

The Dynamic Interaction of Water with Four Dental Impression Materials During Cure

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Keywords

Impression materials; curing time; drop shape analysis; wettability; contact angle; hydrophilicity.

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Abstract

Purpose: The purpose of this work was to investigate the interaction of water with four different dental impression materials: Aquasil (Ultra XLV Type 3), Take 1 (Wash Regular Set), Genie (Light Body, Standard Set), and Impregum Garant (Soft Light Bodied Consistency).

Materials and Methods: Apparent contact angles of de-ionized water made against thin horizontal sample films of the different materials under different conditions were measured from analysis of profile images of symmetrical sessile drops of water placed on the sample films using a Model FTÅ200 dynamic drop shape analysis system, which included a JAI M30 high speed CCD camera combined with a zoom microscope. Data were taken for specimens of dry ages (times following mixing) from a minimum of 20 seconds up to 1220 seconds. Imaging was started before the initial water/impression material contact, and lasted for at least 420 seconds in each case. The interval at the beginning of each run was 0.033 second, and then increased by a factor of 1.012 to the end. During the initial 3 seconds following the drop deposition, the drop's shape oscillated due to inertial effects, so apparent contact angle data during this period were neglected in all cases. All measurements were made at room temperature. The drops were enclosed in a humidified chamber that suppressed evaporation. All data were repeated at least five times, and results were analyzed where appropriate using one-way ANOVA. Microscopic images of the water/impression material interactions for fresh (uncured) materials were acquired to reveal the destructive interactions that resulted from such contact. Finally, surface tension measurements were made of water that had been contacted with material of varying dry age using the pendant drop method capability of the drop shape analysis system. These helped to assess the origin of hydrophilicity development for the different materials.

Results: For short curing times (dry ages), water showed a destructive effect on the integrity of all of the impression materials, as evidenced by the formation of a crater beneath the water drop and a scum of material at its surface. These effects diminished with dry age until a critical curing time was reached, beyond which such destructive interactions were no longer detectable. These critical curing times were determined to be 80, 140, 110, and 185 seconds for Aquasil, Take 1, Genie, and Impregum, respectively. The initial contact angle following the respective critical curing time was lowest for Impregum, at 66°; while values for Aquasil, Genie, and Take 1 were 93°, 104°, and 110°, respectively. Beyond the critical curing times for the different materials, different degrees of hydrophilicity were observed. Aquasil showed the lowest final contact angle (<10°), with Impregum, Take 1, and Genie showing 31°, 34°, and 40°, respectively. Measurements of the surface tension of water after contact with the different materials suggested that for Aquasil, hydrophilicity appears to be developed through the leaching of surfactant from the material, whereas for Impregum, Take 1, and Genie, hydrophilicity is developed at least in part through a change in surface structure in contact with water. Impregum and Aquasil materials of dry ages well beyond the critical curing time exhibited a stick-slip behavior in their interline movement or contact angle evolution. This was believed to be due to the slowness in the leaching of surfactant (in the case of Aquasil) or the re-orientation of

unleachable surface groups (in the case of the other materials) in comparison to the inherent kinetics of water drop spreading.

Conclusions: All materials investigated in the fresh, uncured state showed qualitative decomposition when put in contact with water through the formation of a crater beneath the water drop and a scum of material at its surface. These effects diminished with curing time until beyond a critical value, no such effects were evident. The initial hydrophilicity of the materials as determined by the contact angles obtained at their respective critical dry ages was greatest for Impregum. Beyond the critical curing time, different degrees of hydrophilicity were observed, with Aquasil showing the lowest final contact angle.

The purpose of a dental impression is to capture and reproduce oral tissue detail with a dimensionally stable material as a mold to fabricate an accurate replica for definitive restorations. The fit and ultimate clinical success of a dental restoration is dependent upon the accurate, void-free positive casts or dies of the negative reproduction.¹⁻⁴ The wettability of impression materials is regarded as one of the most important properties for clinical success. Recent advances have focused on making these materials more hydrophilic, thereby allowing the material to make a more intimate contact with the oral tissue, with the aim of capturing better surface detail and fewer defects.⁵⁻⁸ There have been numerous studies of the wettability of cured impression materials;⁹⁻¹¹ however, the wettability of impression materials during setting has been much less investigated, despite the fact that it is behavior under these conditions that may be most clinically relevant. Over the working time, the soft impression material flows so that a new surface of material is generated and comes into contact with moisture. Adequate wetting of the material surface during the working time is thus decisive for clinical success in registering fine detail.¹²⁻¹⁴ Previous investigations^{9,15-19} suggest that the interaction of water with fresh dental impression material is highly time dependent and may be quite complex. There is thus a need for more study of the evolution of hydrophilicity of dental impression materials during cure.

