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**Flow properties of dental impression materials
by means of a modified sharkfin test at clinically
relevant times after mixing**

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1 Introduction

Dental impression materials have been manufactured with a main purpose of providing the dentistry world with contemporary materials which give the ability to register the intraoral hard and soft tissues as a mold for having afterwards a free bubble cast of reproduced fine details and stable dimensions, finally for the fabrication of eventual restorations. The impression materials are usually divided into two groups according to their elastic properties once set: non-elastic and elastic materials.

Non-elastic materials contain impression plaster, impression compound, and impression waxes.

Elastic impression materials are divided into two groups:

1. The hydrocolloid materials which are divided into two groups as well, reversible (agar) and irreversible (alginat) materials.
2. The synthetic elastomeric materials : - Polysulphides.
 - Polyether.
 - Silicones (condensation and addition).

Synthetic elastomeric impression material are widely used due to their ability to produce impressions with stable dimensions and adequate tear resistance.

In recent years, several elastomeric impression materials have been marketed, and many studies have been reported on this field [6,8,9,10,11,15,17].

Normally, the flow properties of a material are characterized by rheological methods using e.g. a rheometer with plate-plate or plate-cone system. Different measurements can be done for determining the yield point like the flowcurve or a hardening curve. The calculation of the yield point is complex and difficult. A special knowledge about rheological parameters like storage modulus, loss modulus, tan delta and stress and strain parameters is necessary.

For dental impression materials another simple test exists which is called sharkfin test. There are quite a few publications about sharkfin test [2,7,12,14,19]. By a modified form of this test, used in this study, a flowcurve can be registered during polymerization.

Up to this point, the measurement of flow properties by means of the sharkfin test has been done at different times after mixing, e.g. at 30, 60, 90 s up to 150 s after

mixing [2,7,14]. In addition, wettability studies of unset materials have been done at such time intervals [2,7,19,20]. Such intervals have been chosen first of all in order to investigate the total working time given by the respective manufacturers of the materials that range from about 60 s up to 150 s.

However, the material properties at relevant working times in clinical practice remain unclear. Therefore, this study focused on determining clinically relevant time intervals between the mixing of the impression material and the first contact of the material with oral tissues. In a second step, flow properties of different type 2 and type 3 impression materials were analyzed at these times after mixing.

1.1 Aims of the study

A main aim of this work is to determine the relevant working times for clinical practice. This clinical part will be done in the Department of Prosthodontics. The clinically working time of a large number of impressions will be measured with stopwatch by the same researcher. The impressions will be taken by fourteen different dentists, who will use ImpregumTM PentaTM (3M ESPE) as type 2 and PermadyneTM GarantTM 2:1 (3M ESPE) as type 3 with the one-step technique.

On the basis of these measured application times, another aim of this work is to analyze the flow properties of several elastomeric impression materials by means of the modified sharkfin test. So, this study consists of an in vivo and in vitro part.

In summary, the aims are:

1. To determine the relevant working times actually used in clinical practice.
2. To characterize the flow properties of type 2 and 3 elastomeric materials under the conditions of these working times by means of the sharkfin test.

2 Materials and Methods

2.1 Clinical trial

2.1.1 Study protocol

Between the middle of October and December of 2006 consecutively the working time of 86 clinical cases were measured. Fourteen different clinicians performed the impression by 48 different patients of which were 51 in the lower and 35 in the upper jaw. In 69 cases the impression was taken of only natural abutment teeth, in 15 cases only implants and in the last 2 cases implants together with natural abutment teeth. In the average case 3 abutments (range 1 to 10 abutments) had to be treated.

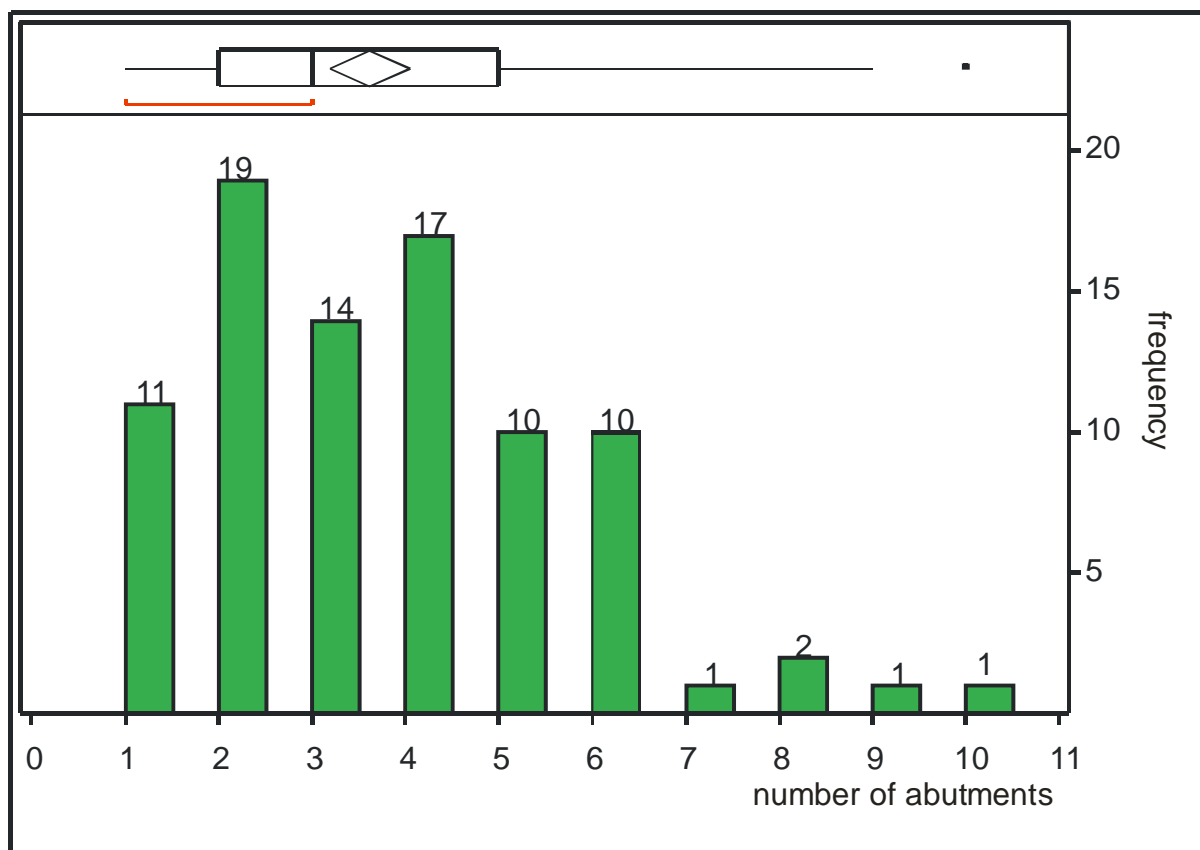


Fig 1: Frequency of cases with different numbers of abutments which the impressions were performed (box plot represents the statistical deviation of the number of cases)

The general interpretation of the box plot and the histogram plots is that the values are summarized in an outlier box plot comprising 50% of the values of the sample in

the box itself. The line across the middle identifies the median and the means diamond indicates the sample mean and the whiskers the 95% confidence intervals (CI). The red bracket on the left hand side identifies the shortest half, which is the most chosen 50% of the observations.

As impression material a combination between Impregum™ Penta™ Typ2® as regular body and Permadyne™ Garant™ Typ3® (Manufacturer 3M ESPE, Seefeld, Germany) as light body was used. In most cases commercially available trays (Algilock® trays from Hager&Werken, Duisburg, Germany) were used.

With a simple stopwatch different times between the loading of the impression tray and the complete setting time of the impression material were measured. All measurements were performed by the same person. Five different time spans were measured as follows:

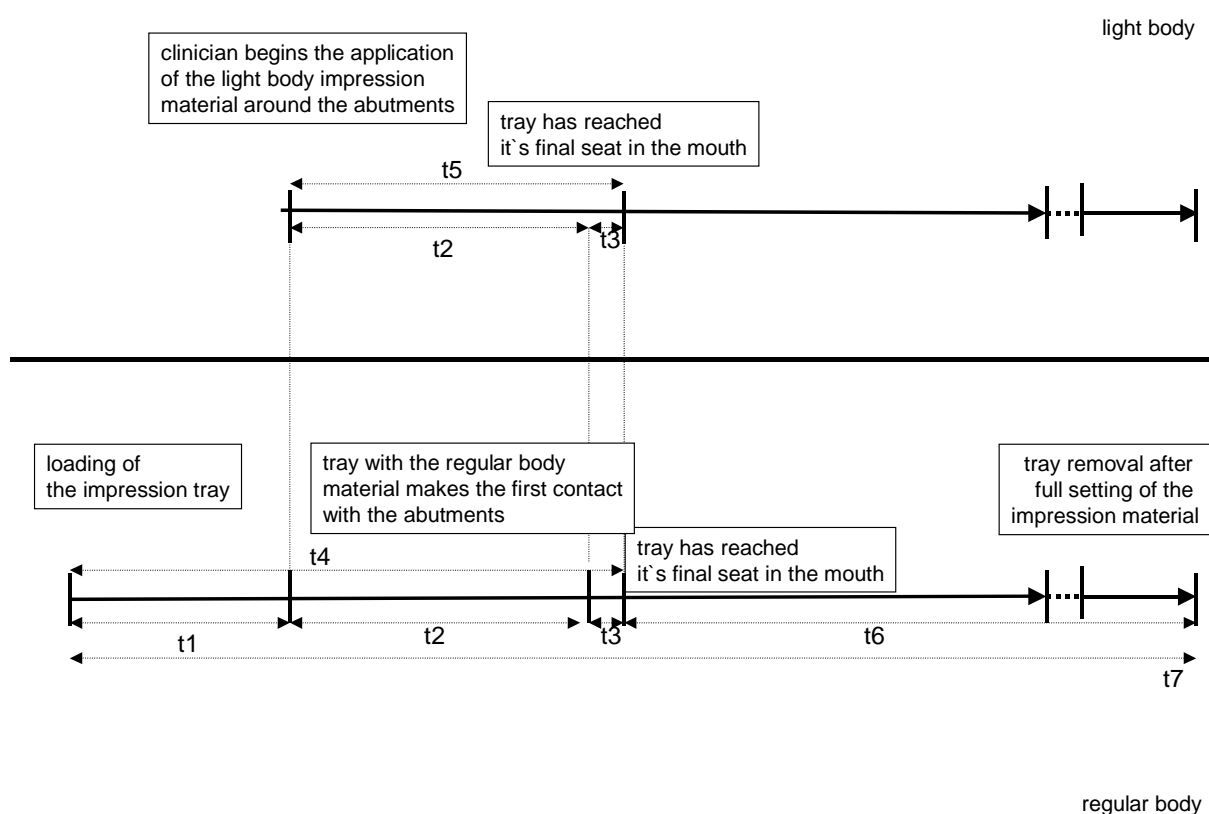


Fig 2: Scheme of the clinically measured times

Tab 1: Clinically measured times

The first stopwatch click	begin of the loading of the impression tray with the regular body material
The second stopwatch click	clinician begins to apply the light body material around the abutments
The third stopwatch click	first contact of the regular body material with the abutments
The fourth stopwatch click	tray has reached it's final seat in the mouth
The last stopwatch click	tray is removed after the full setting of the impression materials

The time t_1 is the time period between the first and the second stopwatch click.

