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Recording surface detail on moist surfaces with elastomeric impression materials

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Abstract - *The objective was to assess the ability to accurately record detail on moist surfaces for three elastomeric impression materials derived from different polymers. One polyvinylsiloxane, one polyether and one hybrid material containing a copolymer of siloxane and polyether polymers were used. Impressions were recorded of moist gypsum casts having both a shallow (~ 20 µm) and deep (~ 180 µm) groove reproduced on their surface. The grooves in the casts and in the impressions were profiled using a non-contacting laser profilometer. Comparisons were made between the groove depths in the casts and impressions (paired t-test). The results indicated that all of the tested materials accurately recorded dimensions in the x-y plane. However, there was evidence that the polyether and hybrid materials were more accurate than the polyvinylsiloxane in recording the true depths of the deep grooves (z plane) under moist conditions. It was concluded that the more hydrophilic nature of the polyether and hybrid materials enabled them to record more accurate impressions of moist surfaces, particularly in areas of difficult access as modelled by the deep grooves.*

KEY WORDS: elastomers, dental impressions, polyvinylsiloxanes, polyethers, detail reproduction

INTRODUCTION

The surface detail reproduction and dimensional accuracy of impression materials plays a major part in determining the extent to which indirectly produced appliances and restorations will have an acceptable 'fit'. Although there is general acceptance that elastomeric materials are more accurate and precise than hydrocolloids there is a degree of uncertainty over the extent to which the elastomers can be relied upon to produce acceptable impressions of moist surfaces [1-4]. The base polymers used to construct some elastomers are inherently hydrophobic [5] and this may have the effect of limiting the extent to which surface detail in a water containing surface can be recorded. Traditionally this problem has been solved by suggesting that the dentist should thoroughly dry the area to be recorded.

One potential advantage of polyether materials over the silicone products is that they are more hydrophilic and are therefore able to attain more intimate contact with moist intra-oral surfaces [6]. Over recent years some silicone products have been modified to increase their hydrophilic nature and this may improve the ability to record detail although there is some disagreement amongst researchers as to the extent of this improvement [1]. Recently, a hybrid material has been produced which combines the chemistry of both polyvinylsiloxane and polyether materials. The material undergoes setting through polymerisation of both silicone and ether moieties and during polymerisation the two polymeric species become linked and this helps to prevent phase separation. The potential advantages of this system are that it combines the very good elastic characteristics associated with polyvinylsiloxanes with the hydrophilic characteristics of the polyethers.

The setting reaction of a polyvinylsiloxane such as Aquasil is characterised by an addition reaction as shown in (1)

$$CH_2{=}CH\text{-}Si\,(CH_3)_2\text{ - }O \sim \quad + \quad H - Si\,(CH_3)_2\text{ - }O\text{ - }Si(CH_3)_2 - O \sim + \text{ Pt catalyst} \longrightarrow$$

$$\sim O - Si\,(CH_3)_2\text{- }CH_2\text{-}CH_2\text{ - }Si\,(CH_3)_2\text{ - }O\text{ - }Si(CH_3)_2 - O \sim \qquad (1)$$

The degree of conversion and cross-linking may be influenced by a high functionality resin containing multiple vinyl groups. However, the essential character of the polymeric species is shown in equation 1, having a high hydrocarbon and siloxane concentration. Surface active agents, typically characterised by an aromatic head and polyether tail are added to improve hydrophilic characteristics but the agent is blended with the polyvinyl siloxane and not attached to it.

In the case of polyether materials, setting occurs through cationic ring opening of aziridine terminated tetrahydrofuran/ethylene oxide polyethers. The polymeric chains formed are as indicated in (2).

$$\text{- }O\,([CH_2\,CH_2\,CH_2 - O]_x\,[CH_2\,CH_2\,O]_y)_n - CH_2\text{ -} \qquad (2)$$

where n dictates the length of the polyether segments and the ratio of x to y indicates the ratio of tetrahydrofuran to ethylene oxide. The repeating ether units produce an inherently hydrophilic character in the polymer which can be controlled to a degree by the ratio of x to y. More ethylene oxide (higher y value) increases the hydrophilic nature.

