ORIGINAL RESEARCH

DOI: 10.62838/ASMJ.2024.2.03

Contact angle comparison of two elastomeric impression materials during initial setting.

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Abstract

Introduction: Hydrophilicity of elastomeric impression materials is a key property in producing an accurate impression in prosthodontic treatment.

Aim: To study the hydrophilicity (wettability) of two different elastomeric impression materials in vitro by comparing the initial water contact angles on five materials during setting.

Materials and methods: Vinyl polysiloxane VPS (Imprint 4 Light and Super Quick Heavy) and Vinyl polyether silicone VPES (EXA'lence Light Regular, Light Fast and Heavy Fast set) were used. The leveled material surface received 1-µl droplet of deionized water 15 seconds after dispensing the impression material. Contact angles were measured at 0, 30, 60, 90, 120, and 150 seconds using a drop shape analysis machine. The data were analyzed using repeated measures ANOVA with time as a within-subjects factor and material as a between-subjects factor (α=0.05).

Results: All material had high contact angles at time 0 (T1). Imprint 4 had lower contact angles than EXA'lence at all times. There was a statistically significant difference in contact angles of all EXA'lence materials compared to Imprint 4 when not accounting for time ($P < .001$). When time was factored in, there was no significant difference at T1 only ($P = .08$). There was no significant difference among the three EXA'lence materials ($P > .990$) at all-time points. However, there was a significant difference between Imprint 4 materials with Imprint 4 Super Quick Heavy having lower means than Imprint 4 Light ($P = .001$). This was true for all time points except T1.

Conclusions: Despite the high contact angle values at time 0 (T1), both materials reached a significant hydrophilic level with Imprint 4 having drastically lower mean values.

Keywords: contact angle, elastomeric impression material, initial hydrophilicity, VPES, VPS.

Introduction

Vinyl polysiloxane (VPS) and polyether (PE) impression materials are widely used in restorative dentistry due to their excellent properties such as elastic recovery, accuracy, and dimensional stability [1-7]. Hydrophilicity is another important property of impression materials since traces of saliva or blood may be present during impression making to fabricate fixed and removable prostheses. Therefore, the selected impression material must wet the surfaces to optimize the quality of the impression and working casts. Advancing contact angle measurement is a well-established method used to evaluate the hydrophilicity of materials by placing a water droplet on the material surface to measure its contact angles as a function of elapsed time.

The lower the contact angle measurement the more hydrophilic the material [8,9].

In general, VPS and PE elastomers are not considered truly hydrophilic, as they possess contact angles greater than 45° [10]. Several wettability studies have been done on elastomers in the set as well as unset status [11-17].

VPES (vinyl polyether silicone) is a relatively new type of elastomer. This hybrid material benefits from the excellent stability of VPS and hydrophilicity of PE without surfactant to improve hydrophilicity due to the use of tetrahydrofuran and ethylene oxide groups [18]. An in vitro study compared the hydrophilicity of set samples of Imprint 4 (VPS), (3M-ESPE, St. Paul, Minn., USA) EXA'lence (VPES) (GC America, Alsip, IL, USA) and Impregum soft (PE) (3M-ESPE) [19]. The results showed that the mean contact angles on set Imprint 4 could reach as low as 10.1° ± 0.2° after 60 seconds indicating a super hydrophilic behavior, whereas VPES recorded higher readings (40.7° ± 0.1°), which were close in value and behavior to those of PE.

To continue investigating these two elastomers, the aim of this in vitro study was to compare the initial contact angles of water on unset samples of EXA'lence and Imprint 4 and compare to data on set surfaces reported in the above-mentioned study. The null hypothesis was that since both materials are claimed to be hydrophilic, there would be no significant difference in water contact angles between unset samples of Imprint 4 and EXA'lence.

Materials and methods

Table 1 details the five materials used in the study. The samples were prepared by dispensing a small amount of the auto-mixed material into a stainless-steel ring (Sabri Dental Enterprises Inc., Downers Grove, Ill.) which was leveled with a spatula to create a flat, smooth surface where the water droplet was dispensed.