The time t_2 is the time period between the second and the third stopwatch click.

The time t_3 is the time period between the third and the fourth stopwatch click.

The time t_4 is exactly the time which was displayed by the fourth stopwatch click.

The time t_5 is the sum of t_2 and t_3 as the time between the beginning of the application of the light body material around the abutments and the final seat of the impression tray.

The time t_6 was calculated as the difference between t_7 and t_4 meaning the time span between the final seat of the impression tray in the mouth and its removal after reaching the full setting of the impression material.

Finally, because the flow properties of the impression material were of major interest in this study, an additional time span, which is on average necessary till the impression tray makes the first contact in the mouth with the abutments, was calculated as the sum of t_1 and t_2 (t_1+t_2). Because in some of the clinical situations the clinician started actually before the loading of the impression tray with the application of the light body around the abutments a negative sign of t_1 was obtained. Therefore, this value was corrected for the addition of t_1+t_2 and the addition was done with the absolute value of t_1 .

Clinical examples for each measured time



Fig 3: Beginning of t1: loading of the impression tray with the regular body material



Fig 4: Beginning of t2: clinician begins to apply the light body material around the abutments



Fig 5: Beginning of t3: first contact of the regular body material with the abutments



Fig 6: t4: tray has reached its final seat in the mouth

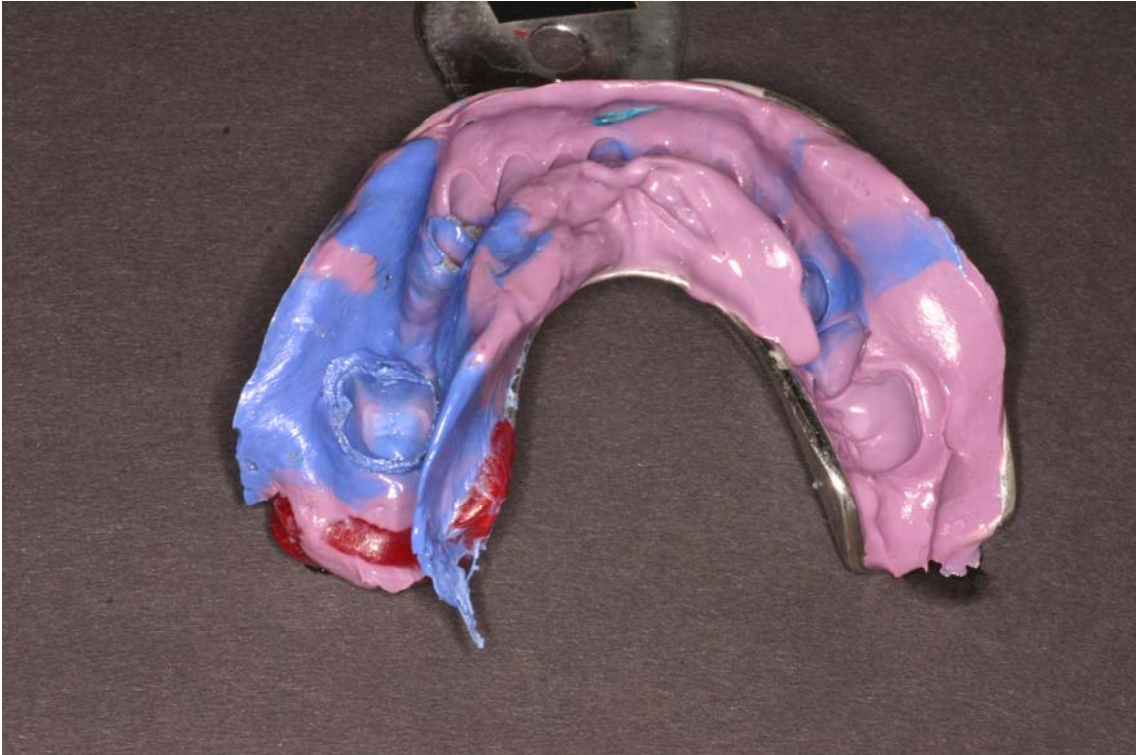


Fig 7: t7: tray is removed after the full setting of the impression materials

2.2 Laboratory trials

2.2.1 Impression materials

Two types of impression materials which were manufactured by three different companies were used.

Tab 2: impression materials

Material	Code	Manufacturer	Viscosity-type	Lot	Chemical type	MrWT [s] *
Aquasil Ultra Monophase	AUM	DENTSPLY Caulk (U.S.A)	2	060818	Addition Silicone	135 –165
Aquadyn medium soft quick base 07003	A-MSQ	KETTENBACH (Germany)	2	60471	Vinyl polysiloxane (VPS)	Unknown
Aquadyn medium soft regular base 07004	A-MSR	KETTENBACH (Germany)	2	60471	Vinyl polysiloxane (VPS)	Unknown
Aquadyn medium transfer base 07005	A-MTR	KETTENBACH (Germany)	2	60471	Vinyl polysiloxane (VPS)	Unknown
Impregum Penta	Impregum-P	3M ESPE (Seefeld,Germany)	2	269530	Polyether	165
Impregum Penta Soft	Impregum-P-S	3M ESPE (Seefeld,Germany)	2	269439	Polyether	165
Impregum Penta Soft Quick	Impregum-P-S-Q	3M ESPE (Seefeld,Germany)	2	270932	Polyether	60
Aquasil Ultra LV	Aquasil-LV	DENTSPLY Caulk (U.S.A)	3	060522 and 060920	Addition Silicone	135 -165
Aquadyn Light	Aquadyn Light	KETTENBACH (Germany)	3	06998 and 06996	Vinyl polysiloxane (VPS)	Unknown
Impregum Garant L DuoSoft	Impregum-LDU-S	3M ESPE (Seefeld,Germany)	3	66029	Polyether	120
Permadyne Garant 2:1	Permadyne-Gar 2:1	3M ESPE (Seefeld,Germany)	3	62315	Polyether	120
* Manufacturer's recommended working time.						

All materials which were produced by Kettenbach were experimental materials. The working times of these materials were unknown.

2.2.2 Method of sharkfin test and data evaluation

2.2.2.1 The apparatus

The sharkfin test was performed on an apparatus designed by 3M ESPE which was further developed by Section "Medical Materials and Technology" [2,7,12,14,19]. The apparatus consists of:

- A base with a cylindrical well (30 mm around, 5 mm deep) in which the pieces of the receptacle (1) are fitted.
- The receptacle pieces (1) consist of 2 identical semi-circular pieces (14 mm high) which sit in the well of the base. The resulting receptacle holds 8 ml of material.
- The sharkfin mold (2) consists of two pieces that fit within the plunger (3). The form produced by the mold is similar in appearance to a sharkfin. Two sizes of molds were used. For type 2 material a mold 2 mm wide at the base was used. For type 3 material a mold 1 mm wide at the base was used.
- The plunger (3) holds the pieces of the mold together. The plunger itself slides up and down freely within the main housing casing (4). The plunger and the mold can then be dropped into the receptacle so that the mold can receive the material. The total combined weight of the plunger and the mold is 147 g.
- The main housing casing (4) fits over the receptacle. It holds the plunger and serves to guide the mold into the material.
- The release pin (5) holds the plunger in place within the housing casing. The housing is then fitted over the receptacle and the release pin is removed when the experiment is ready to be performed. Once the pin is removed the plunger drops into the receptacle so that the mold can receive the material.
- The extracting tool (6) is used to push the mold out of the plunger after the experiment has been completed.
- The reflector plate (7) is fitted on the end of the plunger.

