

Article

Reproduction of Fine Details and Compatibility of Vinyl Polysiloxane Impression Materials

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Abstract: The purpose of this study was to develop novel experimental (Exp) vinyl polysiloxane (VPS) impression materials (ab initio) and to evaluate their reproduction of fine details and compatibility with pouring materials. The Exp materials were compared with three commercial VPSs (Aquasil Ultra Monophase (Aq M), Extrude Medium-Bodied (Extr M), Elite HD Monophase (Elt M)) under dry, moist and wet conditions. Five VPSs (Exp-I-V) were developed, out of which Exp-I and II were hydrophobic while Exp-III, IV and V were hydrophilic. In the current study, Exp-II is the control for Exp-III, IV and V. Exp-I was the control for Exp-II, in which tear strength of the VPS was improved by adding a novel cross-linking agent. This part of the study has already been published by the authors. Under dry conditions, all commercial and Exp materials reproduced the 20 µm line satisfactorily. Under moist conditions, all commercial and some of the Exp (III, IV and V) materials reproduced the 20 µm line satisfactorily, with the exception of Exp-I and II. Under wet conditions, Aq M, Extr M and Exp-IV and V reproduced the continuous line, while Elt M and Exp-I, II and III failed to produce the line. For compatibility, all commercial and Exp VPSs, under dry conditions, reproduced the 50 µm line on the cast. Under moist conditions, Elt M and Exp-I and II did not record the line, while Aq M, Extr M and Exp-III, IV and V reproduced this line. Under wet conditions, Aq M, Extr M and Exp-IV and V reproduced the continuous line of 50 µm, while Elt M and Exp-I, II and III failed to record this line. Performance of the materials depends on the type and amount of surfactant incorporated. These data provide useful knowledge for clinicians on recording and pouring impressions with greater accuracy of reproduction of fine details and compatibility with cast/die materials.

Keywords: impression materials; elastomers; vinyl polysiloxane; hydrophilic; detail reproduction; compatibility; gypsum



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1. Introduction

Impression recording is the primary step in the manufacturing of an indirect prosthetic restoration. Accuracy of the impression in terms of surface detail reproduction and pouring with gypsum are the important steps in successful fabrication of prosthesis [1]. The fit and retention of a denture is dependent on the intimate adaptation of the base of the denture with the underlying oral tissues. The denture base is a replicate of the cast which is an ultimate production of the impression [2]. Elastomeric impression materials (i.e., polysulfides, condensation silicone, VPS and polyethers) have superior properties compared to hydrocolloids and non-elastic impression materials [3]. Among all elastomeric impression materials, the VPS impression materials are the best materials available to dentists due

to their superior elastic recovery [4,5] and excellent dimensional stability [6,7]. These are supplied in four different consistencies (putty, heavy-bodied, medium-bodied and light-bodied). Impressions can be recorded using two different techniques (monophase and dual phase techniques). In the monophase technique, a single-step procedure is followed using a medium-bodied impression material, while in the dual-phase technique, different viscosities, such as the putty and light-bodied materials, are used in a one or two step procedure. Both of the techniques can record detailed impressions accurately, however, it is the medium-bodied/light-bodied materials which come in contact with the tissues and record the fine details [1]. The presence of saliva, moisture, blood and temperature in the oral cavity complicates impression recording [1]. Elastomeric impression materials can record surface details more accurately *in vitro*, compared to the oral hydrated tissues *in vivo*, because of the inherent hydrophobic nature exhibited by some of these impression materials [8,9]. Conflicting results have been reported by various researchers [10,11] who have investigated the reproduction of detail and dimensional changes of these materials. Pratten and Craig [12] investigated the wettability and castability of four types of elastomeric impression materials: polyether (Impregum F), polysulphide (Permlastic), hydrophilic VPS (Express-H) and conventional VPS (President and Express), by taking impressions of a machined brass die and then pouring the impressions in die stones. Their results showed that the wettability of hydrophilic VPS was similar to that of polyether impression materials, and the CA of these materials was half that of conventional hydrophobic VPS. Polysulphides demonstrated CAs as being lower than conventional VPS but higher than polyether and hydrophilic VPS. The hydrophilic VPS produced casts with a smaller number of voids, while the conventional VPS produced casts with a greater number of voids.

