

***Title Page**

Experimental hydrophilic vinyl polysiloxane (VPS) impression materials incorporating a novel surfactant compared with commercial VPS

Dr Shahab Ud Din^{a,c}, Dr Sandra Parker^a, Prof Michael Braden^a, Dr Pete Tomlins^b, Dr Mangala Patel^a

a- Department of Oral Growth and Development (Dental Physical Sciences Unit) b- Centre for Clinical & Diagnostic Oral Sciences

Bart's and The London School of Medicine and Dentistry, Queen Mary, University of

London, UK

c- Shaheed Zulfiqar Ali Bhutto Medical University (SZABMU)/Pakistan Institute of Medical Sciences (PIMS), Islamabad, Pakistan

Corresponding Author:

Dr Shahab Ud Din

BDS(Pb), MSc(London), PhD(London)

Assistant Professor

Shaheed Zulfiqar Ali Bhutto Medical University/

Pakistan Institute of Medical Sciences (PIMS)

Islamabad, Pakistan

e-mail: drshahab728@hotmail.com

Tel: +92(0)3005640453

Abstract: Objectives

To formulate experimental hydrophilic (Exp) VPS impression materials incorporating a novel surfactant (Rhodasurf CET-2), and to compare their contact angles (CAs) with commercial materials, before/after disinfection.

Methods

CAs were measured immediately after setting and after disinfection (1%NaOCl; 30 mins and 24hrs), together with their change whilst a droplet remained on the materials surface (over 10, 20, 30 60 and 120 seconds), on three commercial (Aquasil Ultra-Monophase [Aq M], Elite HD-Monophase [Elt M], Extrude Medium-bodied [Extr M]) and four experimental (Exp I - IV) materials, using the Drop Shape Analysis 100 technique. The results were compared statistically.

Results

CAs of all experimental materials were within the range of those obtained for the commercial materials, with the exception of Exp-IV, which presented with the lowest CAs at the three time points. The control Exp-I was hydrophobic at all three time points (CAs ~100+), as was Elite. Immediately after setting, Aq M had low CAs but these increased significantly after 30 minutes of disinfection. After 24 hours' disinfection CAs of all Exp/commercial VPS increased significantly compared to immediately after setting. The CAs of droplets left on the material (120 seconds) decreased with time, even after disinfection, except for Exp-I.

Significance

The novel surfactant Rhodasurf CET-2 in Exp-III and IV, is an effective surfactant, retaining a low CA after disinfection, compared with Igepal CO-530 in Aq M. Disinfecting VPS impression materials for more than 30 minutes increases their surface CAs, and therefore prolonged disinfection periods should be avoided.

1. Introduction

The hydrophobicity/hydrophilicity of elastomeric impression materials is determined by the chemical structure and nature of these materials [1-3]. To record fine details of the oral hydrated tissues with an impression material, and to transfer these details to die/cast materials by pouring with gypsum slurries, depend on the hydrophilicity and viscosity of the impression material [3-5]. Vinyl polysiloxane (VPS) impression materials are inherently hydrophobic which makes them difficult to flow around the soft and hard tissues of the mouth, and they are not wetted by gypsum slurries. To overcome the problem of hydrophobicity, some manufacturers have incorporated non-ionic surfactants within them and have classed them as hydrophilic VPS (e.g. Aquasil) [3, 5-10].

To prevent cross-contamination, impressions should be properly disinfected after removing from the mouth, since they are always contaminated with saliva, frequently with blood and bacterial plaque. Thus they serve as a potential source of infectious microorganisms to the dental health-care personnel (DHCP), who handle the impressions [11-14]. The casts made from untreated impressions may also cause a spread of microorganisms to the DHCP [15, 16]. However, disinfecting solutions may adversely affect the dimensional stability of impression materials, particularly if they are hydrophilic. As an example, due to their hydrophilic nature, alginates, agar and polyethers are reported to be dimensionally unstable in disinfecting solutions [14, 17- 20]. Conflicting results have been reported by various researchers [20-22] who have investigated the wettability and dimensional stability of the so-called hydrophilic VPS, after disinfection.

The hydrophilicity (wettability) of impression materials can be examined by measuring the contact angle (CA) formed between the surface of the material and the curved surface of a drop of liquid on its surface [1, 8, 23, 24]. CAs can be measured by many methods for example, the

sessile drop method (Drop Shape Analysis – DSA 100, Kruss GmbH, Hamburg, Germany) and the Wilhelmy method. There is not a single, accepted standard method to measure the CA of impression materials [25]. Some researchers have used the Wilhelmy technique, for example, Lepe *et al* [26] who measured the CA of an aqueous solution of calcium sulphate on a solid sample placed in a fixed position, and following the liquid advancing on it (advancing CA), and then retreating the liquid to give the receding CA. However, most researchers have used the sessile drop method [5, 6, 27-29]. This method measures the CA by capturing the profile of a liquid placed on a solid substrate surrounded by a gas, using high resolution cameras and software. According to the sessile drop method the following can be measured:

1. Static CA
2. Dynamic, advancing and receding CAs

For VPS impression materials, static CA measurements are preferred over dynamic due to the fact that the materials are rubbers after setting. However, the surfaces of the specimens should be clean, smooth and horizontal, since this method is very sensitive to contaminated and uneven surfaces [30-32]. When water is the wetting liquid, materials with CAs higher than 90° are considered hydrophobic and indicate poor wetting, whereas materials with a CA lower than 90° are considered as hydrophilic. Materials with complete spreading of the liquid on their surface indicate CAs of 0° and possess perfect wetting properties [1, 2, 26, 33].