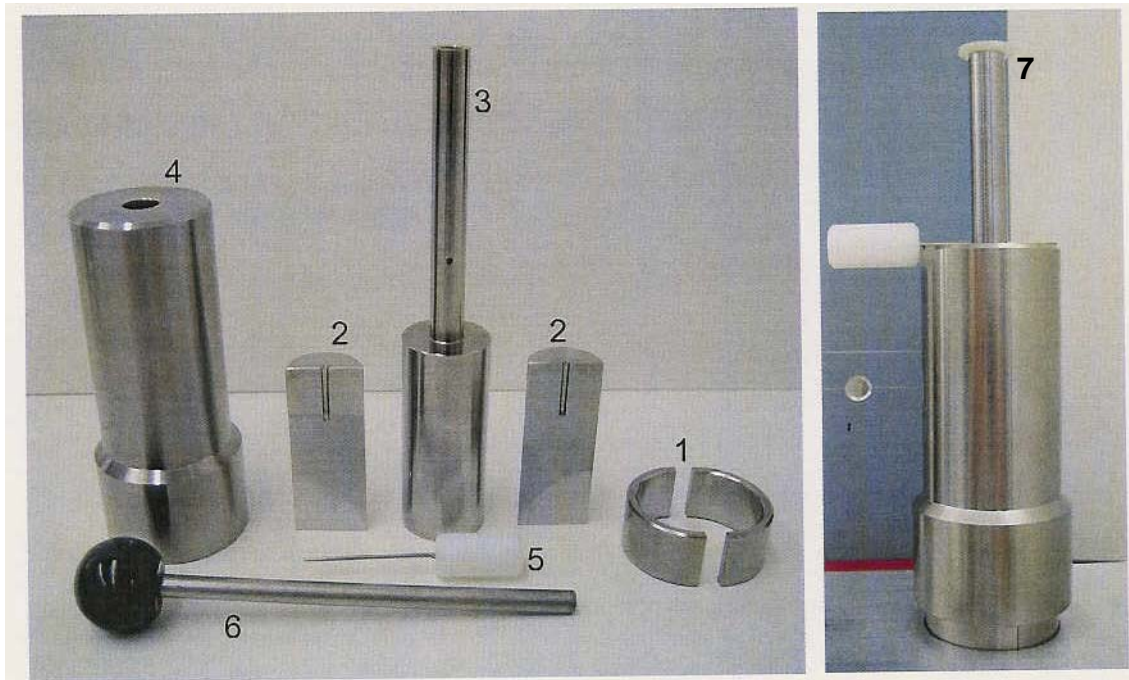


Fig 8: Equipment for Sharkfin test

2.2.2.2 Description of Experiment

To perform the experiment the material and the receptacle (1) were brought to 23° C. The entire assembled housing unit (2, 3, 4, 5, 7) was brought to 35° C. The material was then applied to the receptacle until the receptacle was full (8 ml). Type 3 was applied to the receptacle with a hand dispenser while Type 2 was applied by means of an automatic dispenser ESPE Pentamix 2. The material was applied to the receptacle and the entire assembled housing unit was placed over the receptacle within a total time frame of 26, 50 or 81 seconds. At the end of the respective three times the release pin (5) was pulled. The 147 g plunger and mold (2, 3, 7) were allowed to sink slowly into the receptacle. In order to simulate the conditions of clinical practice, the plunger was not allowed to drop freely into the receptacle, but was held by hand so that it sinks slowly into the receptacle. The mold was allowed to sit in the receptacle for five minutes. During these five minutes the flow of the tested materials was measured by means of a laser, which was connected to a computer. The laser recorded the impression depth of the plunger over the five minutes. Laser equipment and software program were developed by the Section “Medical Materials and Technology”. After a 5 minute period of time the assembled housing unit and the two pieces of the receptacle were removed (1, 2, 3, 4, 7) from the base. The reflector

plate (7) was removed from the plunger. The plunger and receptacle pieces (1, 2, 3) were removed from the housing casing, and the extracting tool (6) was used to push the mold and receptacle pieces (1, 2) out of the plunger (3). The pieces of the receptacle (1) were removed. The excess material was cut away with a scalpel and the two pieces of the mold (2) were separated. The resulting form of the material that was in the mold resembled a sharkfin and the height of the sharkfin was measured with a digital caliper.

With the data of the impression depth of the plunger time-depth-curves were recorded for each material and for all three times.

Statistical calculation of independent t-test with $p=0.05$ was made with the measured sharkfin heights from all the materials and at all clinically measured times by using the software Microcal Origin Vers. 6.0.

2.2.2.2.1 Method of Experiment

This is an example how this experiment was performed:

Step1:



Fig 9: Pentamix 2 will be brought into position.

For all materials of type 2, the automatic dispenser Pentamix 2 (3M ESPE) was used and placed on a lifting platform in order to allow the material to flow more easily into the receptacle.

Step2:



Fig 10: Filling of the receptacle with material, the stopwatch was started.

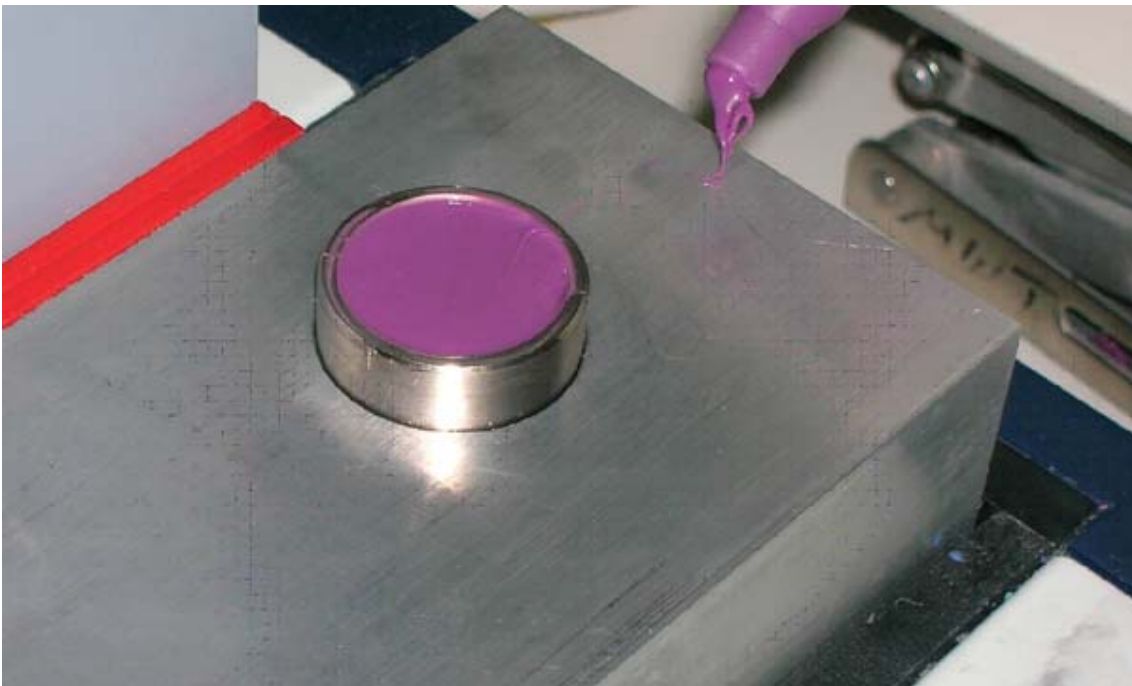


Fig 11: A spatula was used to level the material.

Step 3:



Fig 12: Starting the measurement.

After each of the three clinically measured times, i.e. 26, 50, and 81 seconds, the entire assembled housing unit was fitted over the receptacle which was filled with material. Then the laser was started. Immediately after that, the white release pin was removed. The plunger was held by hand so that it sank slowly into the receptacle in order to simulate the conditions of clinical practice.

Step 4:

After 5 minutes, the experiment was finished. The housing unit was disassembled.



Fig 13: The completed sharkfin.

2.2.2.3 Examples of the results of the experiment

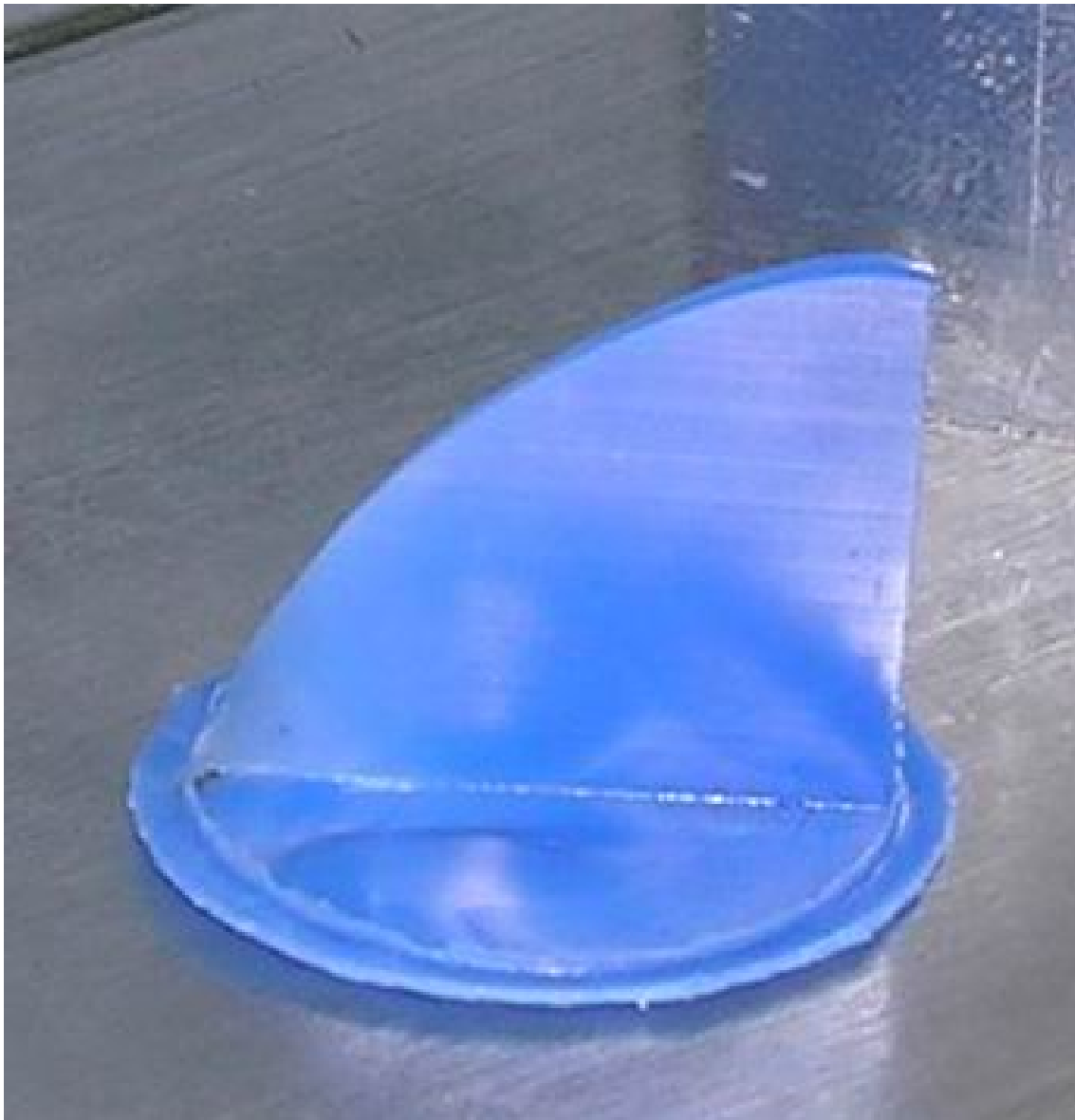


Fig 14: Sharkfin of Permadyne Garant 2:1®.