The hybrid material combines elements of both polyvinylsiloxane and polyether through the use of two addition reactions. The first involves a reaction akin to that in traditional polyvinylsiloxanes (equation 1). The second reaction involves addition of a vinyl terminated polyether to a siloxane as shown in (3).

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$CH_2{=}CH\ CH_2\ O\ (C_mH_{2m}\ O\ )_n - + H - Si\ (CH_3)_2 - O - Si(CH_3)_2 - O \sim + \text{Pt catalyst} \longrightarrow$

$$- (C_mH_{2m}\ O\ )_n\ O\ CH_2\ CH_2\text{-}CH_2 - Si\ (CH_3)_2 - O - Si(CH_3)_2 - O \sim \quad (3)$$

Hence, in this system the polysiloxane and polyether become linked as copolymers in one part of the setting reaction. The polymeric structure thus formed can be considered a hybrid of the polyether and polvinylsiloxane parent polymers. The properties of the hybrid will depend upon the amount of the polyether moiety included in the copolymer (ie the value of n in equation 3). Sufficient polyether is likely to have a profound effect on the surface characteristics of the polymer when compared with a traditional polyvinylsiloxane. It was the purpose of the current work to assess this by investigating the interactions of impression materials containing these different polymers with moist surfaces.

The traditional method for assessing impression accuracy and surface detail reproduction is to use a measurement of the ability to reproduce a series of grooves in a stainless steel block. This method is used in standard specification tests for impression materials [7,8]. Recording such a standardized metal block has the advantage of simplicity and convenience but cannot be considered to be realistic for considerations of the accuracy of recording moist intra-oral structures. Attempts have been made to address this issue by recording impressions of the block after first applying a fine mist of water or under water immersion [1, 4, 9, 10]. This approach yielded only qualitative results on the effect of moisture and excess moisture lying on the surface may not be a good model for representing moisture within the base structure to be recorded. The purpose of this work was to determine the accuracy with which surface detail could be recorded in impressions of gypsum blocks containing a fixed amount of water determined by the recommended powder:water ratio. The hybrid system based on polyvinylsiloxane and polyether chemistry was compared with a hydrophilic polyvinylsiloxane and a polyether material. Surface detail reproduction was evaluated by comparing the profiles of gypsum blocks with grooves of measured depth, determined with a non-contacting laser profiler, with that of a profile of the impression recorded in the test material. The hypothesis to be tested was that the hybrid material would give a surface detail reproduction capability on moist surfaces which was enhanced by the polyether component of the constituent co-polymer.

MATERIALS AND METHODS

The new hybrid polyvinylsiloxane/polyether material (SENN, GC, Japan) was compared with a hydrophilic polyvinylsiloxane impression material and a polyether material as described in table 1. The polyvinylsiloxane and polyether products were supplied and used in the Pentamix format and were mixed using a Pentamix 2 instrument (3M ESPE, Seefeld, Germany). The hybrid material was supplied in a cartridge format and was mixed and used accordingly. SENN is available as light, regular, heavy and putty consistency. The putty is a conventional polyvinylsiloxane and only the light, regular and heavy consistencies are based on the hybrid system. For the purposes of the work described here regular viscosity materials were used for all tests.

A reference block was constructed from stainless steel and machined to take seven grooves having varying and increasing nominal depth from ~ 5 µm to ~ 180 µm. The test block was reproduced in SEM grade silicone material (Microset 101FF, Microset products, U.K.) and this base impression was used to produce a series of gypsum test blocks using Fujirock (GC, Japan) at the powder/water ratio recommended by the manufacturer. Gypsum blocks were separated from the base impression and maintained in a standardized moistened state by soaking in water at 23°C for 5 minutes followed by removing from water and a 3 seconds 'blow dry' with an air syringe. Impressions of the gypsum blocks were made in one of the test materials. Three dimensional laser profiles were recorded of the gypsum test blocks and the final impressions. A freshly made gypsum block was used for each impression material and five impressions were made for each material.

