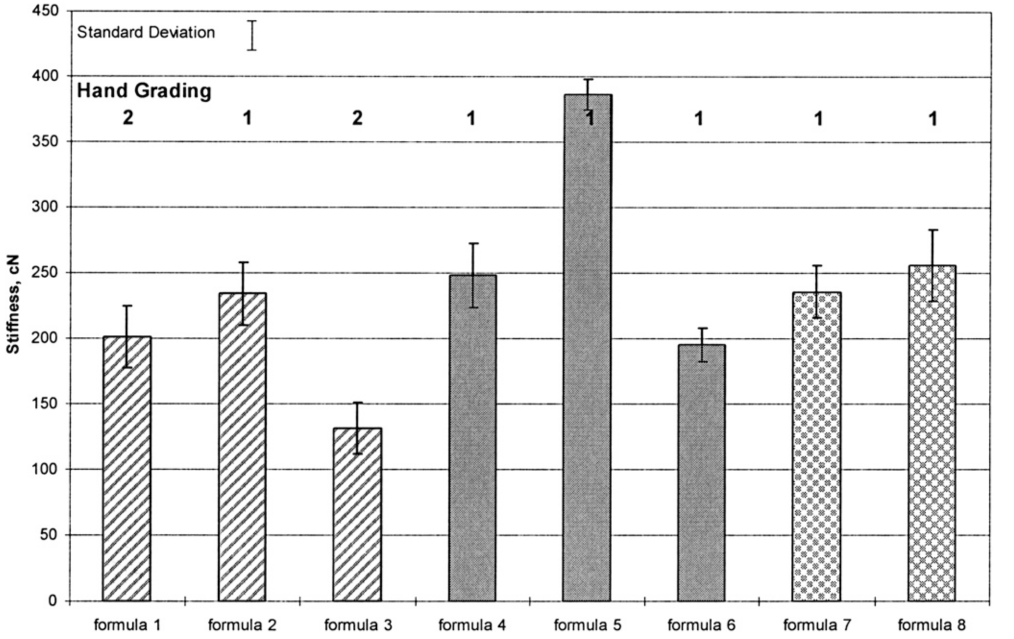

Figure 8. Result of the two-point stiffness test after spray application (striped-grey data set: formulations with 3% polymer content and DME; full-grey data set: formulations with 5% polymer content and DME; checkered-grey data set: formulations with 3% polymer content and P/B). Hand grading: 1 = highest, 4 = lowest. For formulas, see Table II.

with dimethylether (DME) as propellant, while the checkered-grey data sets (formulas 7 and 8 in Figure 8) are the same formulations with propane/butane (P/B) as propellant. In each set of formulations the polymer acrylates/t-butylacrylamide copolymer provides the best stiffness (formulas 2, 5, and 8 in Figure 8), followed by octylacrylamide/acrylates/butylaminoethyl methacrylate copolymer (formulas 1, 4, and 7 in Figure 8). The lowest stiffness was observed with VP/VA copolymer (formulas 3 and 6 in Figure 8).

The stiffness increased with increasing polymer content (striped grey and checkered grey: 3%, full-grey: 5%). The differences between the different polymers are more significant at higher polymer content. However, it was not possible to differentiate the different polymers at 5% solids content in the subjective test by hand grading. Due to the insolubility of VP/VA copolymer in propane/butane, the stiffness of this formulation could not be measured.

The subjective evaluation of the flat hair strains treated with hair spray formulations was less sensitive compared to the evaluation of the gel formulations (see Figures 5 and 7). Only formulas 1 and 3 were subjectively rated with a 2 grading (Figure 7, good setting). All other formulas were subjectively assessed with a 1 grading (very good setting). It was only possible to find subjective differences at 3% polymer content with DME as propellant. Acrylates/t-butylacrylamide copolymer performed best (see formulas 2, 5, and 8 in Figure 7).

The spray formulas 1 and 2 (see Table II) were further investigated in a half-side test (5). The results of the half-side test are depicted in Figure 9. Apart from stiffness (setting), further parameters were characterized. The setting effect is characterized by grading 3+ (good–satisfactory) for both formulas. The half-side test shows no significant differences between formulas 1 and 2.