Table 1. VPS and VPES materials used in the study

Material	Brand	Working time/ total setting time	Lot#	City/Country
Vinyl polysiloxane (3M ESPE)	Imprint 4 Light	2:00 / 4:00	586436	St. Paul, Minn., U.S.A.
	Imprint 4 Super quick Heavy	1:15 / 2:30	592319	St. Paul, Minn., U.S.A.
Vinyl polyether silicone VPES (GC America)	EXA'lence Light Regular	2:00 / 5:00	1406051	Alsip, IL, USA
	EXA'lence Light Fast	1:00 / 2:30	1507061	Alsip, IL, USA
	EXA'lence Heavy Fast	1:00 / 2:30	1504151	Alsip, IL, USA

A drop shape analysis (DSA) machine (VCA Optima, AST products, Inc. Billerica, Mass.) was used to measure the advancing contact angles, which started 15 seconds after dispensing the material into the ring, T0. The ring was placed on the machine platform right below the needle from which a 1-μL water droplet was dispensed from the 100-μL pipette onto the material surface and contact angles were measured in degrees using the software included with the machine. A video recording was made showing the water droplet activity on the surface so that contact angles could be measured at specific intervals. Once captured, the recording was stopped every 30s to make measurements using 5 markers (L, R, 1, 2 and T) around the droplet (Figure 1) using the manufacturer's software which automatically measured the contact angles.

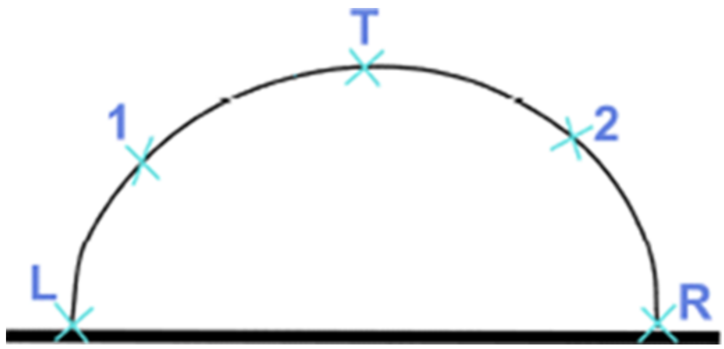

Figure 1. Location of the five measurements made on each water droplet during setting of the material. Source: VCA Optima manual.

Twenty samples of each material were made resulting in 100 samples. The experiments were conducted at room temperature (23˚±1˚) and relative humidity of 55±5%. Each water droplet had measurements made at 30- second intervals: Time 0 (T1), 30 (T2), 60 (T3), 90 (T4), 120 (T5), and 150 (T6) seconds (Figure 2A and 2B).

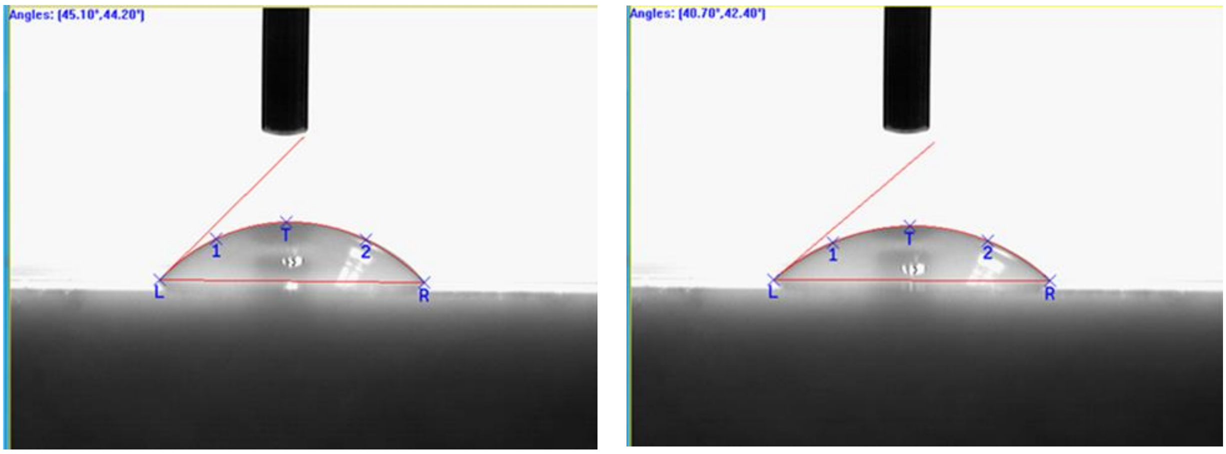

Figure 2. A - A representative image of a water droplet on the surface of VPES light body impression material. B - A representative image of a water droplet on the surface of VPS Light body impression material.