Here is an example from type 3 material. One can see one of the largest fins, but with a thin base.



Fig 15: Five different specimens of experiments with Permadyne Garant 2:1® at 81s.

Fig.15 shows the thin bases and the sharkfins. Sometimes on some bases holes were noticed.

3 Results

3.1 Results of the clinical trial

Table A in the Appendix shows the basic results of the clinically measured times. From this table three different working times were calculated and further used for evaluation of the respective flow properties.

Because the sharkfin tests in the laboratory with different impression materials were performed on the basis of t_1+t_2 , the distribution of this time is depicted in **Fig 16**.

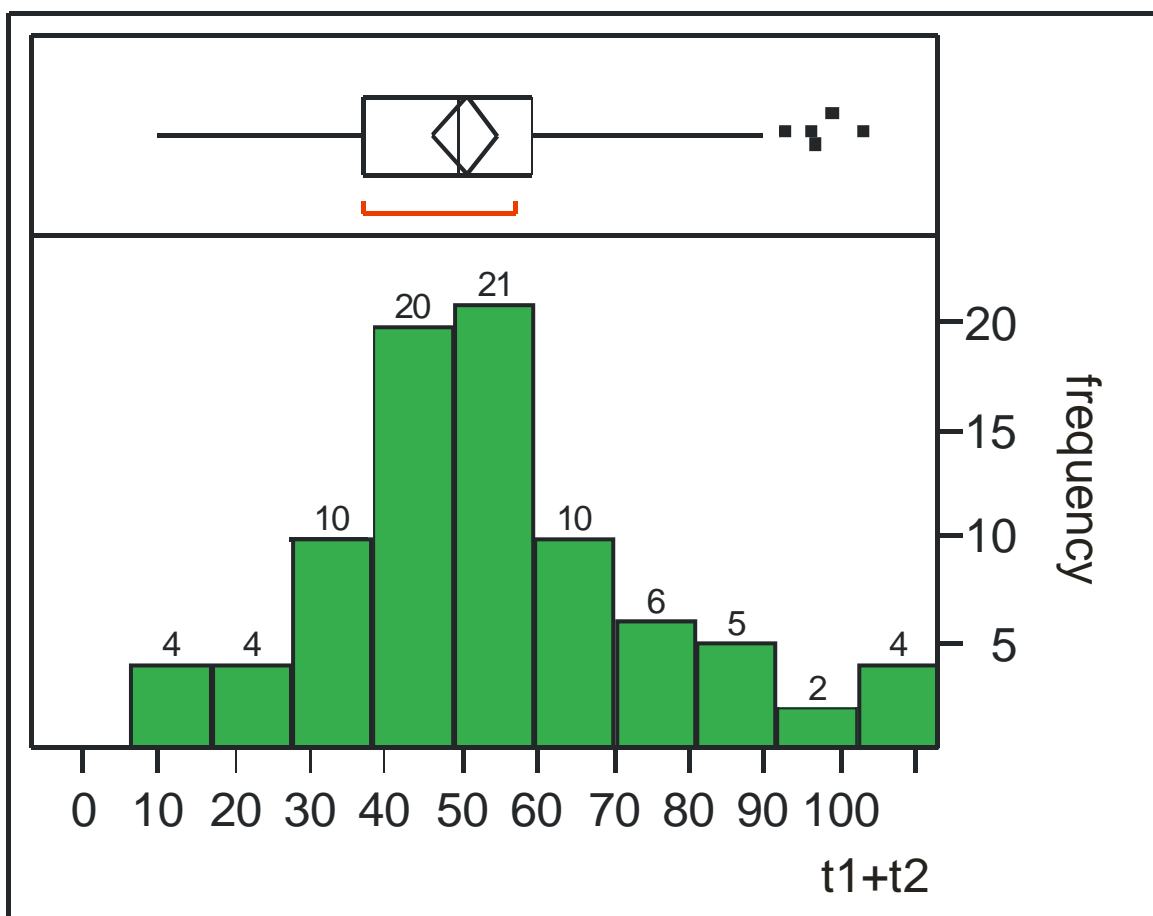


Fig 16: Frequency of t_1+t_2 as time span between loading of the impression tray with the regular impression material at the first contact of the tray with the abutments after applying of the light body material.

On the basis of the results of these clinical measurements we decided to perform the sharkfin test in the laboratory at the times of the 10th and 90th percentiles and of the median value at 49.5s, 25.8s (10th percentile) and 80.6s (90th percentile).

The results include the measured heights of sharkfins and the times analyzed from the flow curves, when the materials cannot flow any more.

3.2 Results of the laboratory trial

3.2.1 Results of the measured sharkfin heights

The tables 3 and 4 show all measured sharkfin heights of all impression materials under research according to all three clinically measured times. From each impression material, five or six sharkfins were made, the height of every shark fin was measured and from each group the mean (=x) and standard deviation (=s) were calculated, which can be seen in table 3 and 4.

Tab 3: Measured sharkfin heights (all values in mm) of impression materials type 2 (regular body).

Product: AUM

time [s]	25.60	49.50	80.60
sample 1	7.23	5.03	2.49
sample 2	7.15	5.12	2.45
sample 3	7.18	5.16	2.43
sample 4	7.22	5.07	2.41
sample 5	7.70	5.14	2.42
sample 6	7.35		
x	7.31	5.10	2.44
s	0.21	0.05	0.03

Product: A-MSQ

time [s]	25.60	49.50	80.60
sample 1	12.75	12.00	8.73
sample 2	13.85	11.98	8.71
sample 3	13.16	12.50	8.15
sample 4	13.53	11.85	8.14
sample 5	13.47	12.43	8.31
sample 6	13.01		
x	13.30	12.15	8.41
s	0.40	0.29	0.29

Product: A-MSR

time [s]	25.60	49.50	80.60
sample 1	14.18	13.74	12.15
sample 2	13.70	14.41	12.75
sample 3	13.60	13.45	12.56
sample 4	14.30	13.29	12.69
sample 5	13.85	13.30	12.23
sample 6			
x	13.93	13.64	12.48
s	0.30	0.47	0.27

Product: A-MTR

time [s]	25.60	49.50	80.60
sample 1	9.90	9.00	7.62
sample 2	9.40	9.04	8.60
sample 3	9.80	9.30	8.10
sample 4	9.80	9.17	8.10
sample 5	9.81	9.16	7.50
sample 6			
x	9.74	9.13	7.98
s	0.20	0.12	0.44

Product: IMPREGUM-P

time [s]	25.60	49.50	80.60
sample 1	13.33	12.00	11.48
sample 2	13.32	12.00	11.80
sample 3	12.50	12.19	11.82
sample 4	12.48	12.15	11.70
sample 5	12.00	12.01	11.69
sample 6	13.00		11.30
x	12.77	12.07	11.63
s	0.53	0.09	0.20

Product: IMPREGUM-P-S

time [s]	25.60	49.50	80.60
sample 1	10.15	9.95	8.47
sample 2	10.16	9.85	8.45
sample 3	10.00	9.75	8.05
sample 4	10.20	9.82	8.42
sample 5	10.15	9.88	8.35
sample 6			8.31
x	10.13	9.85	8.34
s	0.08	0.07	0.16

Product: IMPREGUM-P-S-Q

time [s]	25.60	49.50	80.60
sample 1	9.56	9.30	6.07
sample 2	9.65	9.29	5.08
sample 3	9.85	9.48	5.58
sample 4	10.15	8.63	6.43
sample 5	9.83	9.15	6.07
sample 6	9.82		
x	9.81	9.17	5.85
s	0.20	0.32	0.52

Tab 4: Measured sharkfin height (all values in mm) of impression materials type 3 (light body).

Product: PERMADYNE-GARANT 2:1

time [s]	25.60	49.50	80.60
sample 1	17.16	16.80	16.88
sample 2	16.82	17.08	16.45
sample 3	16.75	16.75	16.55
sample 4	17.45	16.88	16.69
sample 5	17.19	17.03	16.40
sample 6		17.10	16.60
x	17.07	16.94	16.60
s	0.29	0.15	0.17

Product: IMPREGUM-GARANT-L-DU-S

time [s]	25.60	49.50	80.60
sample 1	16.48	15.95	13.97
sample 2	17.35	15.97	13.87
sample 3	16.40	15.93	13.85
sample 4	16.44	15.95	14.29
sample 5	16.20	15.50	13.92
sample 6	16.38	16.12	14.20
x	16.54	15.90	14.02
s	0.41	0.21	0.18

Product: AQUADYN-LIGHT

time [s]	25.60	49.50	80.60
sample 1	17.55	15.99	14.48
sample 2	18.04	16.87	15.20
sample 3	17.30	16.96	13.79
sample 4	17.50	15.98	13.20
sample 5	18.20	16.99	14.28
sample 6	17.87	16.75	13.49
x	17.74	16.59	14.07
s	0.35	0.48	0.73

Product: AQUASIL-U-LV

time [s]	25.60	49.50	80.60
sample 1	5.27	4.75	3.30
sample 2	5.87	5.55	3.75
sample 3	5.85	5.30	3.15
sample 4	6.46	5.65	2.90
sample 5	5.75	4.64	2.66
sample 6			2.87
x	5.84	5.18	3.11
s	0.42	0.46	0.39

The next figures (17 and 18) show the mean values with standard deviation from table 3 and 4 of measured sharkfin heights of all tested impression materials depending on the three clinical times.