Dental stone, plaster, epoxy resin and refractory materials are used to pour dental impressions to produce the cast for further processing in the laboratory. The selection of the pouring material depends on the impression material used for recording the impression and the purpose for which the cast/die is to be used. Impressions taken in hydrocolloids can only be poured using gypsum products, while elastomeric impressions can be poured with gypsum or epoxy die materials [13,14]. Literature reveals that hydrophobic VPS impression materials are more compatible with epoxy resins compared to gypsum products [15–18]. On the contrary, the polyethers and reformulated VPS impression materials show better compatibility with gypsum products compared to conventional VPS, condensation silicone and polysulphides [12,19–21].

Various investigators have studied the compatibility and reproducibility of cast/die materials with elastomeric impression materials [3]. Derrien et al. [22] evaluated the compatibility of dental stone (Fuji rock), epoxy resin die material (Epoxydent) and polyurethane resin (Steady-plast) with two VPS impression materials (ProvilMedium Hydroactive and P-soft). Fifteen impressions (five for each pouring material) of a disc-shaped calibration model with etched grooves were recorded using acrylic resinous custom trays. After pouring with die materials, they were examined using SEM (JSM 35-type, JEOL, Tokyo, Japan) and two-dimensional (2-D) profilometer (Talysurf 5, Rank Taylor Hobson, Leicester, UK). The SEM photos and analysis of profilometric tracings confirmed that gypsum stone was not able to reproduce details smaller than 20 μm , due to its crystalline structure, whereas epoxy resin and polyurethane resin accurately reproduced details down to 1 to 2 μm . The details recorded with epoxy and polyurethane resins were comparable, while the details recorded with gypsum stone were of inferior quality. However, the dimensional accuracy of resinous die/cast materials is critical because of their polymerisation shrinkage, due to which the gypsum products are preferred and have been widely used for decades. The die/cast materials used for pouring impressions taken by elastomeric impression should fulfil the requirements of ISO4823 [23].

Published research has focussed on studying different properties of commercial impression materials without addressing the effects that the various components have on them. This is due to the manufacturers and suppliers not providing the exact composition of their VPS impression materials. It is critical to have such information since each component

- In the current study, Exp-II was the control for Exp-III, IV and V. Exp-I was the control for Exp-II, in which the tear strength of the VPS was improved by adding a novel cross-linking agent (tetra-functional dimethylsilyl orthosilicate). This part of the study has already been published by the authors [24].

2.3. Methods for Pilot Studies

Pilot studies were based on Lee et al.'s [25] protocol, who basically modified Oh et al.'s [26] methods for developing novel VPS impression materials.

The components used in this study were not exactly the same as Lee et al. and Oh et al.'s formulations, with differences in the fillers (Aerosil R 812 S) and surfactants (Ethoxylated ce-tyl-oleyl alcohol) used; however, some of the basic components (prepolymer and conventional cross-linking agent) were similar. Among the advanced formulations of the resulting medium bodied VPS, five formulations (Exp-I, II, III, IV and V) were chosen as the most suitable compositions. The methods for preparing them are described below.

2.3.1. Preparation and Dispensing of the Final Novel Exp VPS

Five Exp formulations (Exp-I, II, III, IV and V) were prepared in the form of base paste and catalyst pastes.

Preparation of Exp-I (Hydrophobic VPS)

Control Novel Base Paste

Vinyl-terminated poly(dimethylsiloxane) pre-polymer (Mw 62,700; 39.90 wt%) and poly (methylhydrosiloxane) cross-linking agent (~Mw 2270; 1.10 wt%) were mixed (5 min) using an electric hand mixer (Kenwood, kMix, Havant, UK). Then, Aerosil R812S (9%) was added (as a filler) and mixed with a pestle and mortar until a uniform paste was formed (~5 min; Table 2, Exp-I, base paste). Finally, the mixture was blended with the electric mixer for a further 10 min to allow even distribution of the filler in the paste. All components were weighed on a four-figure balance (Mettler, Toledo Ltd., Model AG204, Kent, UK).