Statistical analysis was conducted using IBM SPSS Statistics version 29.0.2.0 (IBM Corp.©). The distribution of the recorded values—distribution of contact angles was approximately normal. Statistical significance level was set at Type I error rate, α=0.05.

Results

Figures 3 and 4 show the average contact angle of water on the surface of the materials during setting. The mean values of Imprint 4 were lower than those of EXA'lence at all measurement times ranging from 86.1° ± 3.8° at T1 to 17.3° ± 5.5° at T6 vs. 93.1° ± 8.3° at T1 to 34.3° ± 7.0° at T6 for EXA'lence (Table 2).

Table 2. Pairwise comparisons of mean contact angles of water on unset surfaces of the three EXA'lence and two Imprint 4 materials used at all test times. The last row indicates overall values over the entire 150s.

Time (seconds)	Mean Contact Angle for VPES (°)	Mean Contact Angle for VPS (°)	Mean Difference (°)	*P* value	95% CI
0	93.1 ±8.3	86.1 ±3.8	7.0	.001	(2.9, 11.1)
30	50.2 ±7.2	30.0 ±6.6	20.2	< .001	(16.1, 24.3)
60	45.7 ±7.0	26.5 ±5.3	19.2	< .001	(15.4, 23.0)
90	41.7 ±6.5	22.8 ±4.7	18.9	< .001	(15.4, 22.3)
120	37.9 ±6.8	19.9 ±5.0	18.0	< .001	(14.3, 21.6)
150	34.3 ±7.0	17.3 ±5.5	17.0	< .001	(13.2, 20.8)
Overall	50.5 ±5.3	33.8 ±4.7	16.7	< .001	(13.7, 19.7)

VPES = vinyl polyether silicone, VPS = vinyl polysiloxane, CI = confidence interval. P values are based on Bonferroni correction.

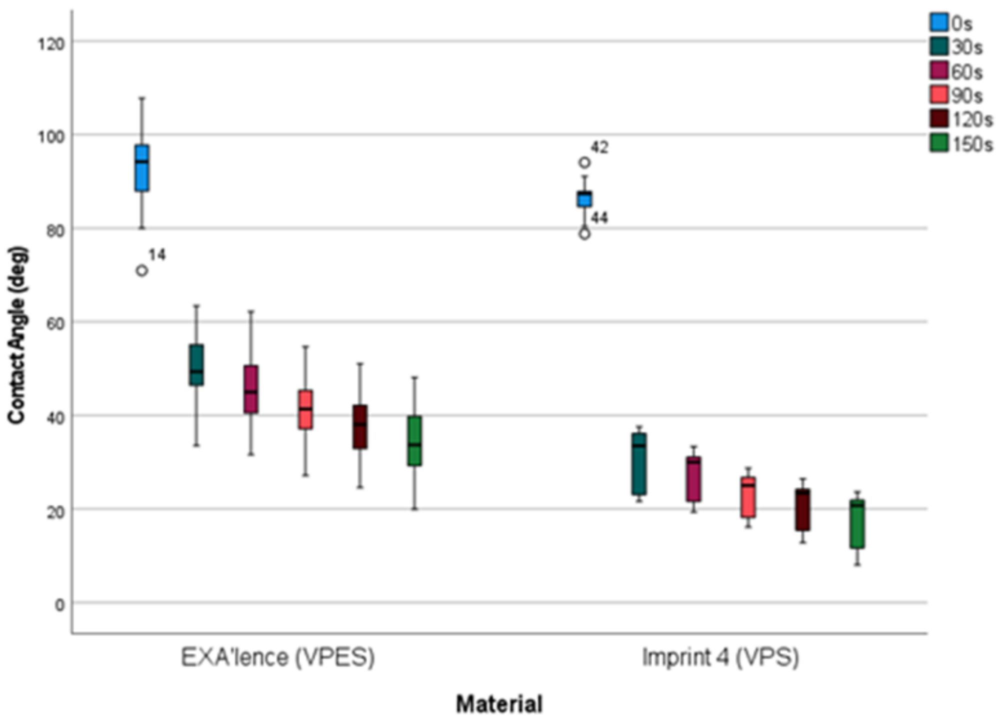

Figure 3. Box and whisker plot of contact angles of water droplets on unset surfaces of EXA'lence and Imprint 4 samples measured at 30s intervals, from 0s to 150s. The lower whisker indicates the minimum value of the set, the lower bound of the box is the first quartile, the horizontal line is the median value, the upper bound of the box is the third quartile, and the top whisker indicates the maximum value of the set. Circles indicate potential outliers.