A wide variety of impression materials is available, including polyethers, vinylpolysiloxanes (VPS), condensation polysiloxanes, reversible hydrocolloids, alginate materials, polysulfides, and others, each with their own properties, advantages, and disadvantages. New impression materials are continually appearing in the dental marketplace, and there is a constant effort by manufacturers to make the inherently rather hydrophobic siloxane-based materials more hydrophilic.^{1,2,20,21} There should be an optimized level of interaction between the impression material and the oral tissue. The hydrophilic nature of the impression material should be strong enough to achieve a satisfactory wetting, but not so strong as to prohibit easy removal of the mold, once the impression is taken. From the surface energy standpoint, when preparing the negative mold, the unset impression material should have a lower surface tension (energy) than that of the oral tissue; however, in creating the positive casting from the negative mold, the cured impression material should have a high surface free energy (relative to the surface tension of positive cast material) to ensure complete wetting by the casting material.

There are two commonly used methods for determining the dynamic wettability of impression materials: dynamic contact angle sessile drop goniometry, and dynamic Wilhelmy tensiometry.^{9,13} Tensiometry is unsuitable, however, because it requires that the material surface be held in a vertical position and would thus continually deform under the influence of gravity. Sessile drop goniometry is thus the suitable choice. Even using this method, there are a number of potential problems that need to be considered. During any initial period of the drop/material contact (~2 to 3 seconds), the drop shape fluctuates significantly due to inertial effects, so the earliest relevant values of contact angle correspond to a water contact time of approximately three seconds. Values taken at three seconds are thus referred to as initial contact angles. Also, during the early stages of setting, the impression material is soft and can be easily deformed and perhaps damaged in more serious ways. Finally, the contact angle measurements may be affected by drop volume decrease due to evaporation.

The goal of the present study is to obtain meaningful results for the water/material interactions of four impression materials, including one polyether and three VPS materials, before, during, and after setting.

Materials and methods

Four dental impression materials were obtained to investigate their hydrophilic characteristics using dynamic contact angle studies. These four materials were as follows:

- Aquasil (Ultra XLV Type 3, light bodied consistency), a quadrofunctional hydrophilic addition reaction silicone obtained from Dentsply Caulk (Milford, DE). The total set time was given as 5:00 minutes.
- Take 1 (wash regular set), a hydrophilic VPS material provided by Kerr Corp. (Romulus, MI). The total set time of the material was 5:00 minutes.
- Genie (light body, standard set, ultra hydrophilic), a VPS addition-cured material obtained from Sultan Chemists, Inc. (Englewood, NJ). The set time for this material was given as 4:30 minutes.
- Impregum Garant (soft light bodied consistency), a polyether impression material provided by 3M ESPE AG Dental Products (Seefeld, Germany). The total set time for this material was given as 5:30 minutes.

These materials were all provided in cartridges together with their matching mixing tips. A 3M ESPE dispenser (type HP; 3M ESPE Dental Products, St. Paul, MN) was used to mix the two components (i.e., base and catalyst) of each material through the extrusion process inside the tip.

The contact angles of de-ionized water made against thin horizontal sample films of the different materials under different conditions were determined from analysis of profile images of symmetrical sessile drops of water placed on the sample films. The sample films were prepared by doctor-blading the fully mixed compound onto a glass slide with the aid of shims that produced a sample thickness of approximately 100 μm and a width of 1 cm. (The first centimeter or so of the mixed paste coming out of the mixing tip was discarded to ensure complete mixing.) The sample was placed on a stage underneath the tip of a water-dispensing disposable blunt-end stainless steel needle with an outer diameter of 0.71 mm, attached to a syringe pump controlled by computer for delivery of the water drop to the test surface. The drop size was approximately 10 μl .

Drop images were acquired as a function of time using a Model FTÅ200 dynamic drop shape analysis system (First Ten Ångströms, Portsmouth, VA), which included a JAI M30 high speed CCD camera combined with a zoom microscope. The dispensing of the drop, the time schedule of image capture, and the subsequent analysis of the contact angles and other drop parameters were effected by computer using the FTÅ32 software supplied with the instrument. Contact angles were measured by fitting a mathematical expression to the shape of the sessile drop and then calculating the slope of the tangent to the drop at the liquid-solid-vapor interface line.