Non–contacting laser profiling was performed using a Uniscan SP100 profiler (AE Optics) over a scan area of 20mm x 8mm at a scan speed of 10mm s^{-1} at 100 measurements per mm. The instrument software enabled an optical measuring cursor to be laid down on the surface to be profiled. The instrument cursor widening facility allowed determination of an average profile across the test grooves from the individual gypsum models and the final impressions. Recorded depths (Z-direction) of two of the grooves were used to determine surface detail reproduction. The deepest groove (nominal 180 µm depth) and the 6^{th} groove (nominal 20 µm depth) were chosen to test the ability to record structures of markedly different depth. The most shallow groove (nominal 5 µm depth) was not used as consistent grooves of this depth could not be reproduced in the gypsum blocks. Measurement of the distance between groove centres was used to determine accuracy in the nominally flat surface plane (x-y dimension).

RESULTS

Figures 1 and 2 show typical 3-dimensional and averaged 2-dimensional profiles (over a 1 mm cursor width). Profiles of all impressions were inverted so that the impression of grooves could be readily compared with the original.

Table 1. *Materials used in the study*

Material	*Manufacture*	*Type*	*Consistency*	*Mixing*	*Batch No*
SENN	GC Corp Japan	Hybrid polvinylsiloxane / polyether	Regular / medium	Cartridge	040311
Aquasil	Dentsply, USA	Polyvinylsiloxane	Regular / medium	Pentamix 2	0311261
Impregum	3M ESPE, Germany	Polyether	Regular / medium	Pentamix 2	163832

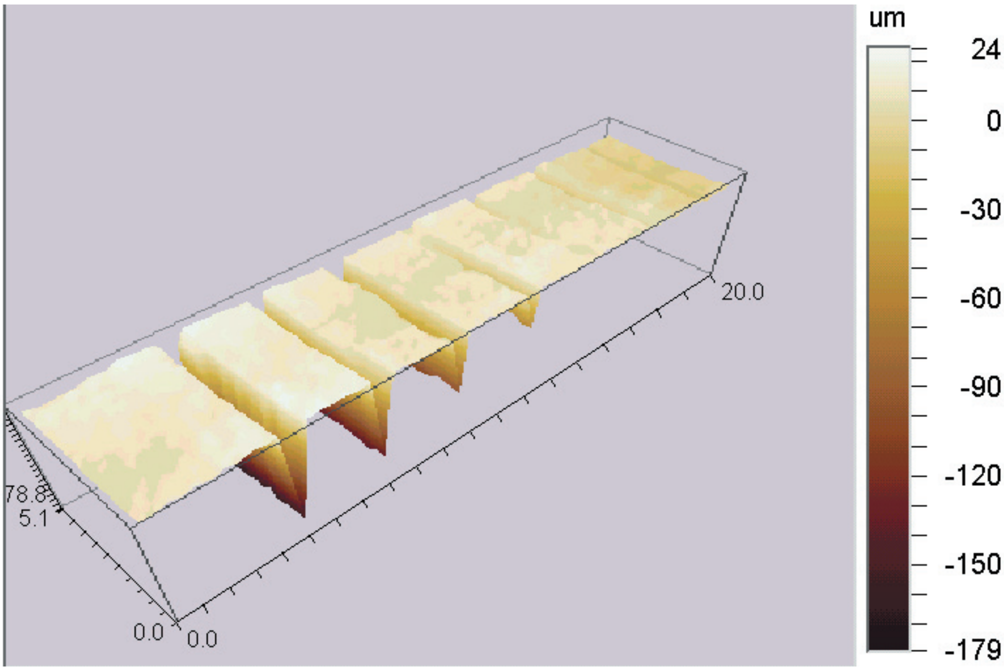

Figure 1. *Typical 3-dimensional profile used to determine surface detail reproduction.*