All materials exhibited similar patterns with highest contact angles at T1 (0s) that dropped sharply after the first 30s and continued to decrease gradually (Figure 4). Considering the average values over the entire 150s, Imprint 4 Super Quick Heavy had the lowest overall mean measurements at 29.1° ± 1.7°, while EXA'lence Heavy Fast had the highest mean at 51.7° ± 4.3°.

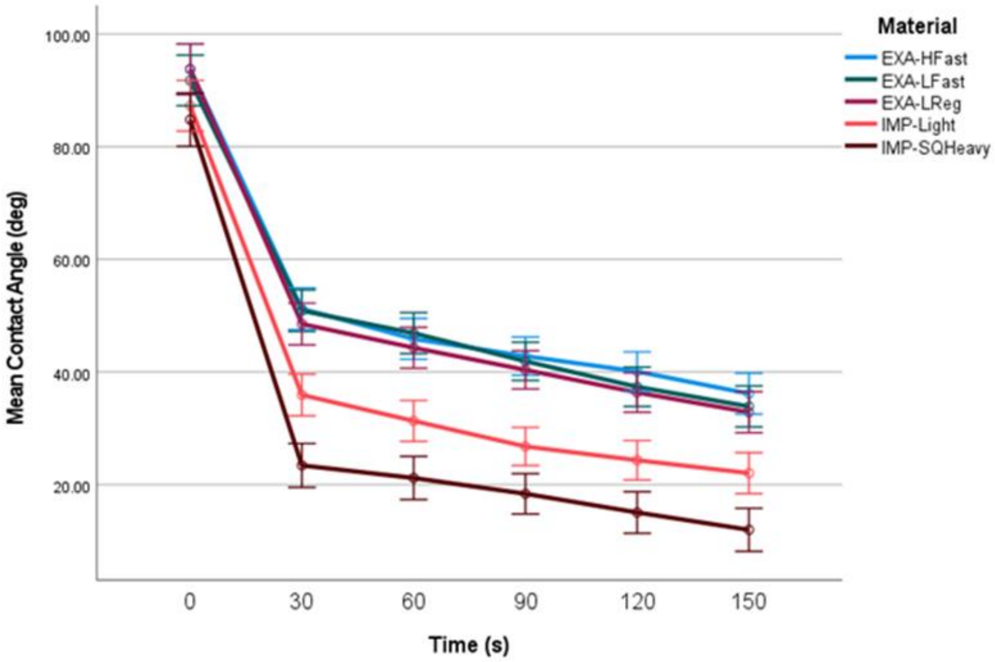

Figure 4. Estimated marginal means of the contact angles of water droplets on unset surfaces of EXA'lence and Imprint 4 samples. Error bars represent 95% confidence intervals.

We applied repeated measures ANOVA with time as a within-subjects factor and material as a between-subjects factor. P values indicate that both factors and the interaction between the two factors were significant. For follow-up analysis, we compared the mean contact angles between the different materials overall as well as between the different materials at each fixed time. For multiple pairwise comparisons, we adjusted the type I error rate by Bonferroni correction (Tables 2, 3 and 4).

Table 3. Pairwise comparisons of the mean difference in contact angles of water on unset surfaces between EXA'lence and Imprint 4 samples over the entire 150s. The breakdown at each specific time point is detailed in Table 4.

Table 3.

Material Comparison	Mean Difference (°)	*P* value	95% CI
EXA HF vs. EXA LF	1.2	> .990	(-4.5, 6.9)
EXA HF vs. EXA LR	2.3	> .990	(-3.4, 8.0)
EXA HF vs. IMP 4 L	13.7	< .001	(8.0, 19.4)
EXA HF vs. IMP 4 SQH	22.5	< .001	(16.6, 28.4)
EXA LF vs. EXA LR	1.1	> .990	(-4.7, 6.8)
EXA LF vs. IMP 4 L	12.5	< .001	(6.8, 18.2)
EXA LF vs. IMP 4 SQH	21.3	< .001	(15.4, 27.2)
EXA LR vs. IMP 4 L	11.4	< .001	(5.7, 17.2)
EXA LR vs. IMP 4 SQH	20.2	< .001	(14.3, 26.1)
IMP 4 L vs. IMP 4 SQH	8.8	.001	(2.9, 14.7)

EXA = EXA'lence, IMP 4 = Imprint 4, HF = Heavy Fast, LF = Light Fast, LR = Light Regular, L = Light, SQH = Super Quick Heavy, CI = confidence interval. P values are based on Bonferroni correction.