Different brands of commercially available impression materials have different compositions and consequently these materials have different properties, such as wettability, viscosity and compatibility with gypsum slurries. Despite the inconsistencies in the properties of these materials, most of the researchers have investigated commercial products [3, 7, 33]. Oh *et al* [5]

however, developed their own compositions of hydrophobic, as well as hydrophilic, VPS impression materials containing a surfactant (nonylphenoxy poly[ethyleneoxy] ethanol; Figure 1). They placed a sessile drop of deionized water (DW) on the surface of the material and after 2 minutes the CA was measured, using a computer aided Kruss G 10-System programme (KRUSS Company, Hamburg, Germany). They found that the surfactant reduced the CA of their VPS impression materials compared to the control. Lee *et al* [6] also developed their own compositions of VPS impression materials following a modified version of Oh *et al*'s protocol, where they varied the concentration of surfactant within the formulations (0.5%, 1.5% and 2.5%). Their CA results were similar to Oh *et al*'s results and, they further explained, that there was a strong negative correlation between the concentration of the surfactant and CA.

Both, Oh *et al* and Lee *et al* studied CAs prior to disinfection of their formulations; however, it should be noted that disinfection may adversely affect the wettability of impression materials. [2, 13, 34, 35]. It is believed that this occurs due to migration of the hydrophilic surfactant from the hydrophobic impression material and into the disinfection solution. Therefore, there appears to be a need to compare CA's of both commercial and experimental, hydrophilic VPS impression materials, before and after disinfection, in order to identify the effect of the incorporated surfactant. Hence, the research presented in this paper investigated whether experimental VPS impression materials incorporating a novel non-ionic surfactant, Rhodasurf CET-2 (ethoxylated cetyl-oleyl alcohol), recommended by Sigma-Aldrich, could retain reduced contact angles after disinfection of the materials.

The corresponding aims of this study were to compare between three hydrophilic commercial VPS impression materials and four experimental materials containing a novel surfactant, Rhodasurf CET-2, in terms of the following characteristics:

- i) The change in contact angles (surface wettability) following disinfection in 1% sodium hypochlorite solution (NaOCl) after 30 minutes and 24 hours.

Therefore, the null hypotheses can be summarised as the mean contact angle change for all materials was the same following 24 hours' disinfection, and the contact angle of a droplet placed on the surface of all materials was unchanged following a 120 second dwell time.

2. Materials and methods

Three commercial VPS impression materials were included in this study:

- (i) Aquasil Ultra Monophase (Medium-Bodied), (Aq M) from Dentsply, USA
- (ii) Elite HD Monophase (Medium-Bodied), (Elt M) from Zhermack, Italy
- (iii) Extrude (Medium-Bodied), (Extr M) from Kerr, USA.

These were classed as hydrophilic according to the literature provided by their manufacturers, and were supplied as auto-mixed cartridge delivery systems.

Four experimental, hydrophilic VPS impression materials, of known compositions, were formulated *ab initio* so that the effect of the surfactant could be assessed on their CAs. The constituents used for preparing these (Exp-I, II, III and IV; Table 1) VPS impression materials were: vinyl-terminated poly(dimethylsiloxane) (pre-polymer; molecular weight-Mw 62700; Fluorochem, UK), Aerosil R812S (filler - from Lawrence Industries, UK), Rhodasurf CET-2 (ethoxylated cetyl-oleyl alcohol non-ionic surfactant, from Rhodia, UK), and the following were purchased from Sigma Aldrich, UK, poly(methylhydrosiloxane) (Mw 2270; conventional cross-linking agent), tetra-functional (dimethylsilyl) orthosilicate (TFDMSOS; Mw 328.73; novel cross-linking agent), platinum catalyst (0.05 M), palladium (<1 μm ; scavenger). The detailed compositions of the four experimental formulations (Exp-I-IV) are given in Table 1.

Exp-I was used as a control (with no surfactant) for Exp-II, III and IV, where the main difference was the incorporation of a novel non-ionic surfactant (Rhodasurf CET-2, in increasing amounts) to form hydrophilic formulations (Exp-II, III and IV). The catalyst paste was kept the same for all the hydrophilic formulations.

1% sodium hypochlorite (NaOCl) was used as a disinfecting solution. The disinfecting solution was supplied as 14% NaOCl, by Fisher Scientific UK Ltd, which was diluted to 1% NaOCl by mixing 100 ml of 14% NaOCl with 1300 ml of deionised water (DW).