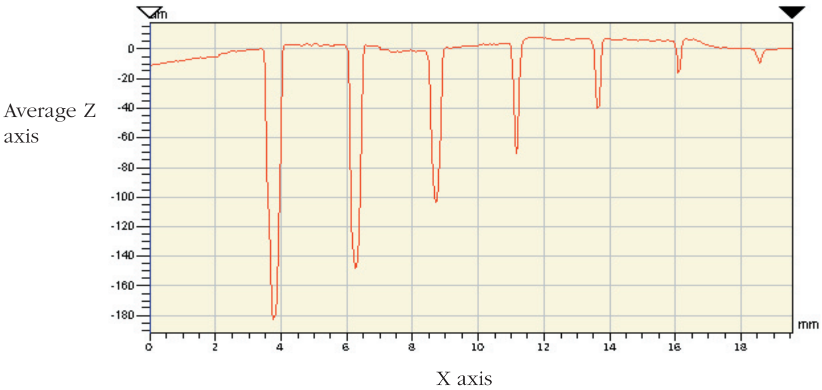

Figure 2*. Typical averaged 2-dimensional profile used to determine surface detail reproduction. Mean z values calculated for a 1 mm wide cursor. The 1st (deepest) and 6th (second shallowest) grooves were used to make direct comparisons between depth in the impression and depth on the moist block. The 7th (shallowest) groove was not used as it was not consistently reproduced in the original blocks.*

Table 2 shows the depths of the deepest and shallowest grooves in moist gypsum casts compared with the depths of equivalent grooves measured in the impression of the same cast for the hybrid impression material. Tables 3 and 4 show the equivalent data for the polyvinylsiloxane and polyether materials. The main difference between materials was in the ability to reproduce the deepest grooves. For the hybrid and polyether materials the depths of the deepest grooves were not significantly different from the originals on the moist casts, whereas for the polyvinylsioxane the deepest groove was significantly shorter in the impressions. For the most shallow groove, the depth recorded in the impression was always greater than that in the cast with the discrepancy being of the order of 10 μm for all three materials.

Table 5 shows the distance between grooves measured on both the moist casts and on the impressions. There were no significant differences in the x-y (between groove) dimension for any of the materials and in each case the dimension was the same as that in the original moist gypsum model.

Discussion

Difficulties in recording accurate surface detail in impressions are likely to be of major concern in areas where the material is required to flow into a confined space. Moist surfaces are likely to compound the problem as any hydrophobic character within the material may cause it to be repelled from such areas.

The interaction between impression materials and aqueous media may be determined by measuring contact angles

Table 2. *The depths of grooves measured in SENN from equivalent grooves in Fujirock models*

Recorded depth µm (mean and sd)		*Paired t-test*
SENN	Fujirock	
163.5 (27.3)	168.1 (5.3)	P>0.05
23.8 (4.3)	17.1 (2.7)	P<0.05

Table 3. *The depths of grooves measured in Aquasil from equivalent grooves in Fujirock models*

Recorded depth µm (mean and sd)		*Paired t-test*
Aquasil	Fujirock	
148.3 (39.2)	173.8 (2.5)	P<0.05
27.9 (5.8)	17.0 (4.0)	P<0.05

Table 4. *The depth of grooves measured in Impregum from equivalent grooves in Fujirock models*

Recorded depth µm (mean and sd)		*Paired t-test*
Impregum	Fujirock	
167.5 (6.0)	170.8 (2.6)	P>0.05
22.2 (4.0)	16.4 (4.1)	P>0.05

Table 5. *Mean distance (sd) mm (x-y plane) between grooves as recorded in the different impression materials*