We postulate that the different polymer distribution on hair is responsible for the lack of correlation between the stiffness test on flat hair strands and the half-side test on model heads. The preparation device (Figure 1) with a comb aligns the hair. This was found out to

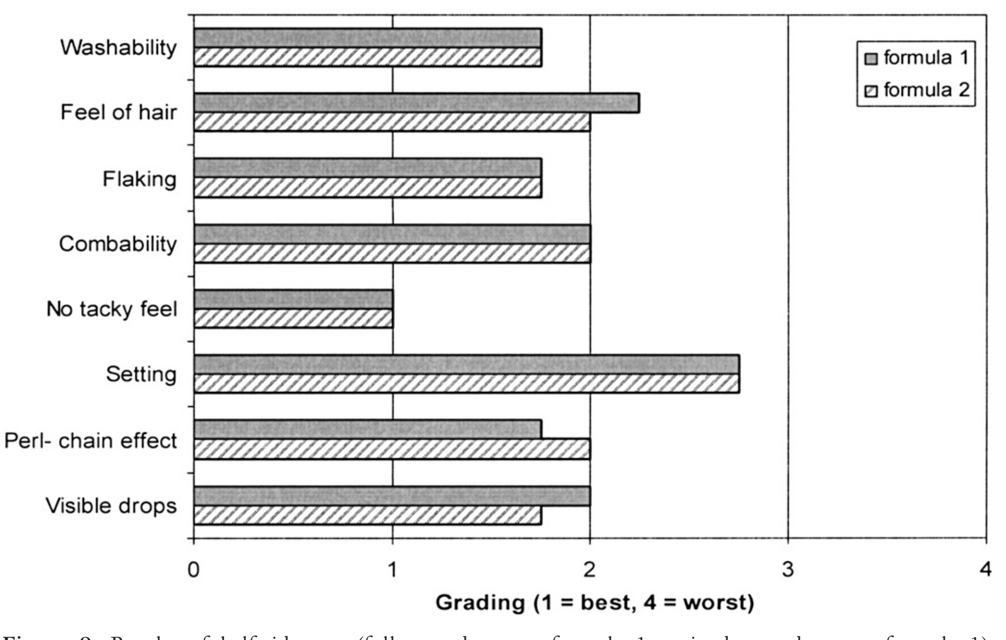

Figure 9. Results of half-side test (full-grey data set: formula 1; striped-grey data set: formula 1). Gradings: 1 = best, 4 = worst. For formulas, see Table II.

be necessary for reproducible stiffness test results. After combing the hair in the preparation device, the polymer/hair composite is seam-welded after drying of the polymer film. The model heads for the half-side tests are sprayed and dried without combing and without any mechanical impact. Therefore the polymer/hair composite is mainly spot-welded. The different polymer/hair architecture leads to different styling grades that overcome the influence of the different polymers. More subjective data like salon tests and panel tests will be necessary to prove if there are differences in setting behavior that have been observed by means of the new two-point stiffness test on flat hair strands.

SUMMARY

The presented two-point bending stiffness test method on flat hair strands is a new test for measuring the bending stiffness of hair styling products. The test can be used to assess the bending stiffness of hair gels as well as hair sprays. A preparation device is used to align the hair fibers to get reproducible results. In addition, this preparation device minimizes the influence of individual processors.

Results obtained with the two-point bending method on flat hair strands are in good agreement with sensorial and consumer tests for styling gels. The correlation between the stiffness test and subjective evaluations are much closer on flat hair strands compared to round-shaped hair strands. The best results by means of the two-point method on flat hair strains are achieved with the combination of acrylates/beheneth-25 methacrylate copolymer and VP/methacrylamide/vinyl imidazole copolymer in hair styling gels. This correlates very well with the subjective tests on those strains.

Stiffness tests on flat hair strands after spray application using different styling polymers did not correlate with practical relevant assessments. We postulate that different polymer/hair composites are responsible for these discrepancies. Hairs are seam-welded in the stiffness tests after alignment of the single hairs and are spot-welded in the half-side tests on model heads after spray application without mechanical impact before the assessment of the styling performance.

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