Basic Science Research

Investigation of a New Approach to Measuring Contact Angles for Hydrophilic Impression Materials

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Purpose: The purpose of this investigation was to examine the initial water contact angles of seven unset impression materials using commercially available equipment, in an effort to determine whether polyether impression materials (Impregum) have lower contact angles and are, therefore, more hydrophilic than VPS impression materials.

Materials and Methods: The hydrophilic properties of unset polyether and VPS impression materials were analyzed with respect to their water contact angle measurements using the commercially available Drop Shape Analysis System DSA 10. Twenty-five data points per second were collected via video analysis. There was no delay from start of measurement and data collection. Data was collected for approximately 12 s. Droplet size was determined on the thickness of canula. If the droplets became too small in volume, the water that evaporated during the measurement was large in comparison to the volume of the droplet. Therefore, 5 μl was chosen as the lowest volume. Five trials were conducted per series for each featured material.

Contact angles were calculated using the circle fitting method. Three tests using this technique were designed to control the variables of contact angle measurement with regard to time, the varying amount of fluid in contact with impression material during clinical use, and material thickness. Sample thickness of impression material was controlled by stripping the paste flat on a glass plate using a marking template to ensure a constant film thickness. Tests were conducted in a climatized room at 24°C ± 1°C. Deionized water was used as the fluid. The device was calibrated according to manufacturer's instruction for Young–Laplace fitting prior to the measurements. Results were analyzed using One-Way ANOVA, Tukey test, and *t*-test, as appropriate.

Results: Comparing the fast setting impression materials by One-Way ANOVA and Tukey tests ($p < .05$) revealed the initial contact angles to range from 66.2 ± 1.5° to 127.5 ± 4.4°, of which the polyether material was the lowest after 45 s (66.2 ± 1.5°), 120 s (70.3 ± 2.8°), and 24 h (80.3 ± 1.0°) after start of the mix. The selected times represent the different stages of unset material, ranging from 45 s as the earliest practical data collection time to 24 h, at which a stone model would be poured. The polyether materials tested exhibited lower contact angles and, thus, significantly higher initial hydrophilicity than all measured VPS materials. Additionally, Impregum impression materials are more hydrophilic in the unset stage than in the set stage. VPS may show a stepwise development of hydrophilicity in the set stage that was not observed in the unset stage.

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Conclusions: **The polyether impression materials tested were significantly more hydrophilic before, during, and after setting than that of VPS impression materials. Regardless of the amount of water in contact with the impression material, the polyether impression materials showed a significantly higher hydrophilicity in the unset stage than the VPS materials. The initial contact angle was not dependent on the thickness of the material. All parameters, including variation of time, volume of water droplet, and thickness of material, resulted in different absolute contact angles, but did not lead to a dramatic change in the ranking of the materials with regard to their hydrophilic behavior.**
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INDEX WORDS: hydrophilicity, flow properties, impression materials, contact angle measurement

CONTEMPORARY IMPRESSION materials have a number of properties that contributeto clinical success.[1-4] Hydrophilicity is critically important during working time, when the material flows and is in contact with moisture on the preparation and in the oral cavity. For this reason, most of the state of the art impression materials claim to be hydrophilic. For an impression material to work, it should wet soft and hard tissues that might be covered with saliva. This saliva can be represented principally by water, and therefore this is the appropriate medium to test in combination. Although initial hydrophilicity is essential to create accurate impressions, the hydrophilicity of impression materials in the set stage also plays a key role. At this point, hydrophilicity is an important parameter in producing accurate casts in the dental laboratory.[5-7]

Water contact angles are most frequently used to determine the hydrophilic properties of impression materials *after* they have set;[8-10] however, it is most clinically relevant to determine the water contact angles in the unset state. Very limited information is available regarding the hydrophilicity of impression materials during the unset stage, since it is difficult to measure on plastic surfaces.