Sample preparation for testing of CAs

Samples were prepared in rectangular stainless steel metal moulds measuring $40 \times 10 \times 1 \text{ mm}^3$, in a temperature controlled environment ($23^\circ\text{C} \pm 1^\circ\text{C}$). An acetate sheet was placed on top of a metal plate, on to which the stainless-steel mould was positioned. The base and catalyst pastes (pre-packed in a double barrel cartridge), were mixed using an auto-mixing syringe and extruded directly into the mould cavity. Another acetate sheet was placed on top followed by another metal plate. Then the whole assembly was placed under a hand-operated hydraulic press (MESTRA MOD-030350, Talleres Mestraitua, S.L) and the pressure was slowly increased to 100 bars in order to distribute the material evenly in the mould cavity, flush out excess material and expel air bubbles [36]. The materials were allowed to set at $23^\circ\text{C} (\pm 1^\circ\text{C})$. (i) for the time specified by manufactures for commercial materials and (ii) for 4 to 11 minutes depending on the overall composition of the experimental materials.

CA measurement using the Drop Shape Analysis (DSA)100 device

The Drop Shape Analysis (DSA)100 technique was used to measure the static (and dynamic) CA of commercial and experimental VPS impression materials.

The DSA 100 equipment was calibrated using the Young-Laplace method. As soon as a sample had set (n=10 per material) it was carefully placed on the sample table and the video recorder with a charge-coupled device (CCD) camera was started. A droplet (~1.5 μ L) of DW was placed carefully onto the surface of the sample using a Gastight #1001 syringe (Hamilton Bonaduz AG, Switzerland - accuracy to 0.01 ml). The dynamic CAs (spreading of the droplets) were measured by capturing the profile of DW on the material, at 10, 30, 60 and 120 seconds (ie over two minutes) [6, 29, 37], using a mounted camera and processing using the software (DSA1).

CAs were measured 10 seconds after placing the drop on each sample i) immediately after setting [3, 5, 6, 26]; ii) after 30 minutes in disinfecting solution [26, 38]; iii) after 24 hours disinfection [26], in order to compare the effect of the hydrophilic agent in the materials.

Statistical analysis

Statistical analysis was performed using SPSS Statistics for Windows (Version 22.0. Armonk, NY: IBM Corp). The null hypothesis that the mean contact angle change for all materials was the same following 24 hours' disinfection was tested using one-way Analysis of Variance (ANOVA). Tukey's Honest Significant Difference (HSD) test was used for post-hoc analysis to perform multi-way comparisons of the change in mean contact angle amongst all materials.

Furthermore, one-way ANOVA was also used to test the null hypothesis that the contact angle of a droplet placed on the surface of all materials was unchanged following a 120 second dwell

time. Tukey's HSD test was used for post-hoc analysis of inter-material mean differences. A Bonferroni factor of two was used to account for multiple hypotheses and thereby the significance level for this study was set at $\alpha=0.025$.

3. Results

Table 1 gives the formulations of all experimental materials, Exp-I (control with no surfactant), and Exp-II, III and IV with increasing amounts of surfactant. Table 2 presents the setting times of commercial and experimental VPS impression materials. The setting times of the experimental materials containing the novel surfactant increased with the amount incorporated. The setting time of the control experimental VPS (no surfactant) material was comparable to the other commercial materials.

Figure 2 shows the baseline CAs (10 s) for all experimental and commercial VPS immediately i) after setting, ii) after 30 minutes of disinfection and iii) after 24 hours of disinfection. Generally, CAs for all materials increased after 30 minutes (with the exception of the control) and 24 hours of disinfection. The CAs for all experimental materials were similar and within the range of those obtained for the commercial materials, with the exception of Exp-IV, which appeared to present with the lowest CA values at the three test periods. Immediately after setting, the CAs for Exp-IV were significantly different (lower) than those obtained for all other experimental and commercial materials. The control experimental material was hydrophobic at all three test periods (CAs ~100+), as was Elt M (commercial material).

Figure 3 shows the mean overall change in CA following 24 hours of disinfection, and the corresponding statistical analysis data is summarised in Table 3. Similar trends were found for the mean overall change in CA following 30 minutes of disinfection, but to a lesser extent.

Significant differences were found for the post-disinfection CA change. The change in CAs was highest (up to 60°) for Aq M and this was significantly different to the other materials. The change in CAs was similar for Elt M, Extr M, Exp-II, III and IV. Exp-I (control) showed no change following 24 hours' disinfection period. Therefore, the null hypothesis 'the mean contact angle change for all materials was the same following 24 hours' disinfection' is only retained for Exp-I.

Figure 4 shows the change in mean CA over a 120s, for 'droplet spreading' period, after 24 hours' disinfection, and the corresponding statistical analysis data is summarised in Table 4. Exp-III and IV exhibited the smallest decrease in CA compared to all other materials (excluding the control). Notably, Exp-II was statistically similar to all of the commercial materials. Aq M, Elt M, Extr M and Exp-II exhibited the greatest decrease in mean CA following a 120 second droplet dwell time. The change in all of these CAs was also statistically similar. Exp-III and IV exhibited the least CA decrease during the 120s droplet dwell time and these were statistically similar. Exp-I showed the smallest decrease in CA over the 120s dwell time, and it was statistically different to all other materials, except Exp-IV (Tukey's HSD). Therefore, the null hypothesis 'the contact angle of a droplet placed on the surface of all materials was unchanged following a 120 second dwell time'.