Control Novel Catalyst Paste

Vinyl-terminated poly(dimethylsiloxane) prepolymer (Mw 62,700; 40.72%), platinum catalyst (0.06%) and palladium (0.23%) were mixed (5 min) with the electric hand mixer. It was assumed that the catalyst had dispersed in the mix after this mixing procedure, and this method was followed throughout all of the experiments to provide consistency and standardised mixing. Then, filler (Aerosil R812S; 9%) was added and mixed with the pestle and mortar until a uniform paste was formed. Finally, the mixture was mixed further with the electric hand mixer for another 10 min (Table 2; Exp I, catalyst paste).

As there was no surfactant added to both of these pastes (base and catalyst), these were termed as Exp hydrophobic VPS impression material.

Preparation of Exp-II (Hydrophobic VPS with Tetra-Functional Dimethylsilyl Orthosilicate (TFDMSOS))

TFDMSOS (0.33 wt%) was incorporated into the control novel base paste as an additional cross-linking agent, prior to the addition and mixing of the filler, and the method for control novel base paste of Exp-I was followed. The amount of conventional cross-linking agent, poly(methylhydrosiloxane), was reduced (from 1.10 wt% to 0.77 wt%) to retain the 1:1 ratio of vinyl to silane groups (Table 2, Exp II, base paste).

The catalyst paste for Exp-II was the same as for Exp-I (Table 2 catalyst paste).

Preparation of Novel Exp Hydrophilic VPS (Exp-III, IV and V)

A non-ionic novel surfactant, Rhodasurf CET-2 (Ethoxylated cetyl-oleyl alcohol) (2%-Exp-III, 2.5%-Exp-IV and 3%-Exp-V), was incorporated in the novel base paste of Exp-II prior to adding the filler, and the method for Exp-I and II (base paste) was followed (Table 2; Exp III, IV, V, base paste).

The catalyst pastes for Exp-III, IV and V contained vinyl-terminated poly(dimethylsiloxane) prepolymer (Mw 62,700; 39.51 wt%), platinum catalyst (1.27 wt%), palladium (0.22 wt%) and the filler (Aerosil R812S; 9 wt%), as shown in Table 2, and the method for Exp-I and II (catalyst paste) was followed. Note, due to the addition of a surfactant in the base paste, the quantities of the constituents in the catalyst paste had to be modified to ensure setting of the materials. The novel Exp materials (Exp I-V) developed in this study were confirmed as being VPS, following analyses of their Fourier Transform infrared spectroscopy (FTIR) spectra and contact angle measurements using Drop Shape Analysis (DSA). Both studies were published by the authors [16,17,24] and have contributed to the discussion in this contribution.

2.4. Dispensing of the Novel Exp VPS

Both pastes (base and catalyst) for Exp VPS impression materials were packed into separate compartments of an auto-mixing cartridge and stored at 4 ± 2 °C, to prevent any premature polymerisation or degradation of the platinum catalyst. The pastes were extruded from mixing tips using a conventional delivery gun.

2.5. Surface Detail Reproduction Test

Impressions ($n = 5$ per material; specimens measuring diameter = 30 mm and thickness = 3.5 mm) were taken of the scribed lines on the steel test block (Figure 1) for the measurement of the surface details' reproduction [23], under three different conditions: dry, moist and wet.

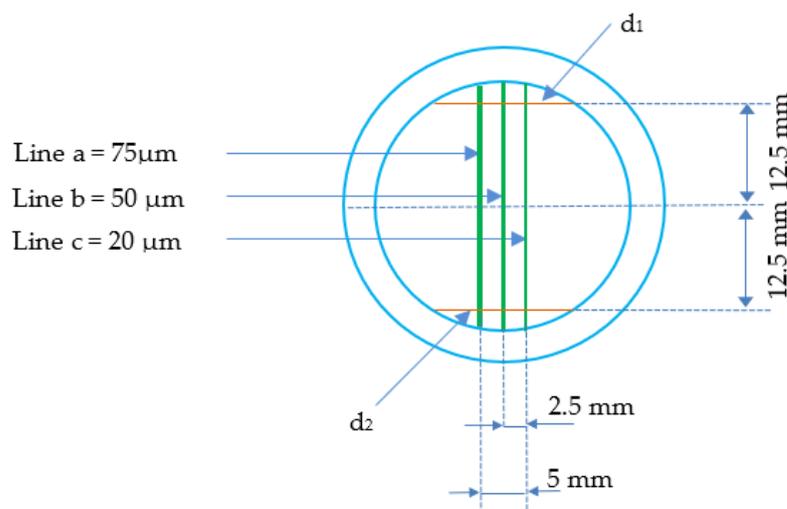


Figure 1. Test block used for detail reproduction and compatibility with gypsum of commercial and Exp VPS impression materials ($n = 5$).