As there was previously no common method to determine the hydrophilic behavior of unset impression materials, a goal of the present study was to develop such a technique using commercially available equipment. This procedure was recently employed to conduct a series of contact angle studies on unset impression materials. The objective of this investigation was to examine the initial water contact angles of seven unset impression materials using commercially available equipment, in an effort to determine whether polyether impression materials (Impregum) have lower contact angles and are, therefore, more hydrophilic than VPS impression materials

Materials and Methods

In testing polyether and VPS impression materials, the tests were designed to control the variables of the measurement. Tests were conducted in a climatized room at 24°C ± 1°C. Deionized water was chosen as the fluid for this study, because other than mercury, water offers the highest surface energy and best available drop contour, is an easily available standardized liquid, and offers clinical significance because it is the essential ingredient of saliva. The device was calibrated according to the manufacturer's instruction for Young–Laplace fitting prior to the measurements. The objective of the first stage was to compare the degree of hydrophilicity of light-bodied precision impression materials with regard to time in the unset stage during and after setting. According to thermodynamics, materials can be defined as hydrophilic when contact angles are lower than 90°, and hydrophobic if the contact angles are above 90°.[11,12] The latest impression materials were selected and prepared according to manufacturer guidelines. The second stage examined the varying amount of fluid in contact with impression material during clinical use. A third stage was designed to analyze contact angle as a function of material thickness. Sample thickness was controlled by stripping the paste flat on a glass plate using a marking template to ensure a constant film thickness.

The Drop Shape Analysis System DSA 10 (Krüss GmbH, Hamburg, Germany) was used to analyze the hydrophilic properties of the unset polyether and VPS impression materials through contact angle measurements (Fig 1). A major advantage of this method is that the device is commercially available. Additionally, contact angle measurements produce very straightforward results.[13,14] For precise determination of contact angles, high-resolution analysis is of major importance since the first contact of water with impression material has to be

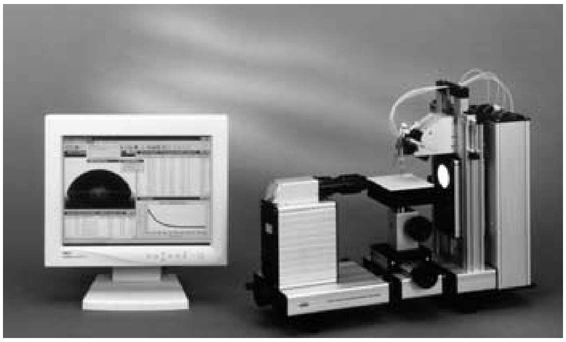

Figure 1. Drop Shape Analysis system DSA 10.

captured. In addition, it was observed that after a period of time, the placed water droplet may alter the surface of the plastic impression material, which may lead to misleading results when not measuring immediately. Therefore, video analysis that allows the collection of 25 data points per second was used. The sequential steps of the contact angle determination are shown in Figure 2.

Test 1: Variation of Time

Timing is essential in the impression-taking process, and it is well known that the hydrophilic behavior of impression materials is different in the set and unset stage. It is important to know that the chosen impression material will offer the best possible hydrophilic performance before, during, and after setting. For example, when multiple preparations are required, using the right impression material can help practitioners avoid potential distortions, pick up the same amount of detail, and maintain consistency. Getting a detailed final impression is imperative. In this stage, the degree of hydrophilicity of light-bodied impression materials was recorded in the unset stage during and after setting.

To investigate the initial hydrophilicity of popular brands of light-body impression materials (ISO Type 3) before, during, and after setting, six fast-setting, light-bodied precision impression materials [Impregum™ Penta™ *Soft* Quick Step Light Body (3M ESPE, Minneapolis, MN, B156147, C1158043), Honigum® Automix Light Fast (Zenith Dental DMG, Englewood, NJ, 523197), Take 1® Fast Set Wash (Kerr, Orange, CA, 3-1282), Aquasil Ultra LV Fast Set (Dentsply, York, PA, 031208), Aquasil Ultra XLV Fast Set (Dentsply, 040112), Affinis™ Light Body Fast (Coltene/Whaledent, Alstätten, Switzerland, MH036)] and one regular setting material [Impregum™ Garant™ *Soft* Light Body (3M ESPE, B169002, C169658)] were compared. All impression materials were prepared according to manufacturer instructions. Five trials were conducted for each featured material.