Figure 5 is a typical plot showing the mean change in CA over two minutes after 24 hours' disinfection (measured at 10, 30, 60 and 120 seconds). Similar trends were obtained for immediately after setting and 30 minutes after disinfection, but with lower CAs as expected. After 24 hours' disinfection, with the exception of Exp-I, the CAs on all other materials were still decreasing with time.

5. Discussion

In this study, the Drop Shape Analysis [DSA]100 technique was used to measure the change in CAs immediately after setting, 30 minutes after disinfection and 24 hours' disinfection (as reported in the literature; eg 26 and 38), on three commercial and four experimental materials, with the latter three incorporating a novel surfactant (Rhodasurf CET-2) in varying concentrations. Although CAs of unset materials have been recorded to be lower than the equivalent set material (3, 27), it should be noted that in this study all CA measurements were made on set materials. Hence, these results can be related to wetting of the impression material surface by gypsum slurries during model/die casting.

Surprisingly, Elt M (commercial material) presented with high CAs (~100+) after the three test periods and yet it is classed as a hydrophilic impression material. The control experimental material (Exp-I) did not contain any surfactant and, as expected, was hydrophobic at all three test periods, as reflected by its high CAs (Fig 2). Exp-I also showed the smallest change in CAs following 24 hours of disinfection (Fig 3) and over the 120s dwell time (Fig 4). This was expected, since there was no surfactant present that could leach from the material.

Generally, CAs for all materials increased after 30 minutes (with the exception of the control) and 24 hours' immersion in 1% NaOCl disinfecting solution, particularly for Aq M (up to 60°; Figure 3) compared to those obtained immediately after setting. These results suggest that the surfactants leached from the VPS materials, during the disinfecting process, thus increasing the CAs of DW on their surface. It is not known what surfactant Elt M and Extr M contain, whereas Aq M contains Igepal CO-530, (nonylphenoxy poly [ethyleneoxy] ethanol) [39], a non-ionic (soluble in water) and widely used detergent. The hydrophilic group of the non-ionic surfactant is a polymerised alkene oxide (water soluble polyether; Fig 1). It is apparent from its chemical

structure that it does not contain a double bond and so it is assumed that it is not chemically bonded to the silicone polymer matrix. From the limited information obtained from the Materials Safety Data Sheets (MSDS) on Rhodasurf CET-2 and Igepal CO-530, it seems that the latter is more soluble in water and has a lower molecular weight than the former. Therefore, these factors, and due to the fact that it is not bonded to the polymer matrix, could explain why Igepal CO-530 leached with ease during disinfection of Aq M at different time points, thus causing an increase in CAs.

Kim *et al.* [38] reported that the CAs of six commercial hydrophilic VPS impression increased after disinfecting them in 0.5% NaOCl. On comparing their CAs with those obtained in this study, Aq M and Extr M showed similar CAs to Fusion and Aquasil Ultra after disinfection, and lower CAs compared to Genie, Imprint II, Twinz and Perfect-F. Lepe *et al.*, [26] have suggested that the surfactants (e.g. Igepal) added to hydrophilic VPS impression materials may be washed out during the disinfection period. Balkenhol *et al.* [40] identified surfactant inside droplets on the surface of hydrophilic VPS, thus demonstrating its leaching. Hence, leaving impressions in disinfecting solution for longer periods (than specified by the manufacturers) will result in further leaching of the surfactant. The results from the current study agree with this finding, where more surfactant was leached after 24 hours in disinfecting solution.

Michalakis *et al.* [33] measured the CAs of elastomeric impression materials prior to disinfection (30 seconds), including Aquasil (medium-bodied) and a polyether. Extr M, Exp-II, III and IV showed lower CAs than those obtained for their materials, with the exception of polyether, which had lower CAs than Extr M and Exp-II. Polyethers are inherently hydrophilic materials; therefore, interestingly in the current study, Exp-III and IV had CAs in the range of those obtained for hydrophilic polyethers.

From Figure 2 it is evident that Exp-IV appeared to present with the lowest CAs at the three test periods and, immediately after setting, the CAs were significantly different (lower) than those obtained for all other experimental and commercial materials. This experimental material contained a higher percentage (3wt%) of surfactant compared with Exp-II and III (2 and 2.5wt%). Also, it is transpired that Exp-III and IV exhibited the smallest change in CAs after 24 hours of disinfection compared to all other materials (excluding the control; Figure 3), and the least change in CAs during the 120s droplet dwell time (Figure 4). Hence, the low CAs obtained for these experimental formulations are encouraging and suggest that Rhodasurf CET-2 is a more effective surfactant compared to, for example nonylphenoxy poly(ethyleneoxy), even after 24 hours of disinfection; it did not leach readily into the disinfecting solution, particularly when incorporated at a higher percentage (3wt%).