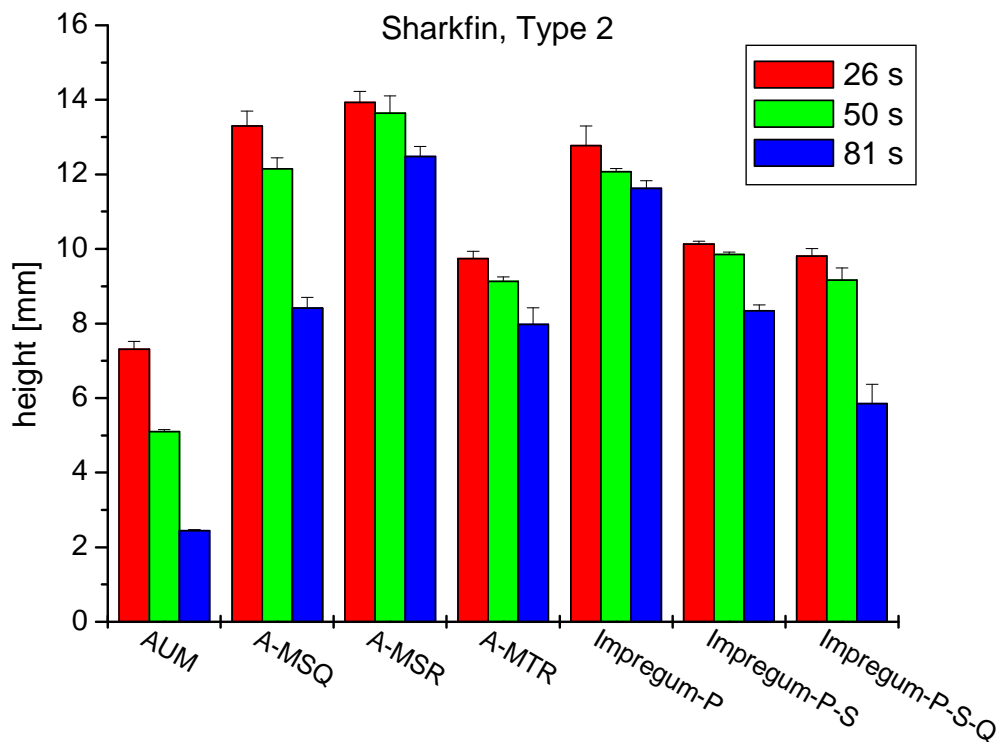


Fig 17: Measured sharkfin heights (values in mm) of Type 2 (regular body).

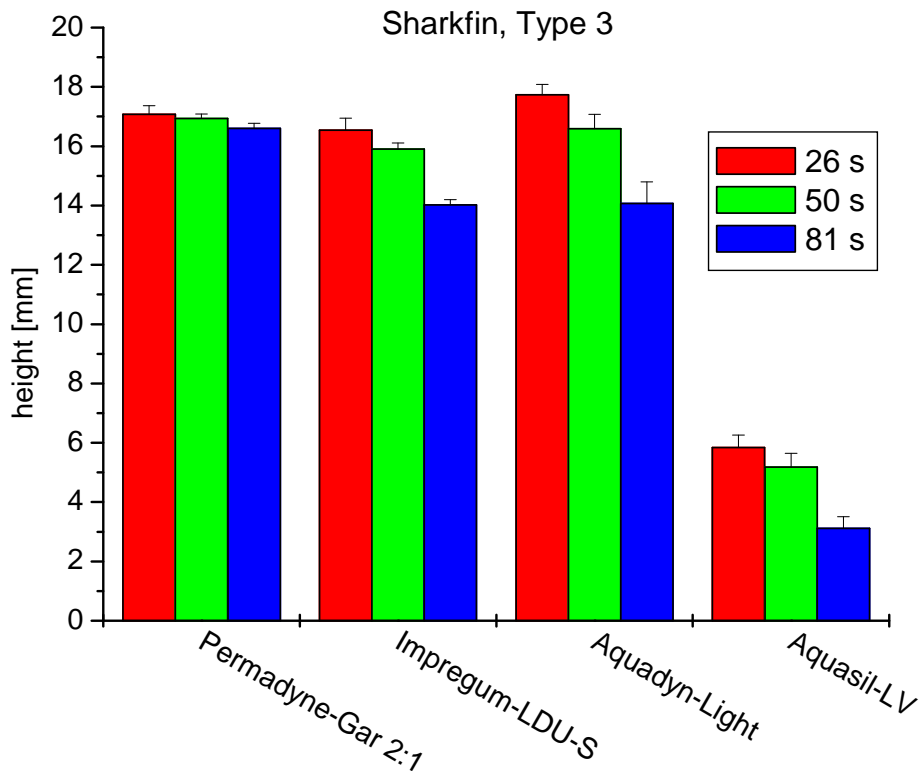


Fig 18: Measured sharkfin heights (values in mm) of Type 3 (light body).

The measured sharkfin heights correlate with the three different clinical times as shown in Fig. 17 and Fig. 18. The results showed that:

- At 26 seconds the fin heights are the highest.
- At 81 seconds the fin heights are the smallest for all the materials.

With Permadyne Garant 2:1 (type3) the heights were basically the same at all times.

The new experimental material Aquadyn Light (type3) had the highest fin of all tested materials at 26 seconds. The new material A-MSR (type2) showed the the highest fins of all the materials at all three measured times.

The material Aquasil ULV (type3) had the shortest fins for all three measured times. At 81 seconds Aquasil had the smallest fins for both type2 and type3. These results were borderline unacceptable (just over 2mm). Aquasil (type2 and type3) consistently produced the shortest fins at all three measured times of all tested materials.



Fig 19: Sharkfins of Aquadyn light® on the left, Permadyne Garand 2:1® in the middle and Aquasil ULV® on the right at 26s.



Fig 20: Sharkfin of Aquasil ULV® (type 2) at 81s.

Fig.19 shows the sharkfins of the materials Aquadyn-Light, Permadyne Garant 2:1 and Aquasil ULV at 26 s. Fig.20 shows AUM (Type 2) at 81 s with a very small sharkfin. At 26 seconds the difference between the new material and Permadyne is minimal. One can clearly see, however, that the difference between these two materials and Aquasil ULV is strong.

3.2.2 Results of the flow curves

In order to calculate the yield point with a numerical method the flow curves were transferred in a difference curve. An example for a flow curve and a difference curve can be seen in Fig. 21.

This graph depicts the equilibrium point by showing distance as a function of time. The focus here is on the first minute of the experiment, as the rise of the slope of the curve was nearly zero after this time. Increased time did not change the results. The equilibrium point can be determined when the slope of the curve showing distance as a function of time shallows and does not continue to rise.

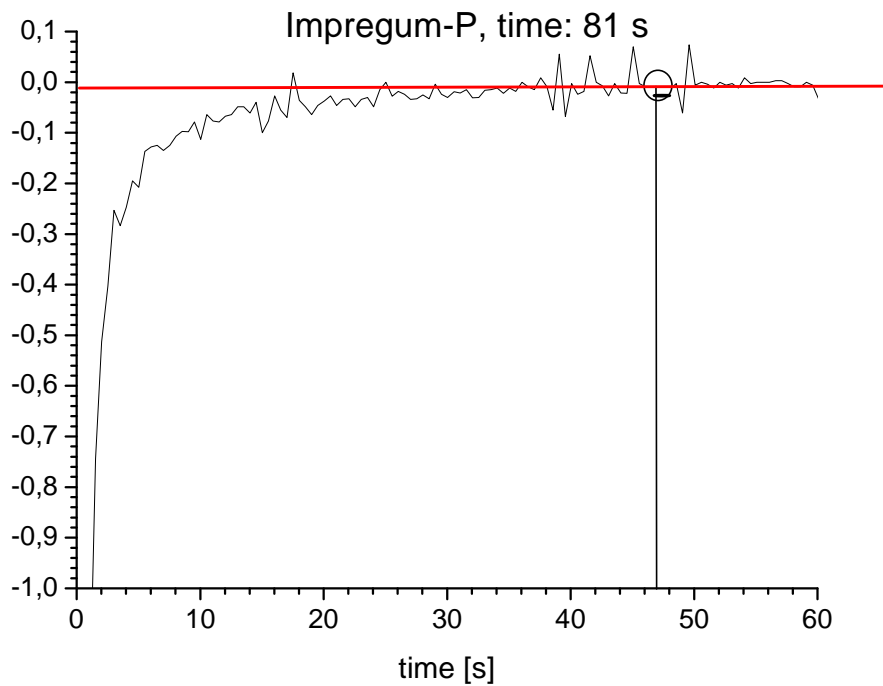
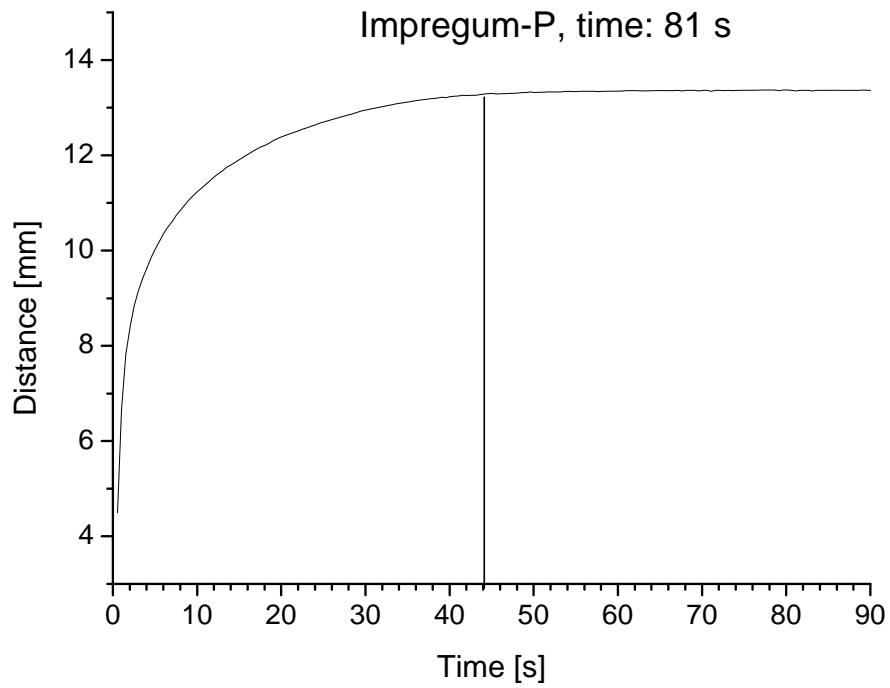


Fig 21: Example of a flow curve (upper) and a difference curve. The red line in the difference curve indicates the value of -0.01 .