For impressions recorded under dry conditions, the steel test block mould was placed in an oven (32 ± 2 °C) for 15 min in order to remove any moisture. The stainless-steel ring was placed on the test block to form the specimen-forming cavity. The VPS impression material was extruded directly into the cavity using an auto-mixing gun; an acetate sheet and a flat metal plate ($50 \times 50 \times 3$ mm³) were placed on top of the mould to contain the material. The whole assembly was placed in a C-clamp, and sufficient force was applied to seat the metal plate firmly against the mould [27]. Subsequently, the specimen-forming assembly was placed in a water bath (32 ± 2 °C) until set to simulate polymerisation in an aqueous oral environment [27,28].

For impressions taken under moist conditions, a fine mist of deionized water (DW) was sprayed on the surface of the steel block before taking an impression of the scribed lines, and then the above procedure (for dry conditions) was followed. For impressions recorded under wet conditions, the steel test block was immersed in DW for 15 min prior to the

impression material being syringed onto the die surface; the procedure for dry conditions was followed.

After removing the sample from the mould, the detail reproduction of each line was observed via blind evaluation by two independent examiners using an optical microscope (Olympus Meiji, Miyoshi-machi, Japan) with $\times 7$ magnification and low angle illumination. In order to comply with the ISO4823 [23], a medium-bodied (type 2) VPS impression material should reproduce a continuous 20 μm line (line c) between the intersecting cross lines (d_1 and d_2), as shown in Figure 1.

2.6. Compatibility with Gypsum

After measuring the reproduction of details on the resulting impressions, the specimens ($n = 5$ per material; diameter = 30 mm and thickness = 3.5 mm) were then cast with dental stone type IV [23,29], 24 h after setting, to simulate the clinical situation [30]. The dental stone was hand mixed according to the manufacturer's instructions, poured onto the specimen with mechanical vibration and allowed to set for 45 min at room temperature. The gypsum cast was then separated from the assembly and observed for the detail reproduction of the lines using an optical microscope (Olympus Meiji, Miyoshi-machi, Japan) with $\times 7$ magnification and low angle illumination, by blind evaluation by two independent examiners. All the samples were of the same colour (dental stone type IV) so the examiners could not distinguish between casts from the various impression materials.

In order to comply with the ISO4823 [23], the casts from a medium-bodied consistency VPS impression material should reproduce a continuous 50 μm line (line b) between d_1 and d_2 (Figure 1)

3. Results

3.1. Detail Reproduction

A medium-bodied (type 2) material is required to reproduce the continuous line of 20 μm between d_1 and d_2 (Figure 1), in accordance with ISO4823 [23].

Table 3 shows the results of surface detail reproduction for three commercial and five Exp VPS (medium bodied) impression materials, under three different conditions (dry, moist and wet). Under dry conditions (Figure 2), impressions recorded with all commercial and Exp materials reproduced the 20 μm line satisfactorily. Under moist conditions, all commercial and some of the Exp (III, IV, V) VPS reproduced the 20 μm line satisfactorily, with the exception of Exp-I and II (controls with no surfactant) (Figure 3 and Table 3). This indicates that moist conditions adversely affected detail reproduction of these materials. Under wet conditions, two commercial VPS (Aq M and Extr M) and two Exp materials (IV and V) reproduced the continuous line between d_1 and d_2 , while Elt M and Exp-I, II and III failed to produce an acceptable continuous line (Figure 4 and Table 3).

Table 3. Surface detail reproduction for commercial and Exp VPS (medium bodied) impression materials under dry, moist and wet conditions.