Rhodasurf CET-2, (ethoxylated cetyl-oleyl alcohol), a non-ionic surfactant, is a mixture of ethoxylated cetyl and ethoxylated oleyl alcohols as shown in Figs 6a and 6b respectively. Ethoxylated oleyl alcohol contains a double bond in its chemical structure (Fig 6b). It could be possible that this double bond was activated during polymerisation and may have taken part in the cross-linking reaction, thus increasing the setting time of the materials, particularly when incorporated at higher concentrations (Exp-III=2.5wt% and Exp-IV= 3wt%). Also, it can be assumed that since Exp-III and Exp-IV exhibited the smallest decrease in CAs (change over a 120s of a 'droplet spreading' period) compared to all other materials (excluding the control), after 24 hours of disinfection (Figs 4 and 5), some of the surfactant was possibly cross-linked to the Exp VPS materials, thus the latter's surfaces remained hydrophilic. As the surfactant became part of the polymer matrix, some of it was prevented from leaching out during the disinfecting period, which contributed to the lower CAs obtained. An alternative explanation points to strong intermolecular (Van der Waals) forces between the cetyl/olely parts of the molecules, or

molecular entanglement, due to its chemical structure (long cetyl and oleyl chains), which limited leaching of the surfactant.

Lee *et al.* [6] developed their own VPS formulations with different concentrations of the surfactant, nonylphenoxy poly(ethyleneoxy) ethanol, and measured CAs using the DSA technique. Exp-I had similar CAs (93°) to Lee *et al.*'s control (without surfactant) (104°) at two minutes, while Aq M, Exp-III and IV of the current study demonstrated lower CAs than all of their formulations. Exp-III gave a lower CA (23.8°) compared to Lee *et al.*'s formulation (CA = 33.9°), where both compositions contained the same amount of surfactant (2.5%) and the CAs were measured at the same time points (over 2 minutes). This again suggests that Rhodasurf CET-2 is a better surfactant than nonylphenoxy poly(ethyleneoxy) ethanol.

All experimental and commercial materials displayed higher CAs at 10 seconds after setting, which decreased after two minutes, to different extents (Figure 5). This data indicates that with time the surfactant travels to the surface of the material, thus reducing its CA. This theory is supported by Grundke *et al.* [3], who studied CAs of VPS, condensation silicone and polyethers also using the DSA technique. The latter authors suggested that the originally hydrophobic surface of the VPS material became hydrophilic due to migration of the surfactant from the deeper layers of the sample to solid-liquid interface. The decrease in surface tension of the water droplet was due to the dissolution of the surfactant in water and its diffusion to the liquid-air interface (3, 7, 29). Balkenhol *et al.* [40] also reported that extrinsic surfactants added to VPS materials reduce the surface tension on the liquid in contact, but do not increase the wettability of the materials' surface. Additional effects may be due to the transfer of surfactant molecules onto the un-wetted surface (solid-vapour interface) in front of the advancing liquid, leading to an increase in the wetting characteristics of the material [3, 41]. From Figure 5, for Exp-IV

which presented with the lowest CA, it appears that even after 24 hours of disinfection the surfactant was still present at the surface, and had not leached. This could be due to the assumption made earlier that the surfactant was retained in the material via bonding (chemical or molecular).

Hence, to overcome the inherently hydrophobic problem of VPS impression materials, for example compatibility with gypsum slurries, manufacturers have incorporated non-ionic surfactants within them and have classed them as hydrophilic VPS [3, 5-10]. The results of this study have shown that surfactants readily leach with time [40], in disinfecting solutions, and so wetting by gypsum slurries during casting of the model/die may still be an issue with some VPS materials, leading to defects in the model/die and inaccuracies in the final product. However, it could be assumed that Rhodasurf CET-2 is a more suitable surfactant compared to nonylphenoxy poly(ethyleneoxy) ethanol, based on the promising findings of this work. It did not leach readily in disinfecting solution and, Exp-IV particularly, retained low CAs throughout the experiments. It should be noted that there is no literature available on the use of this surfactant in impression materials, and clearly this area merits further research.

Conclusions

- The novel surfactant Rhodasurf CET-2 in Exp-III and IV, appears to be an effective surfactant, possibly due to molecular entanglement/cross-linking, since low contact angles were retained after disinfection, compared with Igepal CO-530 in Aq M.
- The amount of surfactant leaching with time, from hydrophilic VPS impression materials, varies according to its chemical structure and solubility in water.

- Disinfecting VPS impression materials for more than 30 minutes increases their surface CAs, which could affect their compatibility with gypsum slurries during casting of the model/die; therefore, manufacturers' guidelines should be followed and prolonged disinfection periods should be avoided.

Acknowledgements

This PhD was partially funded by National University of Sciences & Technology (NUST), Islamabad, Pakistan, and therefore we thank the authorities at NUST.