In this series, the time the water droplet was placed was controlled with regard to the start of mixing the catalyst and base paste together, as well as the time

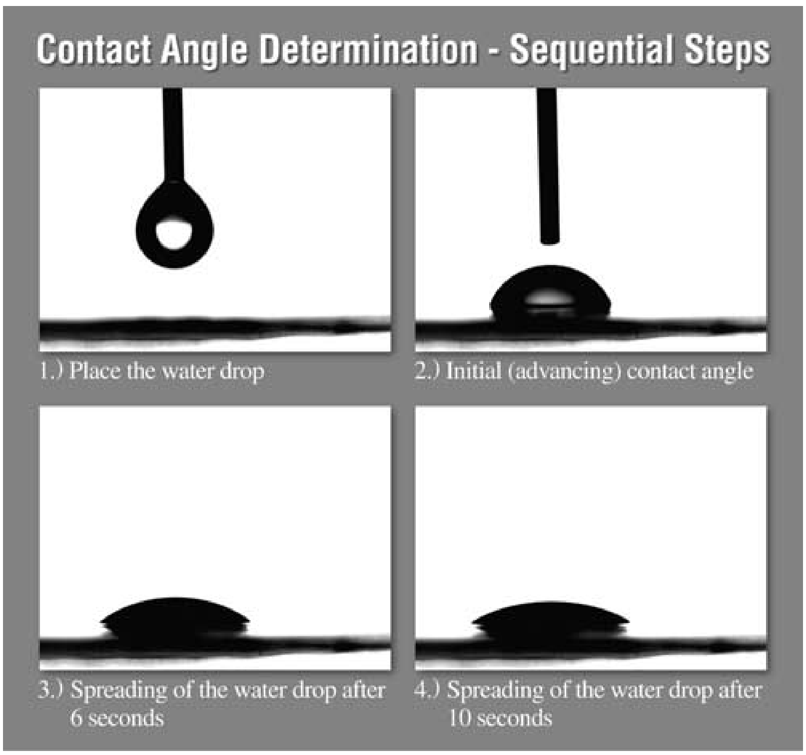

Figure 2. Sequential steps to determine contact angle.

when contact angle was determined with regard to the time of droplet placement. Contact angle measurement was used to determine the hydrophilic properties of the unset impression materials. Water contact angles were then measured at room temperature. The materials were dispensed to yield a layer of 20 μm of unset impression material. At 45 s, 120 s, and 24 h after start of mix, a water drop (5 μl) was placed on each impression material and data collected using the Drop Shape Analysis System DSA 10 and video analysis. Twenty-five data points per second were recorded for a total of 10 s. The high resolution of the measurement allowed us to determine the initial contact angle.

Test 2: Variation of Volume of Water Droplet (Unset Material)

The amount of moisture in contact with an impression material may vary in a clinical setting. Especially with VPS, hydrophilization is gained with nonionic surfactants. These molecules consist of a hydrophilic and a silicone-compatible hydrophobic part. The mode of action is thought to be a diffusion-controlled transfer of surfactant molecules from the VPS into the aqueous phase. In this manner, the surface tension of the surrounding liquid is altered, which reduces its contact angle.[15] Therefore, a second stage investigated the wetting properties of impression material as a function of the amount of liquid, and how this can ultimately impact the quality of an impression.

Four fast-set, light-bodied precision impression materials were compared in this study, including Impregum™ Penta™ *Soft* Quick Step Light Body (3M ESPE, B174999, C173527), Take 1® Fast Set Wash (Kerr, 4-1027), Aquasil Ultra LV Fast Set (Dentsply, 040225), and Aquasil Ultra XLV Fast Set (Dentsply, 040306). Five trials were conducted for each featured material.

Again, contact angle measurement was used to determine hydrophilicity, and the water contact angles were measured at room temperature. The materials were dispensed to yield a layer of 20 μm of unset impression material. Forty-five seconds after start of mix, a water drop (5, 7, 9 μl) was placed on the material and data were collected using the Drop Shape Analysis System DSA 10 and video analysis. Twenty-five data points per second were recorded for a total of 10 s.

Test 3: Variation of the Thickness of Material

Since the migration of surfactants into the water droplet is diffusion controlled,[15] and amount of surfactant increases with increase in the thickness of the material, a third series of tests was conducted to determine whether impression materials become more hydrophilic as material thickness increases (more surfactant "under" the water droplet).

Two polyether and three VPS impression materials were used in this stage, including Impregum™ Garant™ *Soft* Light Body (3M ESPE, B196509, C195967), Impregum™ Penta™ *Soft* Quick Step Light Body (3M ESPE, B194744, B193512), Aquasil Ultra XLV Fast Set (Dentsply, 41110), Aquasil Ultra LV Fast Set (Dentsply, 40907), and Take 1® Fast Set Wash (Kerr, 4-1147). Five trials were conducted for each featured material.