Sr. No.	Materials	Dry	Moist	Wet
1	Exp-I	Satisfactory	Unsatisfactory	Unsatisfactory
2	Exp-II	Satisfactory	Unsatisfactory	Unsatisfactory
3	Exp-III	Satisfactory	Satisfactory	Unsatisfactory
4	Exp-IV	Satisfactory	Satisfactory	Satisfactory
5	Exp-V	Satisfactory	Satisfactory	Satisfactory
6	Aq M	Satisfactory	Satisfactory	Satisfactory
7	Elt M	Satisfactory	Satisfactory	Unsatisfactory
8	Extr M	Satisfactory	Satisfactory	Satisfactory

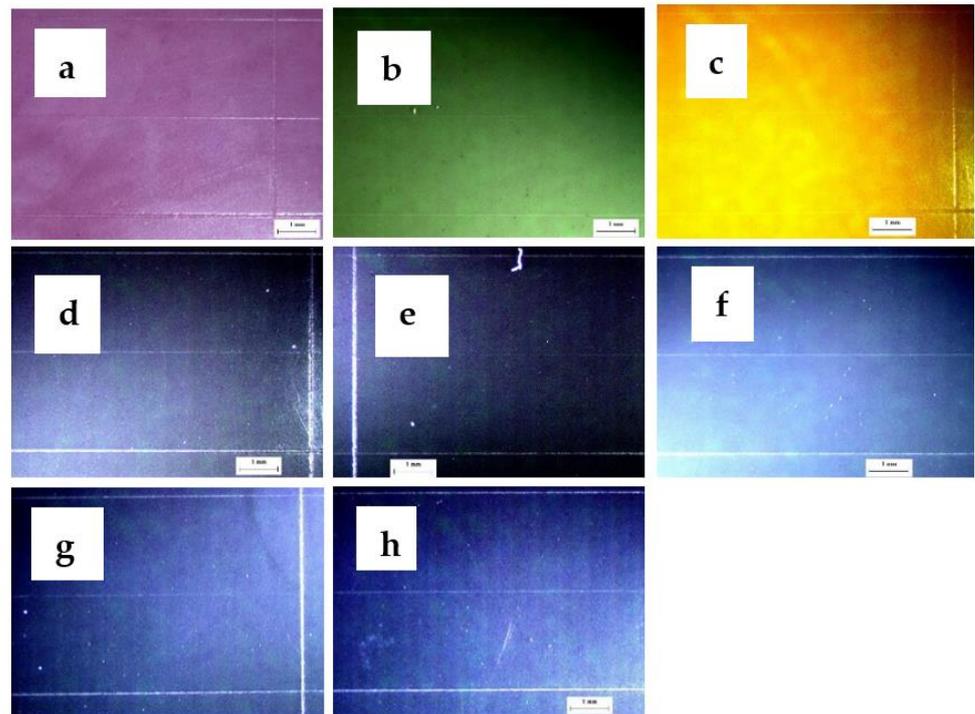


Figure 2. Surface detail reproduction for commercial and Exp VPS (medium-bodied) impression materials under dry conditions, where (a) Aq M, (b) Extr M, (c) Elt M, (d) Exp-I, (e) Exp-II, (f) Exp-III, (g) Exp-IV and (h) Exp-V. The scale bar is 1 mm.

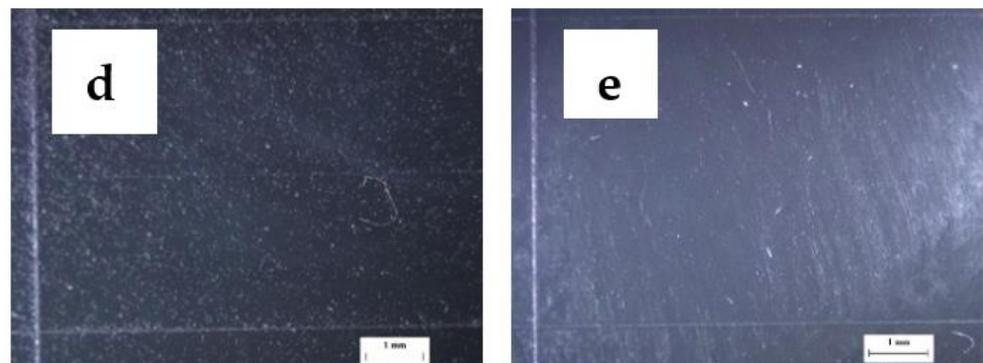


Figure 3. Surface detail reproduction for commercial and Exp VPS (medium-bodied) impression materials under moist conditions, where (d) Exp-I and (e) Exp-II. The scale bar is 1 mm.