There was a statistically significant difference in mean contact angles of all EXA'lence samples compared to Imprint 4 when not accounting for time (Table 2). The mean contact angle of Imprint 4 Super Quick Heavy over 150s was 22.5° lower than that of EXA'lence Heavy Fast (95% CI [16.6, 28.4], P < .001). Imprint 4 Light exhibited a mean that was 12.5° and 11.4° lower than EXA'lence Light Fast and Light Regular over 150s, respectively (95% CI [6.8, 18.2] and [5.7, 17.2], P < .001) (Table 3).

When time was factored in, there was a significant difference in mean contact angles between the three EXA'lence and the two Imprint 4 materials at all time points except T1 (Figure 4). For example, at 0s, the mean contact angle of Imprint 4 Super Quick Heavy was 9.0° less than that of EXA'lence Heavy Fast (95% CI [-0.60, 18.6], P = .08) while at 150s, this difference increased to 24.2° (95% CI [16.4, 31.9], P < .001). Similarly, there was no significant difference between Imprint 4 Light and EXA'lence Light Fast at 0s while the difference was statistically significant at 150s, with the mean contact angle of Imprint 4 Light being 11.8° less than that of EXA'lence Light Fast (95% CI [4.3, 19.4], P < .001) (Table 4). Lastly, there was no significant difference between the three EXA'lence materials (P > .990) at all-time points. However, a significant difference existed between the two Imprint 4 materials at all-time points except T1, with the overall mean contact angle of Imprint 4 Super Quick Heavy being 8.8° less than Imprint 4 Light over 150s (95% CI [2.9, 14.7], P = .001) (Table 3).

Table 4. Pairwise comparison of the mean difference in contact angles of water on unset surfaces between the EXA'lence and Imprint 4 samples at all test times.

Time (seconds)	Material Comparison	Mean Difference (°)	*P* value	95% CI
0	EXA HF vs. EXA LF	2.0	> .990	(-7.3, 11.3)
	EXA HF vs. EXA LR	.02	> .990	(-9.3, 9.3)
	EXA HF vs. IMP 4 L	6.5	.454	(-2.8, 15.8)
	EXA HF vs. IMP 4 SQH	9.0	.082	(-.6, 18.6)
	EXA LF vs. EXA LR	-2.0	> .990	(-11.3, 7.3)
	EXA LF vs. IMP 4 L	4.5	> .990	(-4.8, 13.8)
	EXA LF vs. IMP 4 SQH	7.0	.370	(-2.6, 16.5)
	EXA LR vs. IMP 4 L	6.5	.459	(-2.8, 15.8)
	EXA LR vs. IMP 4 SQH	9.0	.083	(-.6, 18.5)
	IMP 4 L vs. IMP 4 SQH	2.5	> .990	(-7.1, 12.1)
30	EXA HF vs. EXA LF	.3	> .990	(-7.3, 8.0)
	EXA HF vs. EXA LR	2.7	> .990	(-5.0, 10.4)
	EXA HF vs. IMP 4 L	15.3	< .001	(7.6, 22.9)
	EXA HF vs. IMP 4 SQH	27.8	< .001	(19.9, 35.7)
	EXA LF vs. EXA LR	2.4	> .990	(-5.3, 10.0)
	EXA LF vs. IMP 4 L	15.0	< .001	(7.3, 22.6)
	EXA LF vs. IMP 4 SQH	27.5	< .001	(19.6, 35.3)
	EXA LR vs. IMP 4 L	12.6	< .001	(4.9, 20.3)
	EXA LR vs. IMP 4 SQH	25.1	< .001	(17.2, 33.0)
	IMP 4 L vs. IMP 4 SQH	12.5	< .001	(4.6, 20.4)