To capture the initial contact made between the water drop and the impression material, the imaging was started before the initial water/impression material contact, and lasted for at least 420 seconds. The interval at the beginning of each run was 0.033 seconds, and then increased by a factor of 1.012 to the end. The dry age, τ , is defined in this study as the time elapsed between the mixing of the two components (i.e., base and catalyst) of the dental impression material (i.e., pulling the gun trigger) and the time of the initial water drop/material contact. The smallest value τ achievable on a consistent, repeatable basis was 20 seconds, and materials put in contact with water after this brief dry curing time are designated hereafter as fresh materials. The water contact time, Δt , is defined as the time measured following the contact of water with the material. The total age, t , can thus be reckoned as:

$$t(\text{total age}) = \tau(\text{dry age}) + \Delta t(\text{water contact time})$$

For each material, the contact angle was determined as a function of time for a series of dry ages, τ . Each set of measurements was repeated at least five times.

A potentially serious problem in the interpretation of the drop profiles was evaporation. To minimize or eliminate its effect, a transparent Plexiglas® box was constructed to cover the sessile drop sitting over the dental impression material film. The top wall of the box had a small hole allowing for the syringe needle to come to the water dispensing position. Cotton tissue, which was wetted with warm water prior to each set of contact angle

measurements, was attached to the inner side of the top wall. The evaporation of the warm water in the small transparent box resulted in water vapor-saturated air in the box preventing the water drop (dispensed at room temperature) from evaporating. The technique proved to be effective in reducing the water drop evaporation to less than 1% after 600 seconds.

A Model IX70 Olympus invert microscope (Olympus America, Melville, NY) equipped with a high-speed camera (Model: QCOLOR3; Olympus America, Melville, NY) was used to capture images of the interaction of fresh materials with water immediately upon contact. A small sample (100 μm) of fresh material was applied to a glass slide, and water was added at the side of the sample on the glass slide. The top view (magnification 10 \times) of the events occurring upon the contact between the water and the material was captured by the high-speed camera through the microscope.

To investigate the origin of hydrophilicity development, surface tension measurements were made of water that had been put into contact with the different impression materials of different dry ages. Samples of a given dry age were prepared in the shape of small cups filled with some water. After five minutes, the water was sucked into a clean syringe. Then, a pendant drop was formed at the syringe tip, and the surface tension of liquid was determined from the drop shape (pendant drop method) using the FTÅ200 drop shape analysis system and software.

Results

Figure 1 shows the evolution of apparent contact angle for the four fresh impression materials (i.e., with the minimum attainable dry age, τ , of 20 seconds) as a function of total age, t . The initial apparent contact angle was high for each material, after which it at first decreased rapidly ($\Delta t < 50$ seconds) and then more slowly as it reached a final value. Because of oscillating drop shape due to inertial effects during the very first approximately 2 to 3 seconds of the drop impact with the material surface, data for this time interval in all of the cases studied are not reported. For the water contact times, Δt greater than approximately 5 seconds, the error for almost all data (including the final contact angle) was not greater than $\pm 2^\circ$ (95% confidence level).

Figures 2A–C show images of a cap layer removal from a water drop on Impregum using a needle. Simultaneous with the scum formation at the drop surface, a crater was formed beneath the drop. Figure 2D shows an example of the crater left behind following removal of the sessile drop.

Results for the apparent contact angle dependence on time for the different materials over a range of dry ages were as follows. Figure 3 shows contact angle development for different samples of Aquasil of different dry ages. The final contact angle was significantly dependent on the dry age. For the sample with $\tau = 80$ seconds, the final contact angle was slightly greater than the apparent value for the shortest dry age (fresh material), while an increase of τ to 110 seconds resulted in a significant decrease in the final contact angle. For the samples with a $\tau > 110$ seconds, the final contact angle remained small ($< 10^\circ$). It should be noted that final contact angle values were found in all cases despite the apparent abrupt end to the curves for