Each impression material was dispensed to produce a layer of unset impression material in five thicknesses using molds (70, 140, 170, 240, 280 μm). Water contact angles were measured at room temperature. A water drop (5 μl) was placed on the material 45 s after start of mix and data were collected using the Drop Shape Analysis System DSA 10 and video analysis. Twenty-five data points per second were recorded for a total of 10 s.

Results

Test 1: Variation of Time

Initial contact angles were determined 45 s, 120 s, and 24 h after start of mix. Five trials were performed per series. A summary of the data as well as standard deviations of the individual runs is given in Figure 3. The initial contact angles of Impregum Penta *Soft* Quick Step Light Body and Impregum Garant *Soft* Light Body were found to be the lowest after 45 s, 120 s, and 24 h. Results were analyzed using One-Way ANOVA and Tukey test ($p < 0.05$). H0: all impression materials have the same hydrophilicity. It was also found that both polyether materials were significantly more hydrophilic after 45 and 120 s than after 24 h. Results were analyzed using two sample t-test; H0: polyether materials are more hydrophobic after 24 h than after 45 s ($p = 1.0$).

In the unset stage (45 s after mix) all investigated materials showed a continuous decrease of the contact angle with time (Fig 4).

In contrast, VPS impression material in the set stage may show a stepwise development of hydrophilicity (Fig 5) whereas polyether materials do not (Fig 6).

Test 2: Variation of Volume of Water Droplet (Unset Material)

Initial contact angles were determined 45 s after start of mix with water droplets having a volume of

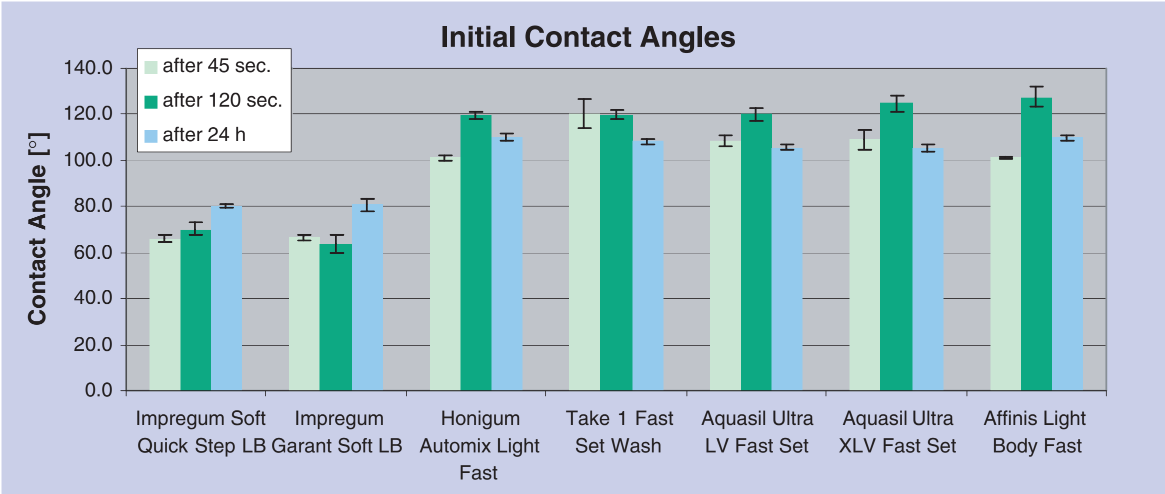

Figure 3. Initial contact angle. Water droplet (5 μl) was placed 45 s, 120 s, and 24 h after start of mix (material thickness 20 μm). Standard deviations are indicated as error bars.

5, 7, and 9 μl. Five trials were performed per series. A summary of the data as well as standard deviations of the individual runs is given in Figure 7. Results were analyzed using One-Way ANOVA and Tukey test ($p < .05$). H0: all impression materials have the same hydrophilicity.