Differences smaller than -0.01 were regarded to be small enough to determine the equilibrium point. The last number of the first five values < -0.01 was taken to determine the equilibrium time point (Fig. 21).

The time was respectively taken at this value for each material and each clinically measured time. The results can be seen in Fig 22.

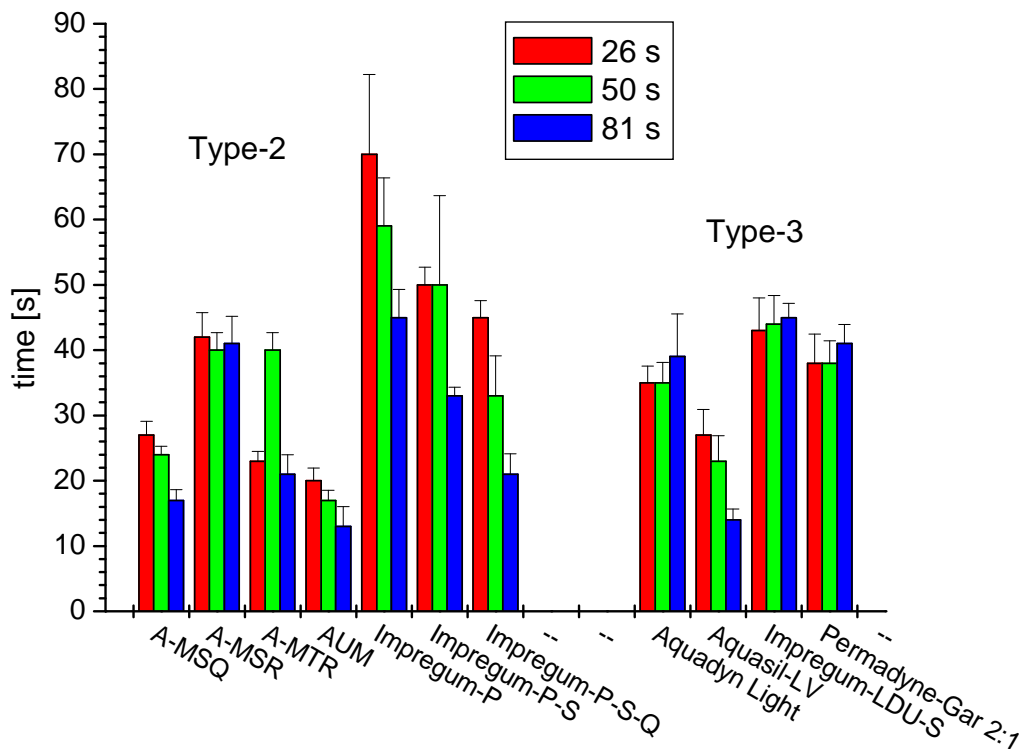


Fig 22: Calculated flowtimes from difference curves.

For type 2 materials, the shortest clinically measured time consistently produced the highest equilibrium point, and the longest clinically measured time always produced the lowest equilibrium point, with one exception of A-MTR and Impregum-P-S.

The Impregum-P material produced the highest equilibrium points of all type 2 materials at all three clinically measured times.

The AUM material had the lowest equilibrium points of all type 2 materials at the longest clinically measured time (81 s).

The A-MSR material had the smallest differences between the three equilibrium points of the three clinically measured times (practically the same flow time).

Impregum-P-S showed the same flow time at 21 and 50 seconds.

For type 3 materials, the polyether materials Impregum-LDU-S and Permadyne-Garant 2:1 had the highest equilibrium points at all three clinically measured times. Impregum-LDU-S had the highest equilibrium points for all three clinically measured times of all type 3 materials.

At the longest clinically measured time (81 sec), Aquasil-LV showed the lowest equilibrium point of all type 3 materials.

The material Aquadyn-Light and Permadyne-Gar 2:1 had very similar equilibrium points at the three clinically measured times.

The highest equilibrium points for type 3 materials were determined at the longest clinically measured time (81 sec), with one exception of Aquasil-LV.

3.2.3 Comparison of the flowtime and the sharkfin test

In this investigation all measured sharkfins and flowtimes were compared for each tested material and for each clinically measured time which can be seen in Tab 5 and Tab 6.

Tab 5: Comparison of measured flowtime and sharkfin height for type 2 (light body)
depending on clinically measured times

Type 2 (regular body)

time: 26 s

impression material	flowtime [s]	sharkfin height [mm]
A-MSQ	27	13.30
A-MSR	42	13.93
A-MTR	23	9.74
AUM	20	7.31
IMPREGUM-P	70	12.77
IMPREGUM-P-S	50	10.13
IMPREGUM-P-S-Q	45	9.81

time: 50 s

impression material	flowtime [s]	sharkfin height [mm]
A-MSQ	24	12.15
A-MSR	40	13.64
A-MTR	40	9.13
AUM	17	5.10
IMPREGUM-P	59	12.07
IMPREGUM-P-S	50	9.85
IMPREGUM-P-S-Q	33	9.17

time: 81 s

impression material	flowtime [s]	sharkfin height [mm]
A-MSQ	17	8.41
A-MSR	41	12.48
A-MTR	21	7.98
AUM	13	2.44
IMPREGUM-P	45	11.63
IMPREGUM-P-S	33	8.34
IMPREGUM-P-S-Q	21	5.85

The tables with type 2 materials show that the polyether based materials have always the longest flowtime of all tested materials and at all clinically measured times with exception of Impregum-P-S-Q at 50 and 81 seconds and Impregum-P-S at 81 seconds. According to the manufacturer's recommended working time (60 seconds), Impregum-P-S-Q was not supposed to give good results at 81 s. Compared to the new materials A-MSQ and A-MSR the sharkfin heights of the polyether materials are similar.

- At 26 seconds:

The material Impregum-P had the longest flowtime of all tested materials, but the highest sharkfin was shown by the new material A-MSR.

The materials AUM and A-MTR showed the shortest sharkfins and flowtimes, respectively.

The new material A-MTR and AUM have the same flowtime, but the new material A-MTR showed bigger sharkfins.

From all Polyether materials, Impregum-P-S-Q showed always the shortest flowtime, however, compared to the new material A-MSQ, the flowtime of Impregum-P-S-Q was 18 seconds longer, and its sharkfin height was 3.49 mm shorter.

The materials A-MSR, A-MSQ, and Impregum-P had the highest sharkfins, respectively.

- At 50 seconds:

In general, the situation is similar to 26 seconds, but all the values were respectively lower, with one exception; A-MTR showed a 17 seconds longer flowtime.

- At 81 seconds:

A-MSR, Impregum-P, and A-MSQ, respectively, showed the highest sharkfins.

All materials showed a lower flowtime with exception of A-MSR. AUM had the shortest flowtime and sharkfin.

At 26, 50, and 81 seconds, respectively, all the materials showed a reduction of flowability.

The materials Impregum-P-S-Q, A-MSQ and AUM showed a significantly reduction of flowability at 81 seconds contrary to the materials Impregum-P and A-MSR which showed a slight reduction.

In spite of the manufacturer's recommended working time for Impregum-P-S-Q of 60 seconds, it gives a sufficient result at 81 seconds.

Tab 6: Comparison of measured flowtime and sharkfin height for type 3 (light body) depending on clinically measured times.

Type 3 (light body)

time: 26 s

impression material	flowtime [s]	sharkfin height [mm]
AQUADYN-LIGHT	35	17.74
AQUASIL-LV	27	5.84
IMPREGUM-LDU-S	43	16.54
PERMADYNE-GAR2:1	38	17.07

time: 50 s

impression material	flowtime [s]	sharkfin height [mm]
AQUADYN-LIGHT	35	16.59
AQUASIL-LV	23	5.18
IMPREGUM-LDU-S	44	15.90
PERMADYNE-GAR2:1	38	16.94

time: 81 s

impression material	flowtime [s]	sharkfin height [mm]
AQUADYN-LIGHT	39	14.07
AQUASIL-LV	14	3.11
IMPREGUM-LDU-S	45	14.02
PERMADYNE-GAR2:1	41	16.60

Table 6 shows that the materials of polyether base and the new material Aquadyn-Light had a similar behaviour with a little different in the values at every clinically measured time.

The material Aquasil-LV had always the shortest flowtime and sharkfin, however, the material Impregum-LDU-S had always the longest flowtime from all tested materials and at all clinically measured times.

- At 26 seconds:

The new material Aquadyn-Light had the highest sharkfin, but the shortest sharkfin and flowtime was noticed by the material Aquasil-LV.

The new material Aquadyn-Light and Permadyne-Gar 2:1 have practically the same sharkfin height and the same flowtime with only a slight difference. All materials showed practically the same sharkfin height.

- At 50 seconds:

Permadyne-Gar 2:1 showed the highest sharkfin with 0.5 -1 mm differences compared to Aquadyn-Light and Impregum-LDU-S.

- At 81 seconds:

Aquasil-LV has the lowest values. The new material Aquadyn-Light and Impregum-LDU-S had practically the same sharkfin height (14.07 and 14.02 mm). The material Permadyne-Gar 2:1 had the highest sharkfin. The material Impregum-LDU-S had the longest flowtime and a big sharkfin as well.

3.2.4 Sharkfin heights: statistical results

With the measured heights of the fins, independent t-tests with $p=0,05$ were made in two ways.

The fin heights of every material were compared at the 3 clinically measured times for type 2 and type 3 materials.

In all tables of this chapter the sign + indicates that the values are significantly different and the sign – indicates that the values are not significantly different.

3.2.4.1 Results for every single materials.

Tab 7: Results of independent t-test for type 2 materials.

Material: AUM

	50 s	81 s
26 s	+	+
50 s		+

Material: A-MSQ

	50 s	81 s
26 s	+	+
50 s		+

Material: A-MSR

	50 s	81 s
26 s	-	+
50 s		+

Material: A-MTR

	50 s	81 s
26 s	+	+
50 s		+

Material: Impregum-P

	50 s	81 s
26 s	-	+
50 s		+

Material: Impregum-P-S

	50 s	81 s
26 s	+	+
50 s		+

Material: Impregum-P-S-Q

	50 s	81 s
26 s	+	+
50 s		+

For the type 2 materials, the statistical results (Table 7) showed that all the sharkfin heights were significantly different ($p < 0.05$) with the exception of the new material A-MSR and Impregum-P at 50 seconds.