Material	*distance 1 – 2**	*distance 2 – 3**	*distance 3 - 4*	*distance 4 - 5*	*distance 5 - 6*	*distance 6 - 7*
SENN	2.49 (0.08)	2.43 (0.05)	2.49 (0.03)	2.49 (0.07)	2.45 (0.07)	2.47 (0.06)
Aquasil	2.50 (0.06)	2.44 (0.07)	2.40 (0.09)	2.50 (0.14)	2.50 (0.05)	2.42 (0.07)
Impregum	2.49 (0.04)	2.41 (0.09)	2.48 (0.06)	2.52 (0.03)	2.45 (0.02)	2.43 (0.05)

* distance between lines 1 and 2 or 2 and 3 etc.

produced when water droplets are placed on samples of the impression material [2,5]. However, this is not necessarily indicative of the situation pertaining at the time when a freshly mixed impression contacts moist tissue. Recently, measurements of contact angles have been made between water and impression materials during the early stages of setting [6]. This is an improvement but does not directly lead to information on the accuracy with which surface detail can be determined.

The method employed here was designed to allow assessment of the replication of moist surfaces by constructing moist gypsum casts having grooves of different depth.

No attempt was made to relate dimensions back to the original metal block as this was thought to be inappropriate as a master model with which to judge materials used to record impressions in the oral environment. Hence, statistical comparisons are made between grooves in the impressions and grooves in the moist casts from which the impressions were made.

It was anticipated that particular difficulty would be encountered in recording the full depth of the deepest grooves on a moist gypsum cast. The hybrid and polyether materials were both able to accurately duplicate the two deepest grooves in the test cast, while the polyvinylsiloxane produced a discrepancy of greater than 20 µm in measuring this groove.

Measuring dimensional accuracy in the x-y direction did not reveal any discrepancies and suggested that such measurements are inappropriate for drawing meaningful conclusions. All elastomers appeared able to record accurate dimensions in the x-y surface plane and this finding agrees with that of Shah et al [11] who used a similar non-contacting profilometry instrument to that used here. The methods used in ISO and ADA specifications are therefore unlikely to discriminate between accurate and inaccurate materials in a manner which will help the dentist. Meaningful changes occurred only in the Z direction and the extent of these changes was related to the nature of the impression materials and to the original dimensions of the feature to be recorded (e.g. groove depth). The deeper groove gave greater absolute discrepancies of the order of ~ 20 -30 µm in magnitude for the polyvinylsiloxane. The shallow groove always resulted in an expanded dimension (approximately 10 µm) for all impression materials.

Since the polyether material is recognised as being hydrophilic it gave a good yardstick by which to judge the silicone products. The hybrid material produced a result which was very similar to that for the polyether. The polyvinylsiloxane produced greater discrepancies, particularly for the deepest grooves.

The increase in hydrophilicity seen in most modern polyvinylsiloxane materials is normally achieved by adding hydrophilic agents which are dispersed throughout the silicone paste. The additive may not be bound to the base polymer and may become phase separated either on storage or during the recording of an impression. If the surface active agent becomes concentrated at the interface between impression and the moist tissues it may result in the loss of surface detail reproduction. In the hybrid material such a separation is prevented by the use of a hybrid polymer system based on both polvinylsiloxane and polyether. The nature of the copolymer used depends critically on the ratio of polysiloxane to polyether in the elastomer (equation 3). The surface characteristics of the elastomer used in the hybrid material were similar to a polyether material in the present study. This suggests that the polyether/polysiloxane ratio used in the copolymer was sufficient to transform the surface characteristics of the polyvinylsiloxane polymer in a significant way.

In conclusion, the ability to record accurate impressions of moist surfaces varied between the test materials. All three materials were able to accurately record dimensions in the x-y plane. However, in the Z plain the hybrid material behaved in a similar fashion to the polyether. Thus the original hypothesis was supported and we are able to confirm that the ability to record groove depth on moist surfaces is greater in materials containing polyether, both alone or as part of a hybrid structure.

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