Impregum Penta *Soft* Quick Step Light Body showed a significantly higher hydrophilicity with

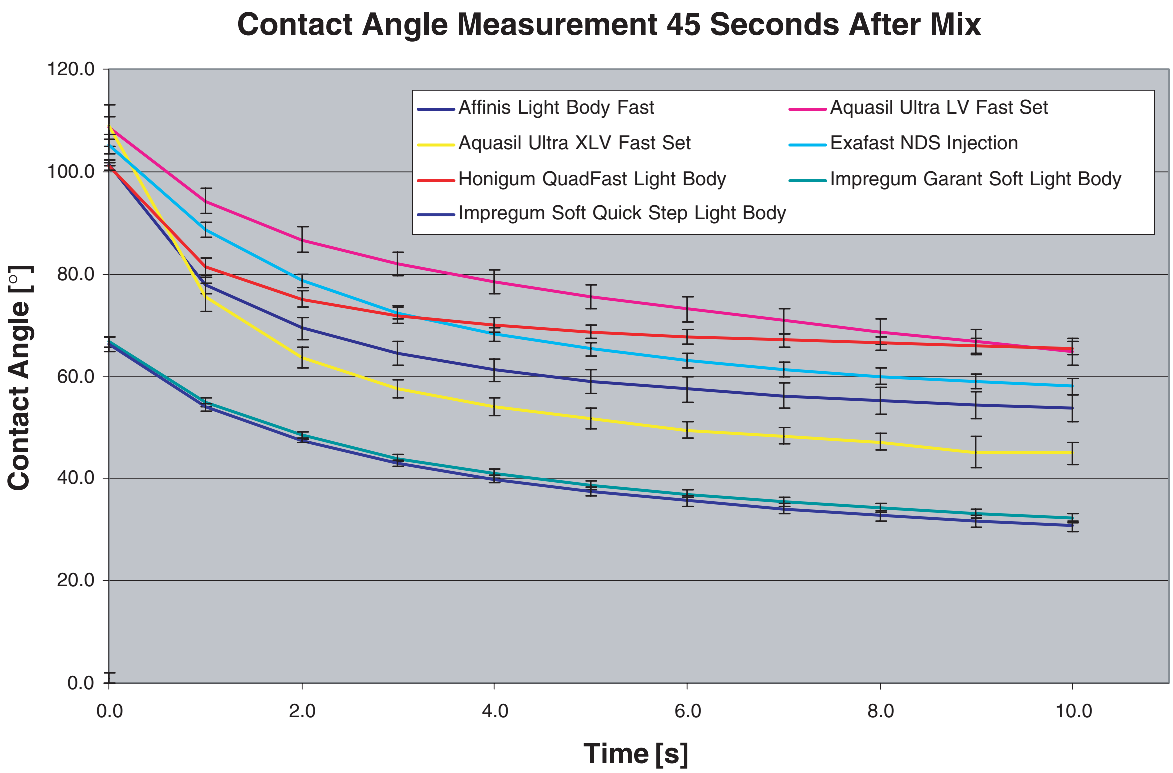

Figure 4. Contact angle measurement. Water droplet (5 μl) was placed 45 s after mix (material thickness 20 μm). Standard deviations are indicated as error bars.

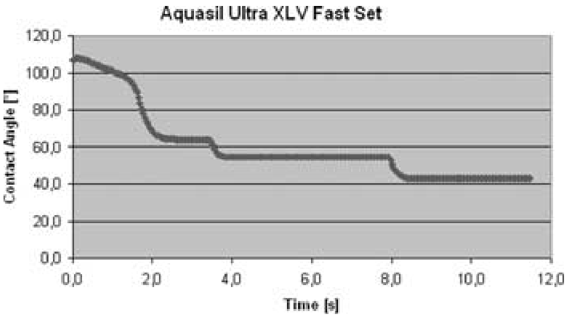

Figure 5. Contact angle measurement for Aquasil Ultra XLV Fast Set. Water droplets (5 μl) were placed 24 h after start of mix (material thickness 20 μm).

a 9 μl drop volume than with a 5 μl drop volume. For the purposes of this study, it is worth noting that while different water droplet sizes may result in statistical differences between contact angles, the statistical differences, as reported, between the polyether and VPS materials are of greater importance. The dependence of a given material from the water droplet size may be explained by its formulation. Leachable hydrophilic components of the formulations like softeners and/or surfactants might contribute to the measured result. In addition, one has to take into account that low droplet volumes potentially give less real results because of water evaporation.

Independent of the water droplet size, the contact angles of Impregum Penta *Soft* Quick Step Light Body were found to be significantly lower than those of the VPS materials—Take 1 Fast Set Wash, Aquasil Ultra LV Fast Set, and Aquasil Ultra XLV Fast Set—for all drop volumes.