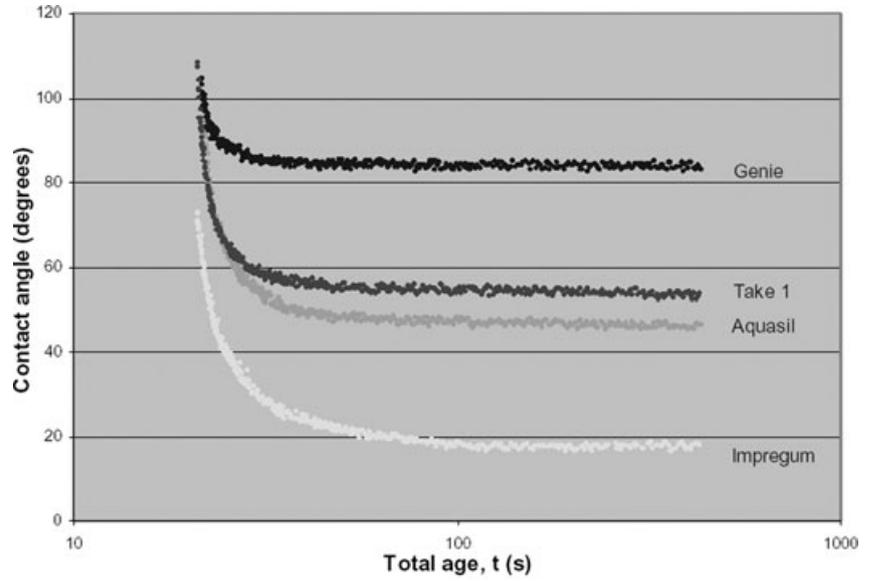


Figure 1 The contact angle of the four fresh dental impression materials as a function of total age t (dry age, $\tau = 20$ seconds).

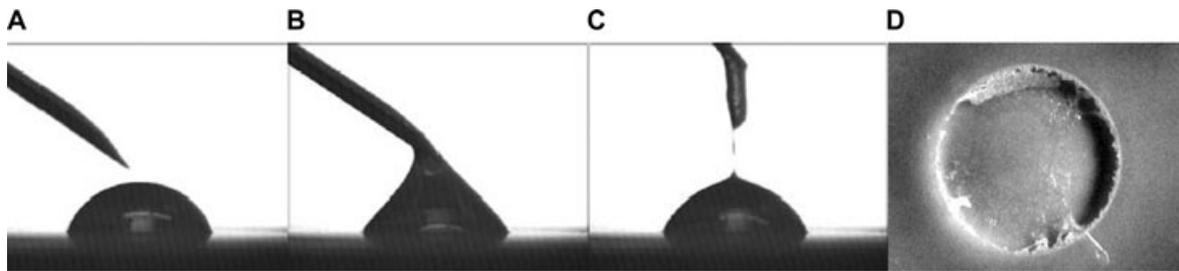


Figure 2 The cap layer removal using a needle. (A) pre-contact; (B) contact; (C) post-contact. A crater left behind on an Impregum surface ($\tau = 20$ seconds) after removal of the sessile drop (D).

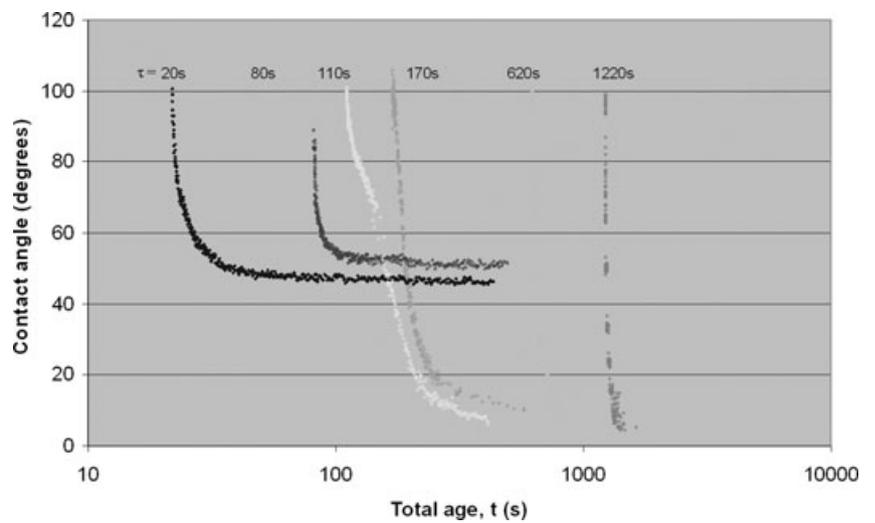


Figure 3 The contact angle behavior for Aquasil dental impression material samples of varying dry age, τ , as a function of total age, t .

