In the field of prosthodontics, precise reproduction of small features all the way from the impression and gypsum cast to the final product is essential for the quality of the processed dental implant, inlay, onlay, crown, or fixed partial denture. The smaller the features that can be molded and the larger the area reached, the more precisely the original object can be pictured and reproduced in the following steps. How well the contact is established between the impression material and the tooth just after the application of the mass depends on various properties like viscosity and surface wettability. In the beginning, when the most important changes are taking place, the impression material is still very viscous, small features can still be molded, and small cavities can be reached. In the course of curing, the polymerization progress influences the physicochemical properties of the material. Therefore, methods that are able to monitor the changes in these properties on a short time scale and from the beginning need to be applied. Investigation thereof will lead to a better understanding of the processes taking place upon establishing contact and the influences on the outcome of the cast.

Rheologic properties are favorable when materials exhibit a low viscosity in the beginning. Dynamic viscosity has been investigated before and during the setting of materials with different chemical compositions (different silicone recipes and polyether materials\(^1\)).

This led to the conclusion that the polyvinyl siloxane elastomers should be used as soon as possible after being mixed, as their viscosity decreases rapidly. Polyether elastomers have a longer induction period during which the viscosity remains low.

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**Changes in Water Contact Angles During the First Phase of Setting of Dental Impression Materials**

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**Christiane Ziegler, Dr Rer Nat\(^b\)**

**Purpose:** The purpose of this investigation was to examine the changes in wettability of dental impression materials during setting. This study compared the properties of the initial water contact of two different dental impression materials and their subsequent development during polymerization. **Materials and Methods:** Two dental impression materials (Impregum Penta Soft and Aquasil) with different chemical compositions (polyether and polyvinyl siloxane, respectively) were investigated with respect to their changing wetting properties by time-resolved static contact angle measurements. Ten sets of measurements each were taken over a period of 400 seconds with 150 points of data each; the first pictures were used for further characterization of the initial interaction. **Results:** With 73 degrees, Impregum Penta Soft exhibited a significantly lower contact angle, which stayed lower during the process of setting, compared to the silicone-based material. The initial interaction of the droplet showed a repulsive interaction of Aquasil with the water droplet. **Conclusion:** Impregum Penta Soft showed a more hydrophilic behavior during the process of setting compared to Aquasil and can therefore be expected to exhibit better flow properties. The method of time-resolved static contact angle measurements is a well-suited analytic instrument to monitor temporally changing wetting phenomena. *Int J Prosthodont* 2003;16:49–53.
Concerning surface wettability, the best results are achieved if both materials present a similar hydrophilic or hydrophobic behavior, leading to an energetically favorable setting. The requirement for spreading impression materials is high hydrophilicity, indicated by a contact angle toward water of less than 90 degrees, as the substrate enamel (hydroxyapatite) is hydrophilic itself and often covered by hydrophilic liquids like saliva and blood, especially for a subgingival preparation. The spreading behavior and contact angle of a liquid on a solid are determined by the surface tensions of the media in contact being in balance at this triple point. This represents the thermodynamic equilibrium with the lowest Gibbs-Helmholtz enthalpy (Fig 1). The balance of the forces can be described by the Young equation. Wettability has been investigated mostly on static, unchanging states of impression materials with contact angle measurements. The influence of disinfection on the hydrophilicity of materials was investigated on already-set impressions to determine the behavior in relation to the cast of the gypsum,2 the influence of the mixing techniques on this property,3 and the influence of surface activation through surfactants in polyvinyl siloxane–based materials.4

Different hydrophilic and hydrophobic impression materials on ground moist dentin surfaces show no difference in detail-reproducing ability.5 That study focused on the reproduction of plain surfaces, whereas for the reproduction of a tooth, more difficult structures are present. Changes in wettability during setting were investigated with low time resolution by dynamic contact angle measurements with a modified Wilhelmy plate method,6 indicating a strong change in surface wettability in the process of setting.