Figure 4. Cont.

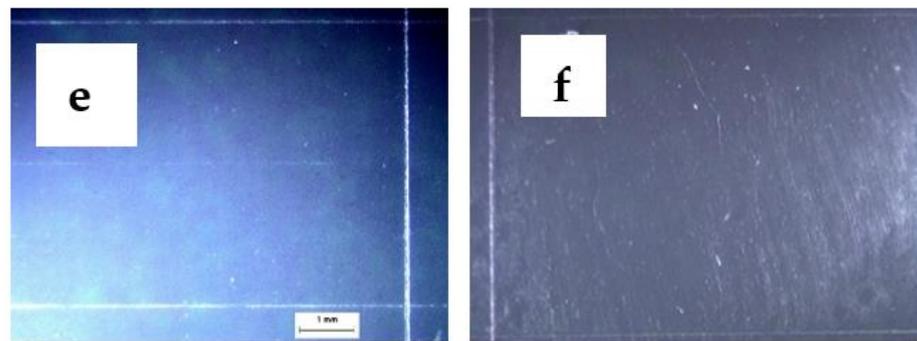


Figure 4. Surface detail reproduction for commercial and Exp VPS (medium-bodied) impression materials under wet conditions, where (c) Elt M, (d) Exp-I, (e) Exp-II and (f) Exp-III. The scale bar is 1 mm.

3.2. Compatibility with Gypsum

Table 4 shows the results for the compatibility of the three commercial and five Exp VPS (medium bodied) impression materials with gypsum products, when poured under dry, moist and wet conditions. According to ISO4823 Standards [17], the cast of a medium-bodied VPS impression material should reproduce a continuous line of 50 μm between d_1 and d_2 when poured with gypsum [23]. All commercial and Exp VPS impressions made under dry conditions demonstrated compatibility with dental stone type IV and reproduced the 50 μm line between d_1 and d_2 on the casts. For the casts of the impressions made under moist conditions, Elt M and Exp-I and II did not record the continuous line (Figure 5 and Table 4), while Aq M, Extr M and Exp-III, IV and V reproduced this line satisfactorily. Under wet conditions, the casts of Aq M, Extr M and Exp-IV and V reproduced the continuous line of 50 μm , while the rest of the materials (Elt M and Exp-I, II and III) failed to record this line (Figure 6 and Table 4).

Table 4. Compatibility with gypsum products for commercial and Exp VPS (medium bodied) impression materials under dry, moist and wet conditions.

Sr. No.	Materials	Dry	Moist	Wet
1	Exp-I	Satisfactory	Unsatisfactory	Unsatisfactory
2	Exp-II	Satisfactory	Unsatisfactory	Unsatisfactory
3	Exp-III	Satisfactory	Satisfactory	Unsatisfactory
4	Exp-IV	Satisfactory	Satisfactory	Satisfactory
5	Exp-V	Satisfactory	Satisfactory	Satisfactory
6	Aq M	Satisfactory	Satisfactory	Satisfactory
7	Elt M	Satisfactory	Unsatisfactory	Unsatisfactory
8	Extr M	Satisfactory	Satisfactory	Satisfactory

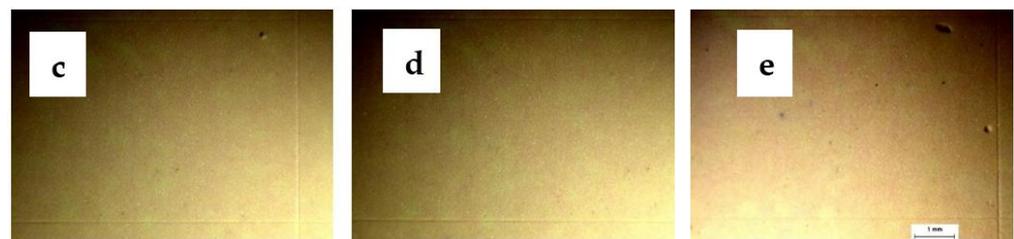


Figure 5. Compatibility with gypsum for commercial and Exp VPS (medium-bodied) impression materials under moist conditions, where (c) Elt M, (d) Exp-I, (e) Exp-II. The scale bar is 1 mm.