60	EXA HF vs. EXA LF	-1.0	> .990	(-8.5, 6.5)
	EXA HF vs. EXA LR	1.6	> .990	(-6.0, 9.1)
	EXA HF vs. IMP 4 L	14.6	< .001	(7.0, 22.1)
	EXA HF vs. IMP 4 SQH	24.7	< .001	(17.0, 32.4)
	EXA LF vs. EXA LR	2.6	> .990	(-4.9, 10.1)
	EXA LF vs. IMP 4 L	15.6	< .001	(8.0, 23.1)
	EXA LF vs. IMP 4 SQH	25.7	< .001	(18.0, 33.4)
	EXA LR vs. IMP 4 L	13.0	< .001	(5.5, 20.5)
	EXA LR vs. IMP 4 SQH	23.1	< .001	(15.4, 30.9)
	IMP 4 L vs. IMP 4 SQH	10.1	.004	(2.4, 17.9)
90	EXA HF vs. EXA LF	.9	> .990	(-6.1, 7.9)
	EXA HF vs. EXA LR	2.4	> .990	(-4.6, 9.5)
	EXA HF vs. IMP 4 L	16.0	< .001	(9.0, 23.0)
	EXA HF vs. IMP 4 SQH	24.4	< .001	(17.2, 31.6)
	EXA LF vs. EXA LR	1.5	> .990	(-5.5, 8.6)
	EXA LF vs. IMP 4 L	15.1	< .001	(8.1, 22.1)
	EXA LF vs. IMP 4 SQH	23.5	< .001	(16.3, 30.7)
	EXA LR vs. IMP 4 L	13.6	< .001	(6.6, 20.6)
	EXA LR vs. IMP 4 SQH	22.0	< .001	(14.8, 29.2)
	IMP 4 L vs. IMP 4 SQH	8.4	.013	(1.2, 15.6)
120	EXA HF vs. EXA LF	2.7	> .990	(-4.5, 10.0)
	EXA HF vs. EXA LR	3.7	> .990	(-3.5, 11.0)
	EXA HF vs. IMP 4 L	15.7	< .001	(8.5, 23.0)
	EXA HF vs. IMP 4 SQH	25.0	< .001	(17.6, 32.5)
	EXA LF vs. EXA LR	1.0	> .990	(-6.2, 8.3)
	EXA LF vs. IMP 4 L	13.0	< .001	(5.8, 20.3)
	EXA LF vs. IMP 4 SQH	22.3	< .001	(14.9, 29.7)
	EXA LR vs. IMP 4 L	12.0	< .001	(4.8, 19.3)
	EXA LR vs. IMP 4 SQH	21.3	< .001	(13.9, 28.7)
	IMP 4 L vs. IMP 4 SQH	9.3	.006	(1.8, 16.7)
150	EXA HF vs. EXA LF	2.3	> .990	(-5.3, 9.8)
	EXA HF vs. EXA LR	3.3	> .990	(-4.2, 10.8)
	EXA HF vs. IMP 4 L	14.1	< .001	(6.6, 21.6)
	EXA HF vs. IMP 4 SQH	24.2	< .001	(16.4, 31.9)
	EXA LF vs. EXA LR	1.0	> .990	(-6.5. 8.6)
	EXA LF vs. IMP 4 L	11.8	< .001	(4.3, 19.4)
	EXA LF vs. IMP 4 SQH	21.9	< .001	(14.2, 29.6)
	EXA LR vs. IMP 4 L	10.8	.001	(3.3, 18.3)

EXA LR vs. IMP 4 SQH	20.9	< .001	(13.1, 28.6)
IMP 4 L vs. IMP 4 SQH	10.1	.004	(2.3, 17.8)

EXA = EXA'lence, IMP 4 = Imprint 4, HF = Heavy Fast, LF = Light Fast, LR = Light Regular, L = Light, SQH = Super Quick Heavy, CI = confidence interval. P values are based on Bonferroni correction.

Discussion

The null hypothesis that there would be no significant difference in water contact angles between unset Imprint 4 and EXA'lence was rejected when not accounting for time. When time was factored in, the null hypothesis was rejected except for at T1 (0s).

Regarding the initial contact angles, both elastomers exhibited high values at T1 (15 seconds from dispensing) despite their different composition. However, they underwent a considerable drop in values from T1 to T2 (45 seconds from dispensing) and continued a gradual decrease from T2 to T6.