The two impression materials of different chemical compositions used in this study were chosen arbitrarily to investigate the possibilities of the use of the method of static contact angle measurements to monitor temporal changes. The physical interactions were modeled by inverting the setting using a hydrophilic liquid, deionized water, to spread on the impression material under investigation.

**Materials and Methods**

Time-resolved static contact angle measurements7 were performed with a CAM 200 optical contact angle meter (KSV Instruments). Pictures of the droplet were taken with a fast video camera that enabled a time resolution of 0.1 seconds. The pictures were further processed by the software supplied by KSV and were used to investigate changes in the water contact angle during setting of the dental impression material. Deionized water with a resistance of 18.2 MOhm (Millipore) was used for the contact angle measurements and applied by a syringe to the substrate. One polyether-based material, Impregum Penta Soft (3M/ESPE) and one polyvinyl siloxane–based material (Aquasil, Dentsply/DeTrey) were investigated; for setting times, see Table 1. Both impression materials consist of a base paste and a catalyst paste. The silicone-based material uses a surfactant as an additive.8 Other additives were not specified further.

Mixing was done by an automatic mixing machine (Pentamix, 3M/ESPE) from the original manufacturers’ containers with the corresponding supplied mixing tips. The first few centimeters of mixed paste were discarded to ensure complete mixing. The paste was spread out on a glass cover slip to produce a thin film of dental impression material. The prepared glass slide was transferred immediately to the contact angle instrument and moved toward the water droplet with a controlled volume on the syringe. The time between application of the material to the glass slide and the start of the measurement was 50 seconds. The measurements were started before the droplet touched the surface to record the whole process of jump to contact, and the following developments of the contact angle were determined with three sets of 55 pictures each at intervals of 0.52, 1, and 5 seconds. Zero seconds represented the first picture evaluated after the droplet was deposited on the surface, which also served for the jump to contact evaluation. The water was exchanged for each measurement, and the instruments were cleaned with deionized water twice.

All values gained in this experiment were relative measurements and can therefore only be used in this or similar experimental setups to compare the materials under investigation to each other. The difficulty of achieving absolute values is based on the properties of the impression materials. The surface energy of water is very sensitive to impurities and surfactants and is lowered in a significant way from its original
value of 72.6 mN/m upon contact with an unset dental impression material, as shown in earlier measurements, which can be attributed to the release of surfactants and other additives.

**Results**

**Dynamic Changes in Development of Water Contact Angle**

Both impression materials changed in their surface properties while setting, as the initial high contact angle toward water was lowered, enhancing the wettability. These changes took place fastest during the first 40 seconds. During this initial time, the impact of wettability has to be considered highest, as the impression material still had a low viscosity. The two materials also showed significant differences in various parameters.

The starting value of the polyether material Impregum Penta Soft (73 ± 2 degrees) was below 90 degrees and therefore hydrophilic. Aquasil exhibited a more hydrophobic behavior, with 117 ± 5 degrees at the start. For clarity reasons, the further development of the contact angles was represented by 10 values from the first 150 seconds (Fig 2). The values taken in the first 40 seconds clearly showed the strong decline in contact angle values in the first part of the application. The whole set of data can be seen in Fig 3, with each point of data representing the median of the 10 measurements and an error bar clearly indicating the significant difference in the behavior of the two materials.

**Jump to Contact of a Water Droplet to the Surface**

The establishment of the contact between the water droplet and the impression mass surface showed distinct differences for the two materials. In Figs 4 and 5, the approach of the support to the needle and the contact can be seen within a period of 4 seconds. The detachment of the droplet from the tip of the syringe happened on different time scales.