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Figure 1

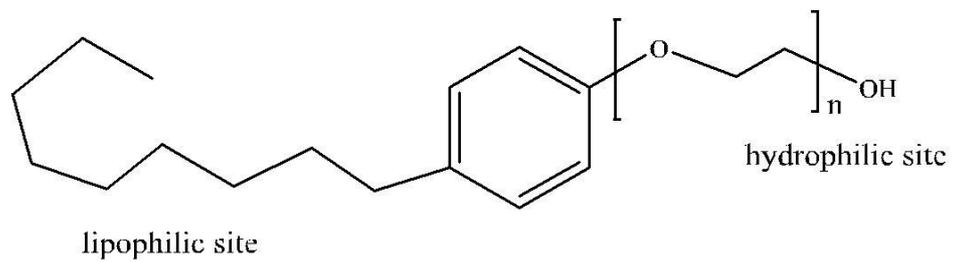


Fig 1: Structural formula of nonylphenoxy poly(ethyleneoxy) ethanol (non-ionic surfactant) showing hydrophilic (ethyleneoxy) and lipophilic (nonylphenoxy) sites.

Figure 2

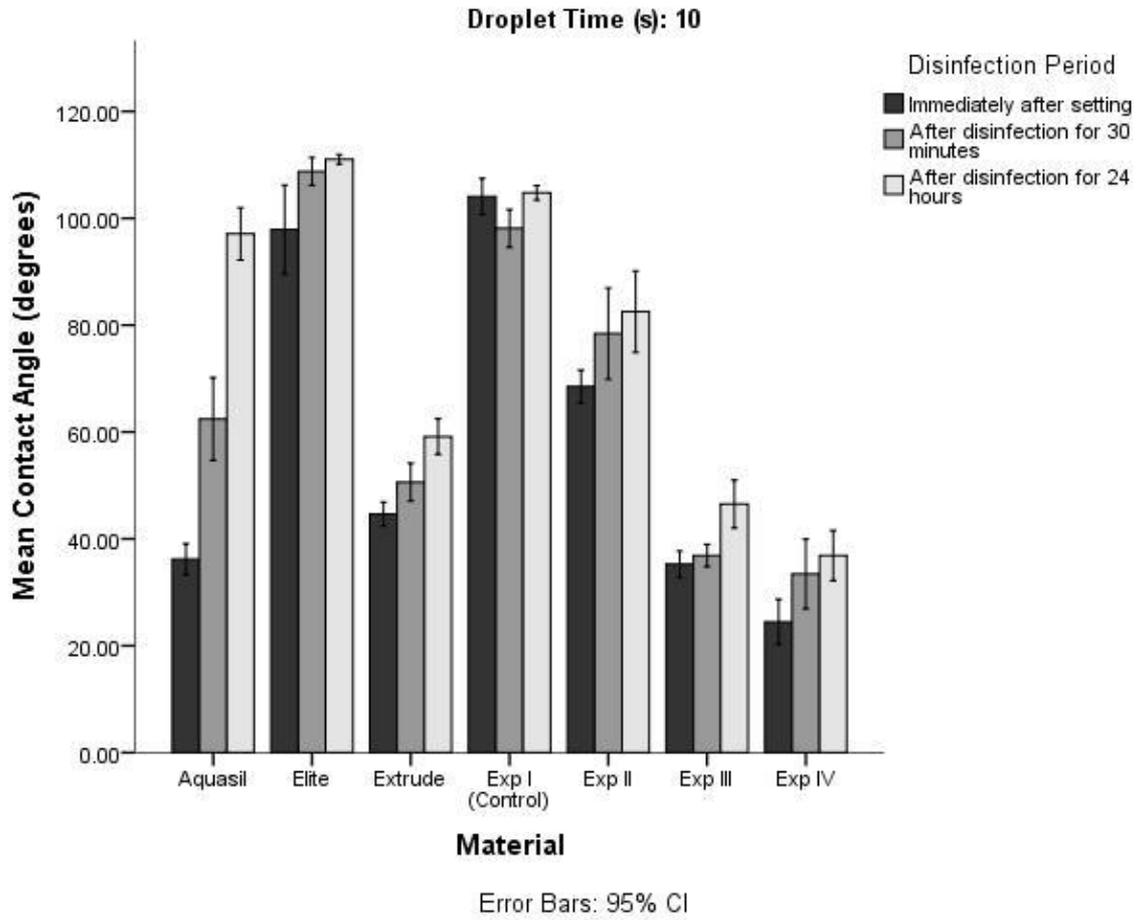


Fig 2: Comparison of the baseline contact angle immediately after setting, after 30 minutes of disinfection and after 24 hours of disinfection.