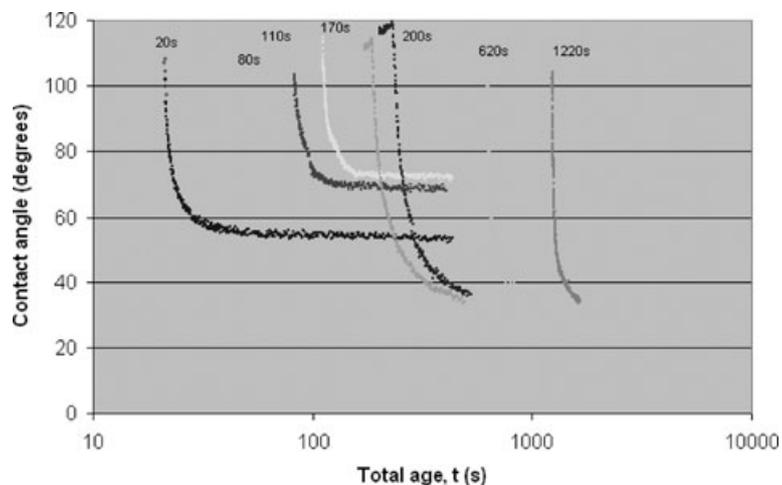


Figure 4 The contact angle development for Take 1 samples of varying dry age, τ , as a function of total age, t .

higher τ produced by the time compression of the logarithmic coordinates. A similar pattern of behavior was observed for Take 1, as seen in Figure 4. Increasing the dry age from 20 to 110 seconds, resulted in an increase in the water/Take 1 contact angle from 53° to 72° . For the samples with τ values of 170 and 200 seconds, there was some weak pinning (stick-slip interline motion) for early water contact times, prior to the significant decrease in the final contact angle achieved. The final contact angle for Take 1 samples with $\tau > 170$ seconds, was approximately 35° . Figure 5 shows the contact angle evolution with time for Genie samples of increasing dry age. The general trend was a decrease in final contact angle with the dry age, dropping from 82° to 39° as dry age was increased from 20 to 140 seconds. Further increases in dry age did not change the final contact angle significantly. Results for Impregum, shown in Figure 6, differed from those obtained for the other materials in that a significant increase in the final contact angle (from 19° to 61°) was observed as the dry age increased from 20 to 185 seconds. Increases in τ beyond 185 seconds, however, produced a decrease in the final contact angle (down to 30° for a dry age of 620 seconds and 40° for a dry age of 1220 seconds).

Figure 7 shows the final contact angle for the four dental impression materials as a function of dry age τ .

Discussion

An important observation for the fresh materials was that a layer of scum formed on the water drop surface. Once brought into contact with the water phase, some of the solid material started to disintegrate at the water/solid boundary and migrate to the surface of water drop. The detached fragments of solid material at the drop surface merged into a coherent film (i.e., a cap) on the sessile drop. Figures 2A–C show images of a cap layer removal from a water drop on Impregum using a needle. Simultaneous with the scum formation at the drop surface, a crater was formed beneath the drop. Figure 2D shows an example of the crater left behind following removal of the sessile drop. Some portion of the cap is seen to be attached to the crater edge. Because the integrity of fresh material is thus compromised upon contact

with water, interpretation of the resulting behavior in terms of a simple contact angle development would be misleading.

To better understand the nature of the fresh material disintegration, the process of water contact was observed microscopically. A small portion of each material was applied to a glass slide, and a drop of water was put in contact with it from the side. The pattern of interaction of the polyether Impregum was one of a network of strands of polymer with filler separating from the edge of the solid and spreading at the surface of water. This network later consolidated into a coherent film. For Take 1, the disintegration produced discrete particles migrating into the water phase. This may indicate that the migrating phase was mainly filler. The particles later consolidated into coherent rafts on the water surface. The behavior of Take 1 was representative of that of the VPS group (Aquasil, Genie, and Take 1). The penetration of water into the soft solid could be noted (as a dark band) for both materials, but was more pronounced for the Impregum. For each material, the water/material apparent contact angle measurements were thus carried out for a series of dry ages, τ . This provided a systematic approach to the water/material contact behavior, before, during, and after setting. Disintegration, water penetration, scum capping, and crater formation were explicitly examined as a function of dry age, τ . These complex effects were reduced and eventually eliminated as sample dry age times were increased. One of the important objectives was to determine the minimum required dry age in order not to have disintegration effects for each material, identified as its critical dry age or curing time, τ_c .