Tab 8: Results of independent t-test for type 3 materials

Material: Permadyne-Garant 2:1

	50 s	81 s
26 s	-	+
50 s		+

Material: Impregum-Garant-LDU-S

	50 s	81 s
26 s	+	+
50 s		+

Material: Aquadyn-Light

	50 s	81 s
26 s	+	+
50 s		+

Material: Aquasil-ULV

	50 s	81 s
26 s	+	+
50 s		+

All the type 3 materials showed significant differences ($p < 0.05$), with the exception of Permadyne-Gar 2:1 at 50 seconds compared to 26 seconds (Table 8).

3.2.4.2 Comparison of every material at every clinically measured time

3.2.4.2.1 Type 2 (regular body)

Tab 9: time: 26 s

	AUM	A-MSQ	A-MSR	A-MTR	Im-P	Im-P-S	Im-P-S-Q
AUM		+	+	+	+	+	+
A-MSQ			+	+	-	+	+
A-MSR				+	+	+	+
A-MTR					+	+	-
Im-P						+	+
Im-P-S							+

Tab 10: time: 50 s

	AUM	A-MSQ	A-MSR	A-MTR	Im-P	Im-P-S	Im-P-S-Q
AUM		+	+	+	+	+	+
A-MSQ			+	+	-	+	+
A-MSR				+	+	+	+
A-MTR					+	+	-
Im-P						+	+
Im-P-S							+

Tab 11: time: 81 s

	AUM	A-MSQ	A-MSR	A-MTR	Im-P	Im-P-S	Im-P-S-Q
AUM		+	+	+	+	+	+
A-MSQ			+	-	+	-	+
A-MSR				+	+	+	+
A-MTR					+	-	+
Im-P						+	+
Im-P-S							+

For the type 2 materials the statistical results depending on $p=0.05$ showed:

At 26 seconds, the new material A-MSQ did not show differences compared to Impregum-P, and the new material A-MTR was not significantly different from Impregum-P-S-Q.

All the other materials were significantly different as shown in Table 9.

At 50 seconds (Table 10), the new material A-MSQ did not show significant differences to Impregum-P, and the new material A-MTR was not significantly different from Impregum-P-S-Q.

At 81 seconds (table 11), the new material A-MSQ did not show significant differences to both A-MTR and Impregum P-S.

3.2.4.2.2 Type 3

Tab 12: time:26 s

	Per-Gar 2:1	Imp-G-LDS	Aquadyn-Light	Aquasil-ULV
Per-Gar 2:1		+	+	+
Imp-LDU-S			+	+
Aquadyn-Light				+

Tab 13: time:50 s

	Per-Gar 2:1	Imp-G-LDS	Aquadyn-Light	Aquasil-ULV
Per-Gar 2:1		+	-	+
Imp-LDU-S			+	+
Aquadyn-Light				+

Tab 14: time:81 s

	Per-Gar 2:1	Imp-G-LDS	Aquadyn-Light	Aquasil-ULV
Per-Gar 2:1		+	+	+
Imp-LDU-S			-	+
Aquadyn-Light				+

All materials showed significant differences with the exception of the new material Aquadyn Light compared to Permadyne-Garant 2:1 at 50 seconds (Table 13), and Aquadyn Light compared to Impregum-LDU-S at 81 seconds (Table 14).

4 Discussion

The sharkfin test was developed by the company 3M ESPE and was modified by the Section of Medical Materials and Technologies (MWT) with a laser distance sensor. The laser gives here the possibility to correlate the measured heights of the shark fins with the registered flowcurves. With the modified sharkfin test it is possible to get information about the flow properties of an impression material, a property which is usually characterized by rheological measurements. In general, a big sharkfin means good flow characteristics of a dental impression material.

Most authors of publications were using the sharkfin test by calculation of the sharkfin height only [2, 7, 12, 19]. The results of sharkfin heights from these publications] were compared to the datas from this investigation, which can be seen in table 15. Only in common used materials were compared.

Table 15: Sharkfin heights (mean values) of the readed publications compared with the results of this investigation

Publication No.	Materials	Sharkfin heights [mm] *	Sharkfin heights [mm] **
[2]	Impregum Garant LDu Soft	At 30 s: 16,1 At 60 s: 15.6 At 90 s: 14.4	At 26 s: 16.54 At 50 s: 15.90 At 81 s: 14.02
	Impregum Penta Soft	At 30 s: 9.9 At 60 s: 9.4 At 90 s: 9.1	At 26 s: 10.13 At 50 s: 9.85 At 81 s: 8.34
[7]	Impregum Penta	At 25 s: 12.6	At 26 s: 12.77
	Aquasil Ultra LV	At 25 s: 14.6	At 26 s: 5.84
	Permadyne Garant 2:1	At 25 s: 23.67	At 26 s: 17.07
	Impregum Garant LDu Soft	At 25 s: 23.69	At 26 s: 16.54
[12]	Impregum Garant LDu Soft	At 25 s: 13.0 At 90 s: 14.0	At 26 s: 16.54 At 81 s: 14.02

* results of publications

** results of this investigation

The table 15 shows similar results between this investigation and the publications [2,12], in spite of the different temperature conditions.

Different results were found compared to the publication [7], with one exception of Impregum Penta (type 2).

The publication of von Pastau [14] was the first which used the modified sharkfin test like in this study. The flowtimes in that study were calculated by using the flowplots and reading the values of time and distance with a ruler. In order to improve this manual method, one aim of this study was to find out an objective way to calculate the yield point from the flow curve.

Thus, the difference curve was used and the fifth continuing value <-0.01 mm and the corresponding time was determined.

The difference curve was an easy method to calculate the yield point for each tested material at all three clinically measured times. However, it was difficult to find out the respective correct smallest value for all materials which indicates equilibrium. Many values were tried, but only the value < -0.01 was proven useful, because the other ones were always still in the flow region.

Using difference curves is a fast method to determine the yield point, however, it is obligate to compare the time values from this difference curves with the time values from the respective flowcurves. The time differences between calculations by difference curves and manually read times using the flow curve were smaller than five seconds.

Actually, it is an open question if the value < -0.01 is always valid for other studies with the modified sharkfin test.

The advantage of the modified sharkfin test is that the experiment could be done in short time (about ten minutes) and with good reliability. Furthermore, the researcher does not need so much efforts and time to go well with it, due to its unproblematic features, and the test offers a good opportunity to determine the equilibrium point through the flowcurve which has the same meaning as the yield point in rheological measurements.

On the other hand, the researcher will always need someone else to assist him, if the clinically circumstances are considered (the researcher is supposed to remove the release pin with one hand and to hold the plunger with the other hand to allow to the plunger to sink slowly in the material, simultaneously the computer mouse should be clicked).

In the clinic and in the oral cavity where the impression will be taken, the possibility of wetting and contamination of surfaces with saliva or blood is always present. This case would be worth to be simulated with the sharkfin test but is very difficult to realize.

The entire assembled housing unit has to be put in the climatic exposure cabinet for about two hours to reach the temperature of 35°C and to keep it stable. The temperature of 35°C simulated the mouth temperature, and the materials and the laser with the well were brought to 23°C in order to simulate the temperature of the stored materials and trays.

From the point of view of the clinician, the best impression material is that, which captures and reproduces finest details of hard and soft tissues in the oral cavity, which has the longest processing time from start of mixing, and which has a short residence time in the mouth which is really more comfortable for the patient.

Therefore, another point of discussion is, if this test allows to give a recommendation to the working times of all tested experimental materials. At least it offers a possibility to choose the light and regular body materials, which will be used together in one-step technique. It will be better if both of light and regular body have the most similar flow features.

The following table (Tab. 15) shows the clinically measured processing time at 81 seconds in addition with the flowtimes from Tables 5 and 6 which is compared to the manufacturer's working time.

Comparison of Manufacturer's recommended working time and calculated flow times + clinically processing time in relation to the measured sharkfin heights.

Tab 16 : Comparison of manufacturers recommended working time and calculated flow times + clinically processing time in relation to the measured sharkfin

Material	Clinically measured processing time +flowtime +5 seconds **	MrWT [s] *	sharkfin height [mm]
AUM	99	135 -165	2.44
A-MSQ	103	Unknown	8.41
A-MSR	127	Unknown	12.48
A-MTR	107	Unknown	7.98
Impregum-P	131	165	11.63
Impregum-P-S	118	165	8.34
Impregum-P-S-Q	107	60	5.85
Aquasil- LV	100	135 -165	3.11
Aquadyn Light	125	Unknown	14.07
Impregum-LDU-S	131	120	14.02
Permadyne Garant 2:1	127	120	16.60
* Manufacturer's recommended working time			
** 5sec = from the start of mixing till the material flows out from the mixing tips			

According to the technical way of mixing, it is obligated to add 5 seconds to the clinically measured processing time. This five seconds include the time period from the start of mixing till the material flows out from the mixing tips.

The polyether based materials showed a high flowability independent of the temperature of the entire assembled unit. A similar behaviour was noticed between the new material A-MSR and Impregum-P. These materials had practically the same working time and the same sharkfin height.

The new material A-MTR and Impregum –P-S showed similar results. The new material A-MSQ and the Impregum-P-S have practically the same sharkfin height, however, pretty similar workingtime.

Impregum-P-S-Q showed superior flow properties, in addition to a reasonable sharkfin.

The materials Aquadyn-Light, Impregum Garant LDU Soft, and Permadyne Garant 2:1 showed excellent flow properties and very high sharkfins, with regard to Permadyne Garant 2:1 has the highest sharkfin with 2 mm difference, and the new material Aquadyn-Light has the lowest flowtime with 10 seconds difference.

From a practical point of view, the sharkfin itself will be acceptable, if it achieves 2 mm height as it is recommended by von Pastau [14]. Therefore, all the tested materials performed acceptable sharkfins at all clinically relevant measured times. However, a very important point was noticed, that Aquasil light and regular body gave very poor results compared to the others. At 81 seconds Aquasil Ultra Monophase was barely sufficient.