The high initial values of EXA'lence indicate a similar behavior to that of VPS, and not polyether which is incorporated in this elastomer according to the manufacturer. Studies have shown that initial contact angles for polyether were much lower than the 93.1 ± 8.3 measured in this study. Polyether values were more hydrophilic, ranging from 58°- 70°.8 [11, 15, 19]. However, polyether may lack distinct hydrophilization [11], which means that its hydrophilicity did not drastically increase with time. For EXA'lence to resemble Imprint 4 while trying to reach equilibrium could mean that its VPS component was dominant over its PE one despite containing no surfactant [18].

Following the sharp drop in contact angles at T2, the five materials, which have different consistencies and setting times (Table 1), continued to record a gradual decrease until they reached low values (Figure 3). During hydrophilicity development, materials could act differently where one could reach it due to containing surfactants while others could achieve it by having an unleachable modifier at the surface [15]. Considering this aspect of material behavior, Kugel et al. reported that VPS surfactants have to migrate to the surface [8], which may explain the delayed initial contact angle values. In this regard, samples of unset Imprint 4 were shown to behave differently from set ones [19]. In that study, set Imprint 4 showed initial contact angles where only the two heavy body consistencies recorded high initial readings while regular and light body had mean values below 30° at T1. For unset Imprint 4 in this investigation, both Imprint 4 heavy (working time 1:15 minutes) and light body (working time 2:00 minutes) (Table 1) had high initial values, which could be due to the time surfactants need to reach the surface in the unset samples.

Similar behavior has been reported for different brands of VPS [11,15,17] where the initial contact angle values were high before dropping to values that were lower than the final ones reached by the tested polyether.

It has been reported that Imprint 4 contains a novel surfactant, a modified polyalkylene oxide, which contains hydrophilic regions [18].

Whether the surfactant migrates to the surface to increase its wettability or leaches out to decrease water surface tension, the mean contact angles with water indicate excellent wettability when considering the working time of the material (Table 1). The mean contact angle of Imprint 4 in our study was 22.8° ± 4.7° at T3 (60 seconds) which is well within the working time the clinician can use to dispense the light body at crown margin/implant component and the heavy body into the custom tray. In this regard, Balkenhol et al. studied surfactant release from a hydrophilized VPS material [14]. The study reported that the higher the concentration of leached non-ionic surfactant, the lower the contact angles. The surfactant incorporated into the hydrophilized VPS formulation was released from its surface and diffused into the liquid in contact. Another study examined the influence of non-ionic surfactants on hydrophilicity. Lee et al. reported that the higher the concentration of surfactants the lower the contact angles too [20]. However, the study indicated that hydrophilicity of VPS is determined by surfactant concentration at the surface, which is the spatial distribution of surfactants in the outermost region of the material. It is also interesting to note that concentration of the surfactant was not the only factor impacting contact angle because the smaller chain, less hydrophilic surfactant homolog recorded the lowest values.

A study by Menees et al. confirmed the influence of surfactants on hydrophilicity of hybrid elastomers by comparing EXA'lence, which contains no surfactants, to Identium that contains two surfactants, a surface tension eraser, and a wetting conditioner [18]. While EXA'lence recorded high contact angles, Identium recorded the lowest ones, which may be due to the presence of these surfactants as well as the grafted polyether groups.

Surfactants were shown to sustain wettability improvement after 6 months of storage possibly due to the retention of the surfactant matrix by means of physio-chemical bonding [21], which may explain the low initial contact angle means recorded in set Imprint 4 [19].

Not all available consistencies of Imprint 4 and EXA'lence were reported in this study. Expanding the investigation to include the remaining consistencies may shed more light on the behavior of these materials. Furthermore, it would be insightful to study the behavior of these materials after subjecting them to different types and protocols of disinfection procedures.

Conclusions

- Despite their different composition, Imprint 4 and EXA'lence recorded high initial contact angles. However, Imprint 4 materials had lower contact angle means at all measurement times than did EXA'lence.
- Both materials underwent a similar gradual decrease in contact angles towards equilibrium.

Conflict of Interest: The authors declare no conflict of interest.

Financial disclosure and acknowledgement: This study was supported by the University of Alberta Fund for Dentistry grant DEN 18.

Ethics Committee Approval: No need.

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Received: May 15, 2024 / Accepted: October 17, 2024