As can be noticed (Fig 7), the dry age can affect the final contact angle of material significantly. The general trend is that the final contact angle increases at the beginning, followed by a significant decrease. At short values of τ , the uncured material is soft, and the placement of a water drop results in the formation of a crater beneath the sessile drop. For each material, an increase in the dry age time results in a decrease in the crater depth as the sample gels,²² providing more resistance to deformation and disintegration. A shallower (or non-existent) crater means that a greater proportion (or all) of the sessile drop lies above the material free surface, providing an explanation

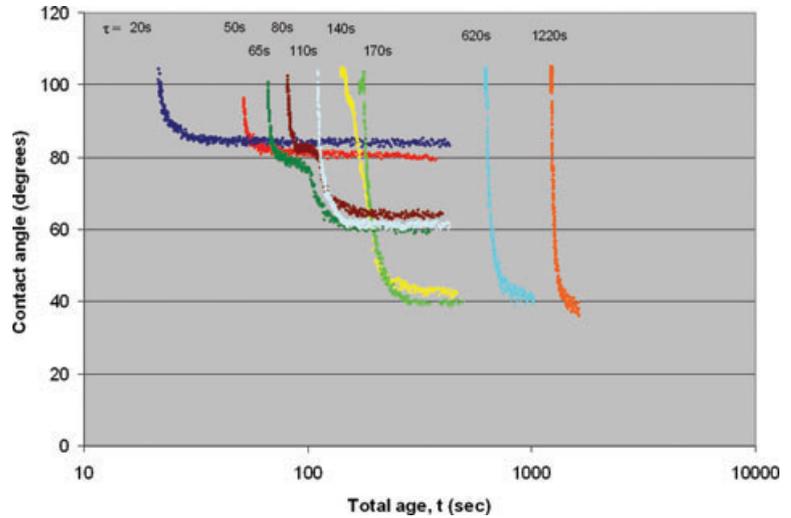


Figure 5 The water/genie contact angle development for Genie samples of varying dry age, τ , as a function of total age, t .

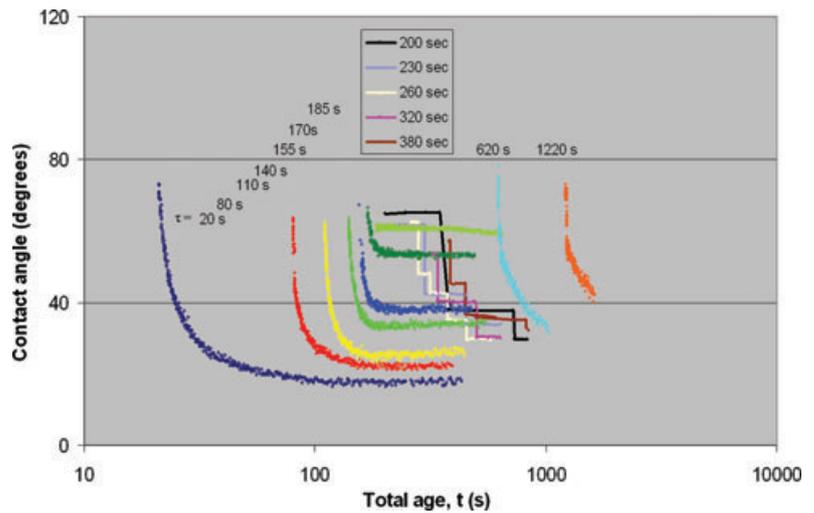


Figure 6 The contact angle behavior for Impregum samples of varying dry age, τ , as a function of total age, t . The samples with dry age $200 \leq \tau \leq 380$, which exhibited stick-slip behavior, are denoted with the corresponding color next to their dry age.

for the final contact angle increase observed for low- τ samples of Aquasil, Take 1, and Impregum.

An additional complication was noticed, first for Impregum. For sample dry ages of 200 to 380 seconds, a step-wise contact angle development occurred, differing from the continuous contact angle decrease observed otherwise. Further examination also revealed a step-wise contact angle development for sample dry ages of 620 and 1220 seconds, not noticeable in Figure 6, due to the compression of the logarithmic time scale. One probable origin of this behavior could be the diffusion-limited migration of surface active compounds from the impression material into the water drop, producing a gradual change in surface tension, which must reach a critical value before the drop can suddenly recede to a new advancing contact angle. Aquasil probably contains a relatively low molecular weight silicone surfactant, which is readily diffusible even after curing. The other silicones, Take 1 and Genie, contain hydrocarbon surfactants with balanced hydrophilic-hydrophobic characters, which tend to lock the hydrophobic end of the molecule into the surface of the impression, yielding limited opportunity for diffusion into the aqueous phase, thus explaining their lack of

“stick-slip” behavior. The lowered surface tension of the water in contact with Impregum, and the observed “stick-slip” behavior, suggest that it, too, might have some compounds capable of diffusing into the water phase.