Figure 3

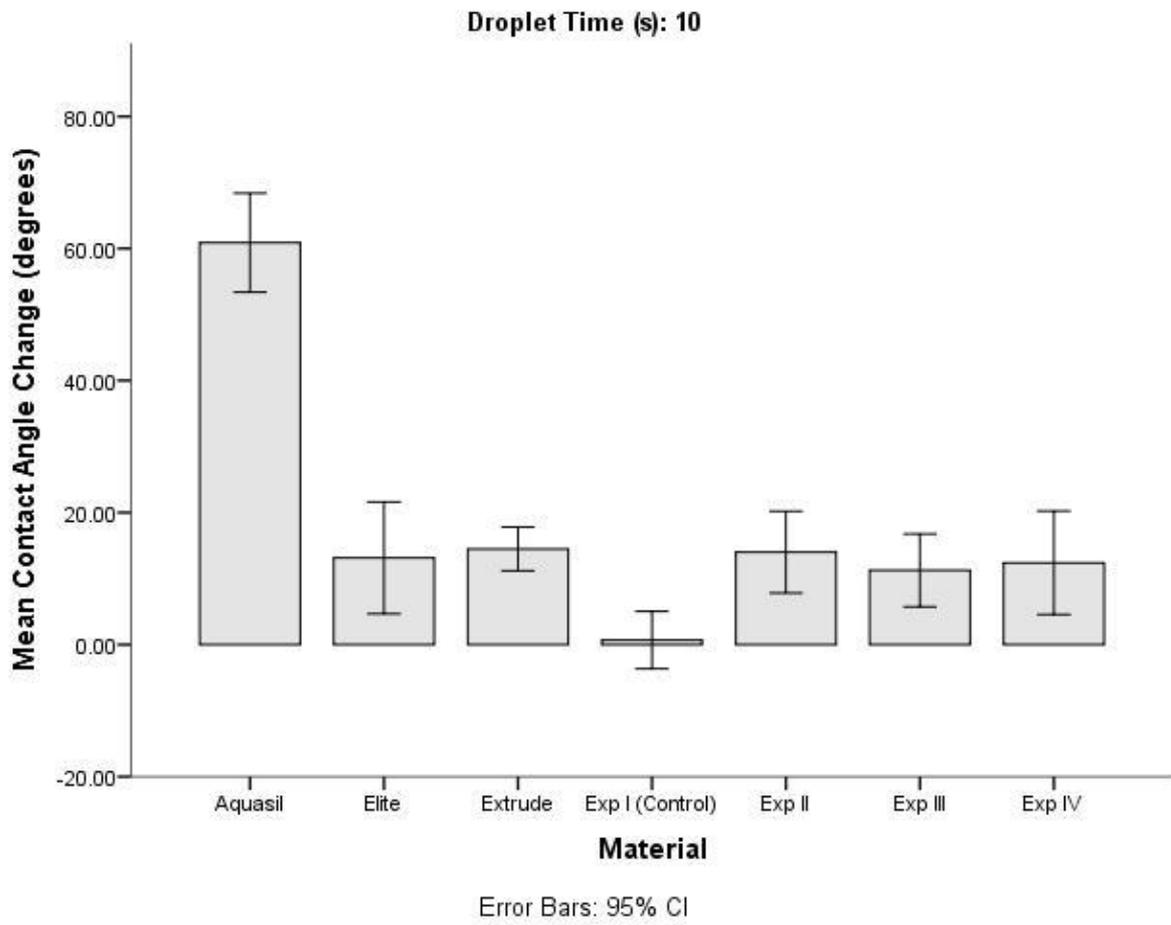


Fig 3: Change in contact angle following 24 hours of disinfection.