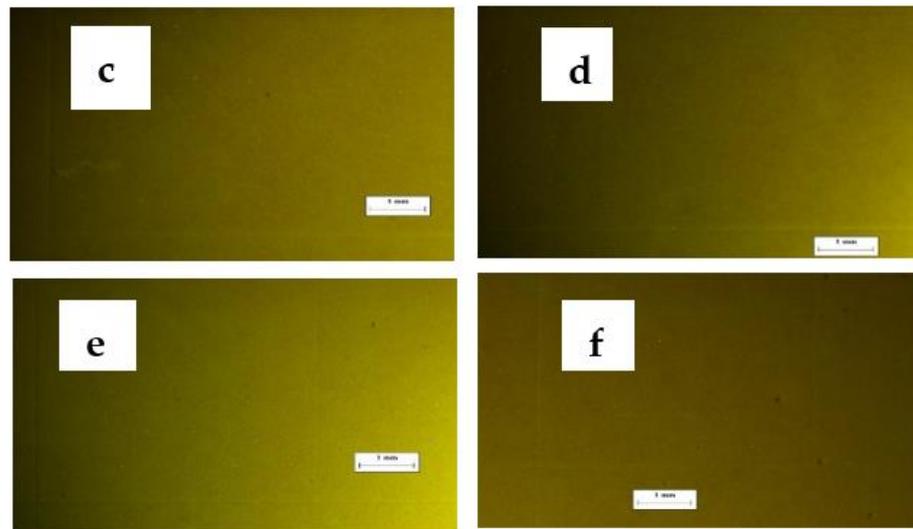


Figure 6. Compatibility with gypsum for commercial and Exp VPS (medium-bodied) impression materials under wet conditions, where (c) Elt M, (d) Exp-I, (e) Exp-II and (f) Exp-III. The scale bar is 1 mm.

4. Discussion

It is critical to have information on the components of a material (VPS), since each component is present for a purpose. As mentioned earlier, in this contribution, the novelty lies in the development of Exp VPS materials *ab initio* and studying the effect of the novel surfactant on the reproduction of fine detail within the impression and after pouring with Type IV stone.

The ability of an impression material to record fine details of a moist or wet surface depends on its wettability and viscosity [16,17,30]. All commercial and Exp VPS used in this study were of medium-bodied consistency. However, they varied in their wetting properties, depending on the type and amount of surfactant incorporated.

The current study investigated the surface detail reproduction of the scribed lines on the steel test block (Figure 1) by commercial and Exp VPS impression materials under dry, moist and wet conditions. To evaluate the detailed reproduction, the criteria set by ISO4823 [23] were followed, which state that a medium-bodied elastomeric impression material should be able to replicate the 20 μm width horizontal line continuously between d_1 and d_2 (Figure 1).

Under dry conditions, all commercial and Exp VPS produced the 20 μm line satisfactorily. However, under moist conditions, all commercial products and the surfactant modified Exp VPS fulfilled these criteria, while the Exp controls (Exp-I and II without surfactant) did not produce acceptable impressions. This clearly indicates that the incorporation of the surfactant (in Exp III, IV and V) conferred a degree of wettability in these materials. Under wet conditions, only two commercial (Aq M and Extr M) and two Exp (Exp-IV and V) VPS reproduced the 20 μm line.

The reproduction of detailed results can be correlated with contact angle (CA) data (REF) and the amount of surfactant incorporated within each material. The two commercial (Aq M and Extr M) and two Exp (Exp-IV and V) VPS that reproduced the 20 μm line under wet conditions had lower CAs than Elt M and Exp III, indicating the former had sufficient wettable/hydrophilic surfaces (published part of the current study [16,17]), under this condition. Exp III had a reduced amount of surfactant (2 wt%) compared with Exp IV and V (2.5 wt% and 3 wt%, respectively). It could be inferred that 2 wt% was not sufficient to render a wettable surface that could deal with the wet condition. These results indicate that, provided the material is wettable (lower CA), it should reproduce the 20 μm line. It is interesting to note that the commercial material, Elt M, was not able to reproduce the 20 μm line under wet conditions, although it is sold as a hydrophilic VPS impression material.