The systematic acquisition of the time-dependent contact angle data as a function of dry age, together with direct observation of the water/material interactions, has permitted the identification of three descriptors in terms of which of the four materials studied may be characterized and distinguished. The first of these is the critical dry age, τ_c , defined earlier as the age beyond which material degradation upon water contact could not be observed. The second is the initial contact angle, defined as the contact angle at $\Delta t = 3$ seconds, the first measurable value obtained after inertial effects have damped out, for material at its respective critical dry age, τ_c . The last is the final contact angle ($t \rightarrow \infty$) for each material at its respective critical dry age, τ_c . These descriptors are summarized in Table 1. The critical dry age varied from 80 seconds, for Aquasil, up to 185 seconds for Impregum. This property would appear to be of clinical relevance because for all light-body or low-consistency impression materials that can be injected directly on oral tissues in the

mouth, such a destructive interaction as might occur for dry ages less than this value, the quality of detail registration by the impression material may be compromised. Specifically, large amounts of moisture that become encapsulated could result in defects in the final impression. On the other hand, products that are able to incorporate small amounts of water may give higher detail reproduction than those that are not able to do so. With regard to initial hydrophilicity, among the materials studied, only Impregum displayed this property. Its initial contact angle ($\Delta t = 3$ seconds) was 66° , in comparison to values of 93° , 104° , and 110° for Aquasil, Genie and Take 1, respectively. Initial hydrophilicity may also play a role in the achievement of good registry, as it is during these early times that the intimacy of contact between impression material and the moist oral surface is established. The final contact angle, which may be relevant to the ease of removal of the set material from the mouth, was seen for Aquasil to be markedly lower ($<10^\circ$) than those recorded for the other materials, which lay between 30° and 40° .

The different patterns of interaction with water in the pre-cured state for the different materials, and the subsequent hydrophilicity development, was believed to be traceable to their different composition. The possible presence of leachable hydrophilic additives (surfactants) could be detected by any significant drop in the surface tension of water that is put in contact with the material. The lowered surface tension would lead to a decrease in contact angle. Surface tension results are tabulated in Table 2. As can be noticed, the surface tension of water after being in contact with Aquasil decreased significantly, whereas for Impregum, Genie, and Take 1, the change was much less,

Table 1 Minimum τ values to avoid the capping effects and crater formation

Material	Aquasil	Take 1	Genie	Impregum
Minimum dry age, τ_c (seconds)	80	140	110	185
Initial contact angle ($\Delta t = 3$ seconds) for materials of minimum dry age	93°	110°	104°	66°
Final contact angle for materials of minimum dry age	$<10^\circ$	34°	40°	31°

Table 2 Surface tension of water (mN/m) measured at 5 minute intervals of contact with different pre-aged materials (confidence limit: 95%)

	$\tau = 5$ minutes	$\tau = 10$ minutes	$\tau = 15$ minutes	$\tau = 20$ minutes
Genie	70.3 ± 0.6	69.8 ± 0.4	70.2 ± 0.7	70.7 ± 0.4
Take 1	69.0 ± 0.1	68.5 ± 0.5	70.2 ± 0.6	70.3 ± 0.3
Impregum	61.1 ± 0.3	60.2 ± 0.2	61.1 ± 0.3	65.2 ± 0.2
Aquasil	49.6 ± 0.2	49.1 ± 0.1	49.5 ± 0.4	49.1 ± 0.7

(The surface tension of the water before contact was 72.6 mN/m).

indicating little or no leaching out of surfactants from these samples. For these materials it is likely that hydrophilicity developed primarily as surface structures re-oriented themselves while in contact with water to become more water compatible.