The light materials, as expected, showed better flow properties than regular materials. Also the Polyether materials and the new silicon materials showed similar flowproperties.

5 Conclusion

- The modified sharkfin test is a reliable test to determine the flow properties of dental impression materials, and it is a good supplement for rheological measurements. In addition, it gives first information about the working time and the comparison with other dental impression materials.
- In this study all tested materials performed acceptable results at all clinically measured times.
- The light materials showed better flow properties than regular materials.
- The new materials on silicon base showed similar characteristics to polyether in regard to clinically measured times, and with this study the new materials have a good chance to find a place in the dental market.

6 Summary

Objectives: The aim of this study was: (1) To determine the relevant processing times for clinical practice. (2) To analyze the flow properties of several elastomeric impression materials depending on these clinically measured times by means of the modified sharkfin test.

Methods and materials: (1) Clinical trial: The processing times of 86 clinical cases were measured by the same person. The impressions were taken by 14 different clinicians with the one-step technique (Impregum-Penta as regular body and Permadyne Garant 2:1 as light body). (2) Laboratory trial: On the basis of the clinical results, the sharkfin test was performed at the times of the 10th and 90th percentiles and the median value at 50 s, 26 s, and 81 s. 11 impression materials were tested: Regular materials (1. Aquasil Ultra Monophase. 2. Aquadyn Medium Soft Quick. 3. Aquadyn Medium Soft regular. 4. Aquadyn Medium Transfer. 5. Impregum Penta. 6. Impregum Penta Soft. 7. Impregum Penta Soft Quick. Light materials (1. Aquasil Ultra LV. 2. Aquadyn Light. 3. Impregum Garant L DuSoft. 4. Permadyne Garant 2:1). Two sizes of molds were used, 2 mm wide at the base for regular materials and 1 mm for light materials. The materials and the receptacle were brought to 23 °C and the entire assembled housing unit was brought to 35 °C to simulate the clinical conditions. Five experiments were made with each material and each of the three clinically measured times.

Results and discussion: The experiments produced sharkfins and yielded flowcurves. The heights of the sharkfins were measured, and from the flowcurve data difference curves were made, in order to calculate the equilibrium points. Mean values, mean flow curves, and statistical analysis of all results at each clinically measured time were made. The longest clinically measured time (81 s) was regarded the most important one to characterize the flow properties. The results of every material at each time were compared to each other. Therefore, all the tested materials at all clinically measured times performed acceptable sharkfins. However, a very important point was significantly noticed, that Aquasil light and regular body gave very poor results compared to the other materials. At 81 seconds Aquasil Ultra Monophase was barely sufficient).

The light materials showed better flow properties than regular materials.

The materials of polyether base show such a high ability of flowing independent of the temperature of the entire assembled unit which was adapted to the mouth temperature.

The impregum-P-S-Q showed superior flow property, in addition to a reasonable sharkfin. According to the manufacturer's recommended working time (60 seconds), Impregum-P-S-Q was not supposed to give good results at 81 seconds.

This study showed that the new materials on silicon base have a similar behavior like polyether concerning the flow properties.

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8 Appendix

Tab A: Clinical datas

clinician	jaw	number of abutment teeth	number of implants	number of abutments	t1	t2	t3	t4	t5	t6	t7	t1+12
1	upper	4	0	4	17	24	4	45	28	312	357	41
1	upper	4	0	4	16	24	3	43	27	306	349	40
2	upper	1	0	1	9	33	4	46	37	210	256	42
1	upper	3	0	3	28	69	3	100	72	232	332	97
1	upper	3	0	3	19	56	3	78	59	260	338	75
3	upper	5	0	5	-5	32	3	30	35	272	302	32
3	upper	5	0	5	-4	37	4	37	41	283	320	37
4	lower	8	0	8	4	51	4	59	55	269	328	55
5	lower	1	0	1	13	38	2	53	40	218	271	51
6	lower	0	4	4	16	30	3	49	33	271	320	46
7	upper	3	0	3	17	32	3	52	35	248	300	49
1	lower	2	0	2	16	42	3	61	45	289	350	58
1	lower	2	0	2	16	40	3	59	43	311	370	56
8	upper	5	0	5	4	28	4	36	32	260	296	32
8	lower	5	0	5	7	37	4	48	41	267	315	44
9	lower	2	0	2	15	40	3	58	43	275	333	55
9	lower	2	0	2	18	41	4	63	45	240	303	59
3	upper	6	0	6	0	39	3	42	42	310	352	39
3	upper	6	0	6	-5	35	4	34	39	318	352	35
6	lower	4	0	4	18	37	3	58	40	227	285	55
6	lower	2	0	2	24	54	3	81	57	216	297	78
6	lower	2	0	2	0	27	3	30	30	256	286	27
9	upper	3	0	3	14	39	4	57	43	258	315	53
9	upper	3	0	3	17	43	3	63	46	262	325	60
1	lower	6	0	6	30	73	3	106	76	264	370	103
1	lower	6	0	6	19	77	3	99	80	281	380	96
6	lower	5	0	5	5	28	4	37	32	244	281	33
6	lower	5	0	5	10	30	3	43	33	248	291	40
3	lower	4	0	4	-4	45	5	46	50	292	338	45
3	lower	4	0	4	-6	41	4	39	45	288	327	41
8	upper	3	0	3	5	23	3	31	26	269	300	28

clinician	jaw	number of abutment teeth	number of implants	number of abutments	t1	t2	t3	t4	t5	t6	t7	t1+12
8	lower	5	0	5	2	25	4	31	29	265	296	27
8	lower	5	0	5	0	15	4	19	19	280	299	15
10	lower	4	0	4	-8	60	4	56	64	334	390	60
10	lower	4	0	4	-7	55	4	52	59	308	360	55
10	upper	3	0	3	-5	53	3	51	56	256	307	53
10	upper	3	0	3	-5	50	3	48	53	253	301	50
1	upper	6	0	6	14	68	4	86	72	284	370	82
1	upper	6	0	6	8	66	3	77	69	336	413	74
6	lower	0	4	4	22	36	4	62	40	274	336	58
11	lower	2	0	2	2	13	3	18	16	282	300	15
11	lower	2	0	2	-2	10	3	11	13	279	290	10
11	upper	2	0	2	3	17	3	23	20	285	308	20
11	upper	2	0	2	0	15	4	19	19	281	300	15
6	lower	0	4	4	2	19	4	25	23	337	362	21
12	upper	6	0	6	-12	37	3	28	40	298	326	37
12	upper	6	0	6	-10	23	4	17	27	291	308	23
6	lower	3	2	5	25	74	3	102	77	246	348	99
6	lower	3	2	5	4	51	3	58	54	232	290	55
2	upper	6	0	6	-4	50	4	50	54	282	332	50
2	upper	6	0	6	-6	42	3	39	45	274	313	42
6	lower	0	4	4	11	23	3	37	26	288	325	34
6	upper	0	3	3	5	24	4	33	28	360	393	29
6	lower	4	0	4	7	47	3	57	50	233	290	54
6	lower	4	0	4	17	63	3	83	66	217	300	80
13	lower	7	0	7	-14	40	3	29	43	292	321	40
8	upper	4	0	4	-55	90	4	39	94	315	354	90
8	upper	4	0	4	0	78	3	81	81	219	300	78
6	lower	2	0	2	15	22	3	40	25	222	262	37
10	upper	3	0	3	8	74	4	86	78	284	370	82
1	upper	3	0	3	20	55	4	79	59	322	401	75
1	upper	3	0	3	13	44	5	62	49	312	374	57
12	lower	9	0	9	-6	45	5	44	50	280	324	45
5	lower	0	3	3	2	53	3	58	56	212	270	55
5	lower	0	3	3	0	40	3	43	43	215	258	40
5	upper	0	8	8	5	62	4	71	66	239	310	67
13	lower	2	0	2	-5	40	3	38	43	262	300	40
13	lower	2	0	2	-18	45	4	31	49	269	300	45

clinician	jaw	number of abutment teeth	number of implants	number of abutments	t1	t2	t3	t4	t5	t6	t7	t1+12
4	lower	0	2	2	13	40	3	56	43	277	333	53
4	upper	0	1	1	12	40	3	55	43	245	300	52
14	lower	0	1	1	20	47	3	70	50	230	300	67
8	lower	4	0	4	-10	49	4	43	53	263	306	49
1	lower	2	0	2	18	44	3	65	47	275	340	62
1	lower	2	0	2	20	40	3	63	43	273	336	60
6	lower	4	0	4	20	36	3	59	39	221	280	56
4	upper	0	1	1	19	34	5	58	39	242	300	53
4	lower	0	1	1	9	28	5	42	33	266	308	37
13	upper	1	0	1	0	45	4	49	49	344	393	45
6	lower	0	1	1	5	32	2	39	34	280	319	37
6	lower	1	0	1	9	38	2	49	40	232	281	47
4	lower	0	2	2	19	47	5	71	52	280	351	66
5	lower	2	0	2	5	25	3	33	28	265	298	30
5	lower	2	0	2	0	22	3	25	25	265	290	22
9	upper	10	0	10	-45	93	4	52	97	305	357	93
6	lower	1	0	1	13	40	2	55	42	300	355	53
6	lower	1	0	1	9	40	3	52	43	242	294	49

Curriculum Vitae

Personal Data:

Name: Odie Saker
 Date of Birth: 15.06.1967
 Local of Birth: Lattakia, Syria
 Marital Status: Married

School Education:

1973 - 1979: Primary school at Damascus - Syria
 1979 - 1982: Secondary school at Damascus - Syria
 1982 - 1985: Grammar school at Damascus - Syria
 Finish: Gen.Certificate of eligibility for university entrance

University Education:

1985 - 1986: Medical school at Damascus
 1986 - 1992: Study of Dentistry at the Dental school of Damascus university
 September 1992: graduation : Doktor of Dentistry

Career

1993 - 1994: Dentist in the Mazza hospital-Maxillofacial surgery-Department
 1995 - 07.2005 Mornings: Dentist in the interior ministry hospital
 Afternoons: Dentist in my own practice
 Since 08.2005 Department of Prosthodontic, University Hospital
 Tuebingen, Germany
 (scholarship from the interior ministry of Syria)