Even though this material is classed as being hydrophilic, it had a higher CA compared to the other two commercial and Exp (III, IV and V) materials. The nature and amount of the surfactant incorporated by the manufacturer is unknown, but the results of this study indicate that it is not sufficient to record details under wet conditions. Clearly, this material is not as hydrophilic as the two other commercial and three Exp (III, IV, V) materials.

Various researchers have reported conflicting results regarding the ability of VPS impression materials to reproduce the complete 20 μm line. Walker et al. [30] reported that hydrophilic VPS (Aq M and Genie Ultra Hydrophilic) performed well only under dry conditions but not under moist conditions, while Petrie et al. [11] reported that Aquasil Monophase and Reprosil satisfactorily reproduced the line under dry and moist conditions but not under wet conditions.

The compatibility of hydrophobic and hydrophilic VPS with stone, with respect to reproducing details, depends on the latter's hydrophilic nature. Stone is mixed with water and, therefore, it is classed as a hydrophilic material. For stone to produce the various lines under moist and wet conditions will depend on whether it can flow into the details recorded by the impression. If the surface of the impression is hydrophilic, then the stone will be able to record detail, while on hydrophobic impression surfaces, the stone will not flow easily into the intricate details and, therefore, these will not be sufficiently produced. The results obtained clearly demonstrate this argument.

All the commercial and Exp VPS impressions made under dry conditions demonstrated compatibility with dental stone type IV and reproduced the 50 μm line between d_1 and d_2 on the casts (Figure 1). Similar results to the above, where impression materials reproduced the various lines under dry, moist and wet conditions, were found in this part of the compatibility study with stone. Under moist conditions, Aq M, Extr M and Exp-III, IV and V reproduced a satisfactory line between d_1 and d_2 , while the remaining materials did not. These results were expected with Exp-I and II and can be explained by the fact that they did not contain a surfactant (Table 4). Under wet conditions, Aq M, Extr M and Exp-IV and V reproduced the continuous line of 50 μm , while the rest of the materials (Elt M and Exp-I, II and III) failed to record this line. Again, it can be assumed that Exp-III contained a lower concentration of surfactant (Table 2) and, hence, this could be the reason why it could not reproduce the 50 μm line. Correlating these data with published CA data indicated that materials with lower CA were able to reproduce the 50 μm [16,17] line.

The results obtained from this study clearly demonstrate that the amount and type of surfactant incorporated within hydrophilic impression materials affects the reproduction of detail under moist and wet conditions. Moreover, the compatibility of these materials with stone to reproduce fine detail was affected. It is not clear which surfactant is incorporated in the three commercial materials used in this study. From the results obtained, it can be assumed that Extr M contains a different surfactant compared to the other materials. Rhodasurf CET-2 (Ethoxylated cetyl-oleyl alcohol) surfactant has proved to be effective and compatible in the Exp materials where Exp III and IV contained a sufficient amount to allow it to reproduce fine detail under dry, moist and wet conditions. This study has shown the importance of knowing the composition and amounts of the various components of an impression material in order to determine their effect on the reproduction of detail and compatibility.

5. Conclusions

This study has shown the importance of the composition of Exp impression materials in order to determine their subtle effects on the reproduction of detail and compatibility with pouring materials (Type IV stone). The role of the surfactant became apparent under moist and wet conditions. Fine detail lines were produced under moist and wet conditions, in both the impression and the resultant stone model, and this was dependent on the amount of surfactant incorporated. The novel surfactant, Rhodasurf CET-2 (Ethoxylated cetyl-oleyl alcohol), was also compatible with the other components of the Exp materials. Recording of fine details is extremely important for clinicians in fabricating accurate

prostheses. These results indicate that clinicians should be cautious when using a VPS impression material which could compromise reproduction of detail within the impression material and in the resulting stone model.

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