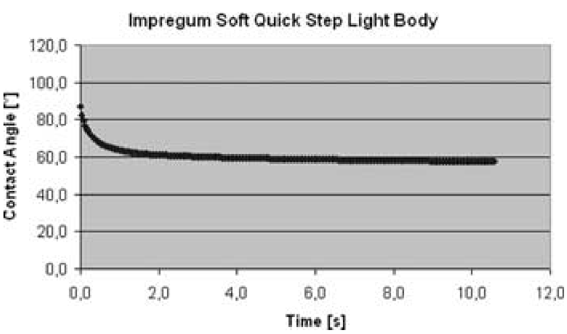

Figure 6. Contact angle measurement for Impregum *Soft* Quick Step Light Body. Water droplets (5 μl) were placed 24 h after start of mix (material thickness 20 μm).

A comparison for the initial contact angles of Impregum Penta *Soft* Quick Step Light Body and Ultra LV Fast Set is shown in Figure 8. The images depict a camera shot through the lens, showing the water droplet interacting with the surface.

Test 3: Variation of the Thickness of Material

Initial contact angles were measured 45 s after start of mix. A water droplet was placed on the surface of each impression material at film thicknesses of 70, 140, 170, 240, and 280 μm. Five trials were performed per series. Means and standard deviations are summarized in Figure 9.

Independent of the material film thicknesses, all measured values for one material were similar—initial contact angles of polyether impression materials were found to be lower than those of the VPS materials for all film thicknesses tested.

Discussion

The Drop Shape Analysis System DSA 10 has been shown to be a useful tool to easily analyze the hydrophilic properties of the unset polyether and VPS impression materials through contact angle measurements.

Results showed that, overall, initial contact angle is not dependent on the thickness of impression material. Polyether materials recorded the lowest contact angles 10 s after the water droplet was placed (45 s after start of mix) and show very small deviations; however, for some VPS impression materials (in this case, Take 1) these contact angles may depend on the thickness of material.

Furthermore, independent of the volume of water in contact with the impression material, the polyether impression materials exhibited lower contact angles, and thus, significantly higher initial hydrophilicity than all measured VPS materials in the unset stage.

Results also showed that the polyether materials exhibited lower contact angles and, thus, significantly higher initial hydrophilicity (contact angle lower than 90°) than all measured VPS materials at 45 s, 120 s, and 24 h after start of mix.

The reason for the differences between these groups is that polyether materials are

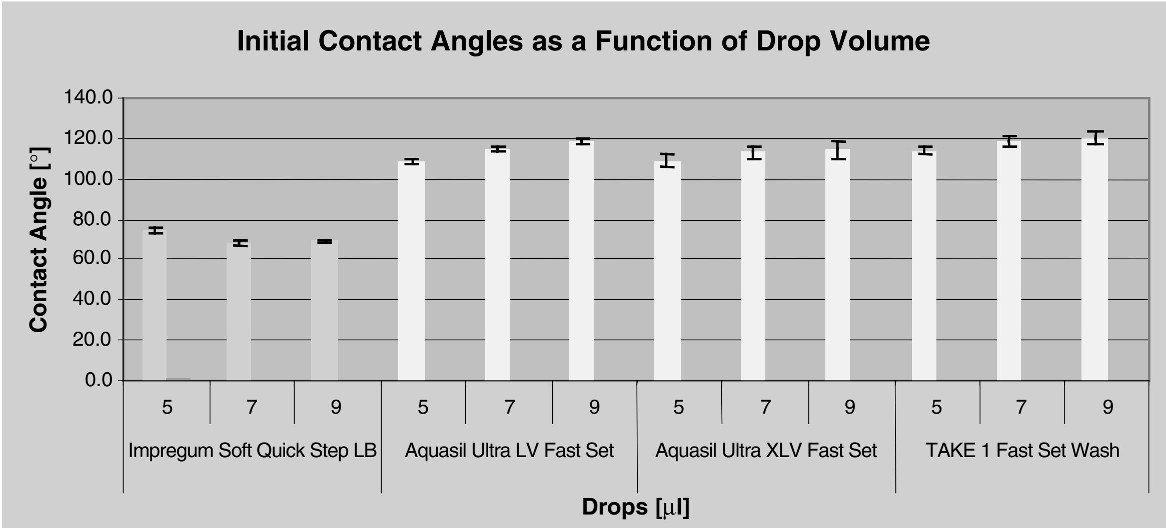

Figure 7. Initial contact angles as a function of drop volume. Water droplet (5, 7, 9 μl) was placed 45 s after start of mix. Standard deviations are indicated as error bars.

hydrophilic[16] by the nature of their chemical make-up while silicones are hydrophobic.[17] This intrinsic hydrophilicity of polyether impression materials ensures lower contact angles compared with silicone impression materials in the unset stage. Other studies[18,19] show that polyether impression materials are characterized by the tendency to favor moist surfaces and produce precise reproductions, and suggest the potential for better intraoral results. This is supported by the results of the current study.