Figure 4

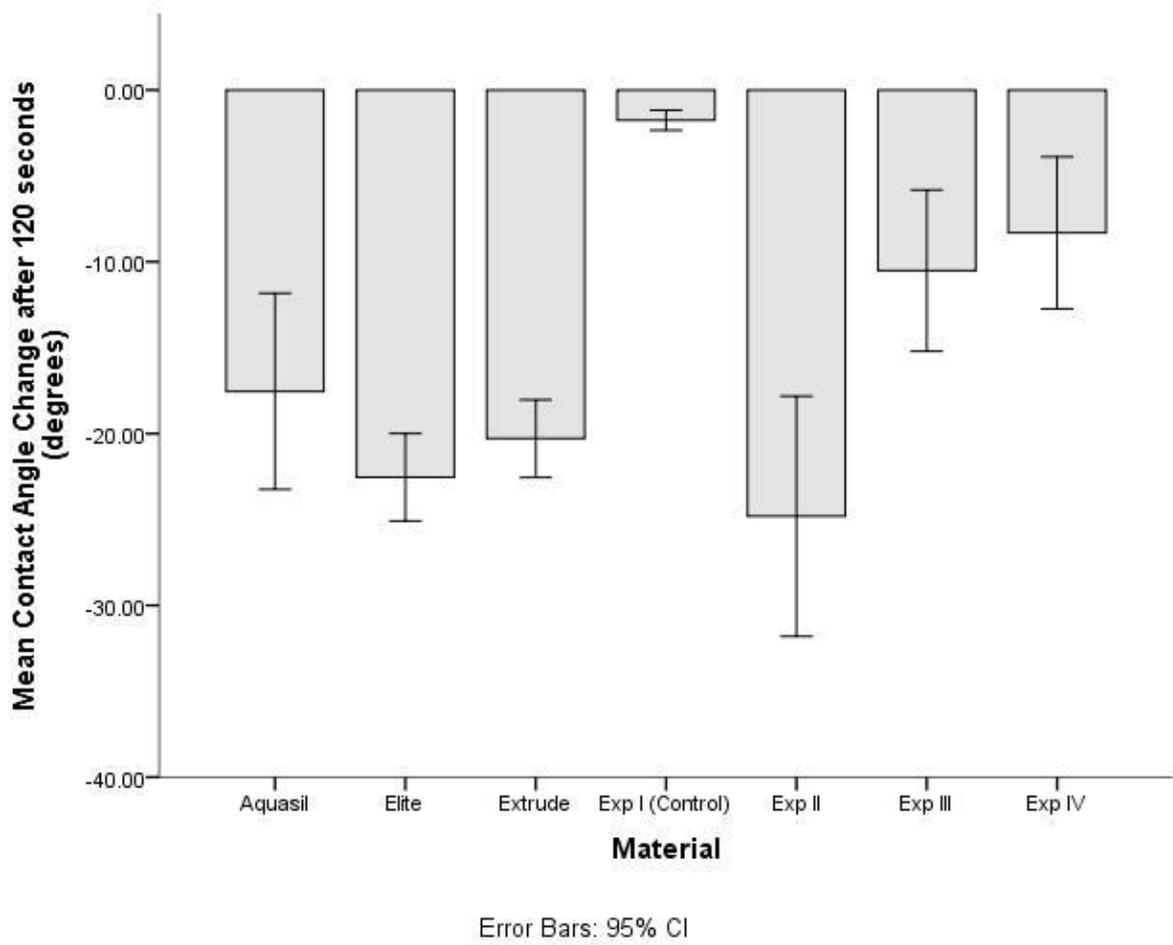


Fig 4: Change in mean contact angle over a 120s ‘droplet spreading’ period (after 24 hours of disinfection).

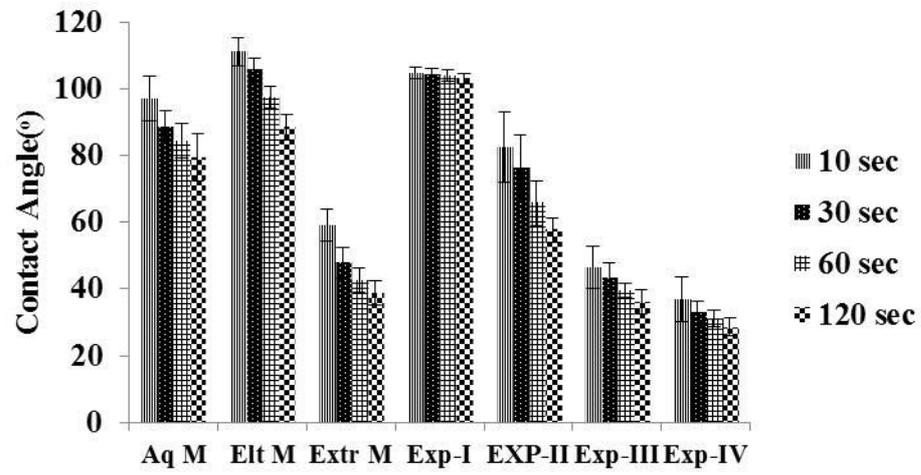


Fig 5: Mean (\pm standard errors; n=10) CAs of Exp and commercial VPS after DI in 1% NaOCl for 24 hours.

Table 2**Table 2:** Setting time (sec) of Exp and Comml VPS impression materials at 23 °C± 1°C

Simples	Aq M	Elt M	Extr M	Exp-I	Exp-II	Exp-III	Exp-IV
S1	480	324	498	450	594	660	684
S2	468	318	480	456	600	612	660
S3	432	312	468	474	600	636	672
S4	462	324	492	474	576	612	684
S5	456	324	474	480	588	636	654
Average	460	320	482	467	592	631	671
SD	18	5	12	13	10	20	14

Table 3: Mean contact angle change for all materials following 24 hours of disinfection with corresponding standard errors (S.E.)

Material	Mean CA Change (deg.)	S.E.
Aquasil	60.9 ^a	3.3
Elite	13.2 ^b	3.7
Extrude	14.5 ^b	1.5
Exp I (Control)	.7 ^c	1.9
Exp II	14.0 ^b	2.7
Exp III	11.3 ^b	2.4
Exp IV	12.4 ^b	3.5

^a Statistically significant mean change in CA compared to all other materials ($p < 0.025$, Tukey's HSD)

^b No statistically significant difference between materials ($p > 0.025$, Tukey's HSD)

^c Statistically significant difference between control and all other materials ($p < 0.025$, Tukey's HSD)

Table 4: Mean contact angle change for a droplet placed on each material for a 120 second dwell time with corresponding standard errors (S.E.)

Material	Mean CA Change (deg.)	S.E.
Aquasil	-17.5 ^a	2.5
Elite	-22.5 ^a	1.1
Extrude	-20.2 ^a	1.0
Exp I (Control)	-1.8 ^c	0.3
Exp II	-24.8 ^a	3.1
Exp III	-10.5 ^b	2.1
Exp IV	-8.3 ^b	2.0

^a No statistically significant difference in the mean CA change between Aquasil, Elite, Extrude and Exp II ($p > 0.025$, Tukey's HSD), but statistically different to all other materials

^b No statistically significant difference in mean CA change between between Exp III and Exp IV ($p > 0.025$, Tukey's HSD) but these were statistically different to all other materials

^c Statistically significant difference between control and all other materials ($p < 0.025$, Tukey's HSD)