Upon approaching the Impregum Penta Soft surface, the water droplet immediately established a large contact area with the polyether surface (Fig 4). The rupture of the contact between the droplet and the tip of the needle happened spontaneously without further retraction of the surface from the needle, so no intermediate picture is available. In the case of the polyvinyl siloxane–covered surface (Aquasil), the established

<table>
<thead>
<tr>
<th>Brand</th>
<th>Material</th>
<th>Processing time from start of mixing</th>
<th>Setting time from start of mixing</th>
<th>Residence time in the mouth</th>
</tr>
</thead>
<tbody>
<tr>
<td>Impregum Penta Soft</td>
<td>Polyether</td>
<td>2:45</td>
<td>6:00</td>
<td>3:15</td>
</tr>
<tr>
<td>Aquasil</td>
<td>Polyvinyl siloxane</td>
<td>2:30</td>
<td>5:00</td>
<td>3:00</td>
</tr>
</tbody>
</table>

*According to manufacturers.
contact area stayed smaller and the droplet shape was spherical, with the large contact angles typical of an interaction with a strongly hydrophobic surface (Fig 5). The water droplet exhibited a stronger adherence to the tip of the needle than to the surface, so rupture of the droplet from the tip could only be realized by a higher force through further retraction of the support. This shows that the overall energy was minimized for Impregum Penta Soft by spreading of the water droplet on the surface and for Aquasil by avoidance of a close contact, so no wetting was seen.

Discussion

The differences in chemical composition and reaction influenced the wetting and flow properties of the impression materials.

Dynamic Changes in Development of Water Contact Angle

The results of this study agree with earlier studies done by dynamic contact angle measurements. The Wilhelmy plate method was used on specially designed plates to hold the impression material while setting, with results exhibiting a decrease in the contact angle toward water in the process of setting in the first 154 seconds of more than 23 degrees in the case of a polyether material and more than 56 degrees for a polyvinyl siloxane material. Lowering of the surface tension of the water was observed as well.

The low contact angle of Impregum Penta Soft can be attributed to the inherent hydrophilicity of the material polyether, originating in the chemical composition, which exhibits oxygen groups between the
more hydrophobic parts. This is not so for the polyvinyl siloxane materials. The drastic change of Aquasil can be attributed in part to the release of surfactants into the water, which lowered the surface tension of water and therefore the contact angle. The release of surfactants minimized the difference in contact angle over time in comparison to nonhydrophilic materials. The change in wettability of both materials can be attributed in part to the influence on the surface tension of water of monomers and other soluble molecules that are part of the chemical composition of the material and not specifically identified by the manufacturer.

The initial interaction of the good wetting behavior of the polyether and a repulsive interaction of water with the polyvinyl siloxane material should lead to better and more detailed reproduction abilities of Impregum Penta Soft. The ability of Impregum Penta Soft to reproduce small features on wet surfaces better than other impression materials, including Aquasil, was demonstrated on wet gypsum. Clinical studies have shown good properties of Impregum Penta Soft in dental applications.

Jump to Contact of a Water Droplet to the Surface

The results of the second part of the experiment were consistent with the data acquired in the first part. The interaction of the droplet with the surface showed extrapolation of the data collected during the further setting of the material. The initial high values of the contact angle were related to Aquasil’s hydrophobic behavior and difficulty of establishing contact between the water and the material’s surface. Together with the information about the starting contact angle, this leads to the conclusion that Impregum Penta Soft exhibits better conditions for wetting of the hydrophilic dental surface than Aquasil.

Conclusion

Time-resolved static contact angle measurements showed that the polyether impression material Impregum Penta Soft has a higher hydrophilicity during the process of setting than the polyvinyl siloxane material Aquasil. Further indication of the more hydrophobic behavior of Aquasil was given by monitoring of the initial contact between the substrate and the droplet, represented by the shape of the droplet and contact area. The hydrophilicity of Impregum Penta Soft in the initial stages during setting should lead to better wetting properties and the possibility to reproduce features of the dental substrate that are not easily accessible. The measurements prove the ability of the method of time-resolved static contact angle measurements to monitor changes in hydrophilicity during the setting phase of impression materials on a fast time scale.

References