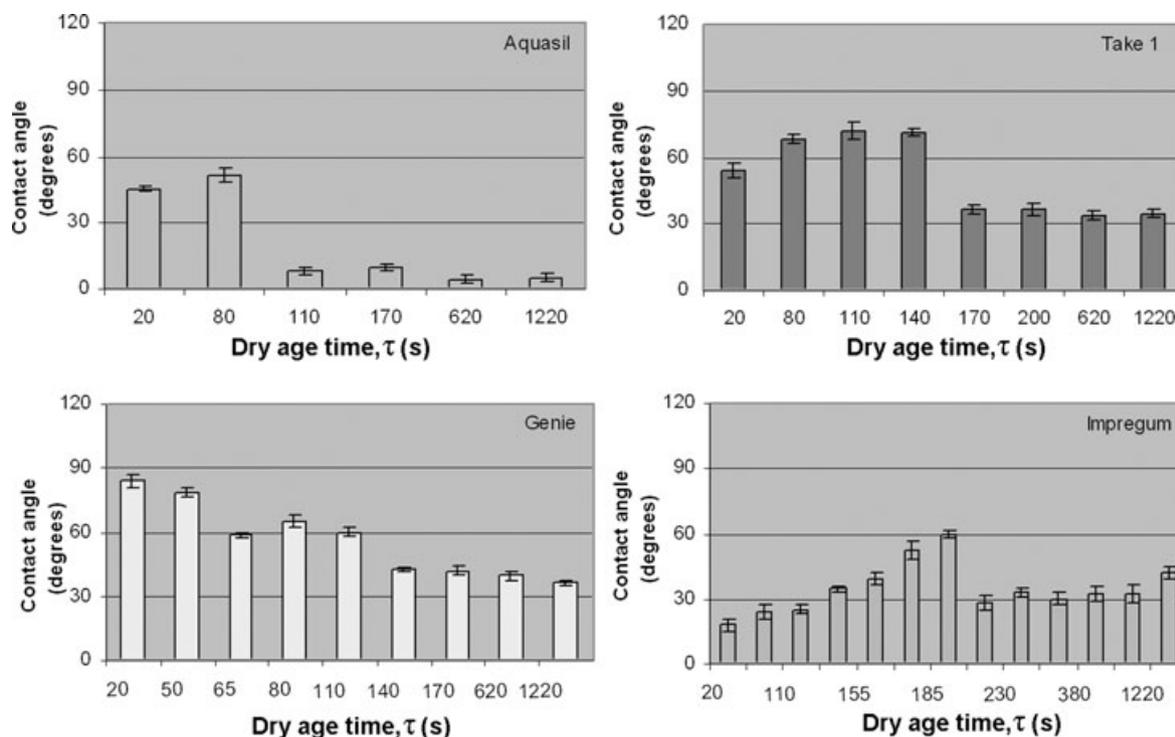


Figure 7 The final contact angle as a function of dry age time, for four dental impression materials (confidence limit for the error bars: 95%).

Stick-slip patterns of interline movement for cured specimens suggest that these processes are slowed as the curing process increases the density of crosslinking in the specimens. The unusual 10° increase in the final contact angle of Impregum on increasing the dry age from 620 to 1220 seconds (Fig 6) is also consistent with this hypothesis.

Conclusions

The dynamic interaction of water with four dental impression materials (Aquasil, Take 1, Genie, Impregum) has been investigated as a function of their dry curing time, that is, dry age, from 20 to 1220 seconds, using contact angle goniometry and direct observation. At the early stages of setting (i.e., for small dry ages), the impression material contact with water resulted in a destructive interaction manifest as a scum formed at the top of the drop and a crater formed beneath it. Thus for these fresh, uncured materials, only apparent contact angles could be reported, but they all showed a monotonic decrease with water contact time from initially higher values to final lower values, suggesting a transition through which each material became more hydrophilic once the water droplet had been placed. The pattern of destructive interaction of the fresh impression material with water, as observed microscopically, was different for Impregum than for the other materials. For Impregum, a network of strands of polymer with filler separated from the edge of the solid and spread at the surface of water, whereas for the other materials the disintegration produced discrete particles migrating into the water phase.

The time course of dynamic contact angle for each material as a function of its dry age (curing time before water contact) was investigated. The apparent water contact angle of these materials was found to be significantly dependent on their dry age, and a critical dry age or curing time, τ_c , different for each material, was identified as that beyond which no detectable disintegration with water contact occurred. These values were found to be 80, 140, 110, and 185 seconds for Aquasil, Take 1, Genie, and Impregum, respectively. For materials at their respective critical dry ages, initial contact angles (obtained 3 seconds after water contact, the time required for drop inertial effects not to influence the apparent contact angle) were obtained and found to be: 93° , 110° , 104° , and 66° for Aquasil, Take 1, Genie, and Impregum, respectively. Impregum was thus found to have substantially greater initial hydrophilicity than the other materials. Final contact angle values (for materials of critical dry age) for Aquasil, Take 1, Genie, and Impregum were: $<10^\circ$, 34° , 40° and 31° , respectively.

While the hydrophilicity developed by Aquasil appeared to have been achieved by the presence of added surfactant (as evidenced by a sharp drop in the surface tension of water that had been contacted with that material), that of Impregum (which showed the highest initial hydrophilicity), Take 1, and Genie, could be attributed to the presence of an essentially unleachable modifier, which can impart a hydrophilic character to the surface structure upon re-orientation.

Impregum and Aquasil materials of dry ages well beyond the critical curing time exhibited a stick-slip behavior in their interline movement or contact angle evolution. This was believed to be due to the slowness in the leaching of surfactant (in the

case of Aquasil) or the re-orientation of unleachable surface groups (in the case of the other materials) in comparison to the inherent kinetics of water drop spreading.

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