The polyether impression materials, including Impregum Penta *Soft* Quick Step Light Body and Impregum Garant *Soft* Light Body, are more hydrophilic in the unset stage than in the set stage. The degree of hydrophilicity in the set stage is optimized to ensure compatibility with the gypsum slurry for pouring out the impression and very good dimensional stability of the impression during disinfection.[20,21]

By contrast, silicone impression materials, which are intrinsically hydrophobic,[17] require adding surfactants, which are surface-active additives, to achieve hydrophilic properties. This results in a different wetting behavior. When an impression material with surfactants comes into contact with moisture, the surfactant must "migrate" to the surface. This prevents hydrophilicity from fully developing at the very first contact with moisture. Hydrophilicity is of utmost importance when the material flows and the new surface of material is generated (i.e., syringing, seating the tray). It is an important finding that the contact angle is a function of the contact water volume; however, regardless of the amount of water in contact with the impression material, the polyether materials showed a significantly higher hydrophilicity in the unset stage than VPS materials. Therefore, the hydrophilic properties of polyether impression materials may result in

Figure 8. Images of initial contact angles (>300 ms after placing the water droplet): Impregum Soft Quick Step Light Body (left) and Aquasil Ultra LV Fast Set (right). Water droplet (5 μl) was placed 45 s after mix (material thickness 20 μm).

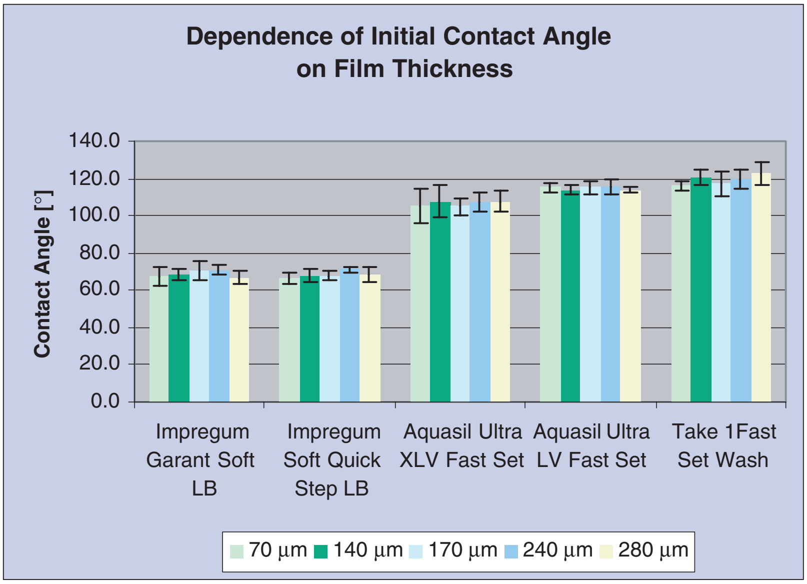

Figure 9. Initial contact angles as a function of material thickness. Water droplet (5 μl) was placed 45 s after start of mix. Standard deviations are indicated as error bars.

superior impressions over VPS materials. Contact angle measurements with the Drop Shape Analysis System DSA 10 might also be applicable to evaluate and optimize other dental materials used for prosthetic and restorative procedures, especially when applied successively.

Conclusion

Within the limits of this experimental approach, the following could be concluded:

1. The new contact angle measurement method employed in this study is useful for the important characterization of the initial hydrophilicity of impression materials in the unset stage. This measurement method produced small standard deviations.
2. Using this method, the results show no correlation between the hydrophilicity data obtained in the set stage and the unset material. Polyether impression materials are more hydrophilic in the unset stage than in the set stage. VPS may show a stepwise development of hydrophilicity in the set stage, which was not observed in the unset stage.
3. Although the parameters, including variation of time, volume of water droplet, and thickness of material, may result in different absolute contact angles, they do not lead to a drastic change in the ranking of the materials with regard to their hydrophilic behavior.

Acknowledgment

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