

ORIGINAL RESEARCH

A comparative evaluation of the initial hydrophilicity of different elastomeric impression materials in the unset and set stages at two different drop ages: an in vitro study

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ABSTRACT

Purpose: To compare the initial hydrophilicity of six various commonly used and commercially available unset and set elastomeric impression materials. **Material and Methods:** 10 samples each for both unset and set stages were made for six elastomeric impression materials IMPREGUM (IM), AQUASIL (AQ), AFFINIS (AF), EXAMIX (EX), PHOTOSIL (PS) and SPEEDEX (SX) using molds of specific dimensions. Contact angles were measured 30 seconds (unset material) and 60 minutes (set material) after mixing using the contact angle goniometer with 8-μL water drops. The shapes of the drops were video-recorded at drop ages of 1 second and 3 seconds. The initial hydrophilicity was quantified by the calculation of the respective left and right contact angles of each drop shape of 1 second and 3 second sold drops using the analytical software.

Results: IMPREGUM was found to have the maximum hydrophilicity in the unset stage and AQUASIL in the set stage. Within each group, significant differences in the contact angles were observed in the unset and set stage at 1 second and 3 seconds ($p = 0.000$) for all elastomeric materials except for Impregum in the unset stage ($p = 0.000$). **Conclusions:** Hydrophilicity during setting is not correlated with hydrophilicity after setting for all materials. Polyether impression material was more hydrophilic in the unset stage than in the set stage. AQUASIL has a considerable amount of hydrophilicity in the set stage among all the elastomers tested.

Key-words: Contact angle, elastomers, impression materials, wettability

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INTRODUCTION

The establishment of a dry field is crucial during final impression procedures if an accurate impression is to be obtained.¹ Achieving this is quite difficult in case of mandibular teeth with subgingivally placed margins, since the presence of organic fluids, such as blood or saliva, can induce void formation in the impression. Although there is a general consensus that hydrophilicity is very important for impression castability, there is some controversy in the literature

as to whether surface wettability is important for detailed tooth surface reproduction.²⁻⁴ Ideal impression material should possess hydrophilic properties both before and after setting which means that the impression material should have a relative affinity for the liquids, which could be either water, organic fluids and/or saturated calcium sulfate dihydrate solutions.^{5,6} Among the final impression materials, reversible hydrocolloid is probably the only one that has true hydrophilic properties.^{7,8} However, its poor tear

strength and the necessity to be poured immediately, have limited its use.^{9,10} Small bubble formation on the occlusal or axial walls is usually of minor importance; however, defects on the marginal areas or at pinhole locations are very important and should be avoided.

The introduction of polyether and polyvinyl siloxanes helped clinicians obtain accurate, dimensionally stable impressions. The hydrophilic properties of polyether have been recognized since its introduction.^{11,12} Polyvinyl siloxanes though, had inherent hydrophobic properties which made both impression making and pouring with dental stone difficult.¹³ Topical surfactants increased addition silicones' wettability when poured with gypsum products, and, as a result, voids were reduced in the master casts.¹⁴⁻¹⁶

Water contact angles are most frequently used to determine the hydrophilic properties of impression materials. Some previous studies concluded that the contact angles of hydrophilic polyvinyl siloxanes were not significantly different from those of polyether, and as a result, their castability with dental stone was comparable.^{17,18} Few other studies have reported better wetting ability of the hydrophilic polyvinyl siloxanes when compared with that of the hydrophobic ones; however, the contact angle values of these materials were significantly higher than those of polyether.¹⁹

There are many investigations on the hydrophilicity of set impression materials reported in the literature. However, very limited information is available about the hydrophilicity of impression materials in the unset stage^{20,21} which is clinically more relevant. Thus, this study was planned to evaluate the hydrophilicity of different elastomeric impression materials in the unset and set stages at two different drop ages, and to compare the findings of contact angle values amongst all these materials in the unset and set stages. The study was planned keeping Indian Scenario in mind as there are varieties of brands available which can be confusing for a dentist and no unbiased study like ours is available.

MATERIALS AND METHOD

Six commercially available elastomeric impression materials; one Polyether: IMPREGUM, 3M ESPE IM), four Addition silicones: AQUASIL, DENTSPLY (AQ), AFFINIS, COLTENE WHALEDENT (AF), EXAMIX, GC AMERICA (EX), PHOTOSIL, DPI (PS) and one condensation silicone: SPEEDEX, COLTENE WHALEDENT (SX) were selected for this study. The specimens were broadly divided into two main groups:

- Group A -unset (A1 – A60).
- Group B - set (B1 – B60).

The specimens from both, group A (unset) and group B (set) were further divided into 6 equal sub-groups with 10 specimens each (n=10).

Loading of elastomeric impression materials onto the molds

For the cartridge system (AQ, AF, EX), mixing was done using automixing gun (GC) loaded with corresponding supplied mixing tips. For the tube system (IM, PS, SX), the base and catalyst were dispensed on the glass slab, mixed with a spatula and loaded with the syringe. The first few centimeters of mixed paste were discarded to ensure complete mixing.

For Unset specimens: The impression material was syringed onto a metal mold of dimensions (60 mm x 25 mm x 5 mm) exhibiting a 300 µm notch, according to the manufacturer's instructions at room temperature (23°C ± 1°C). After syringing the material, the straight cement spatula was used to smoothen the freshly syringed impression material (Fig. 1A).

For Set specimens: The impression material was syringed into the respective notch on Plexiglass plate 1 (Fig. 1B) of the dimensions (60 mm x 25 mm x 5 mm) with a notch (10 mm diameter and 3 mm deep). When the material was filled inside the notch, a thin aluminum foil was used to cover the complete plate with the material filled notch. Over that foil was placed another Plexiglass plate (PMMA-plate 2) which had the same dimensions as Plate 1 and loaded with 1 kg for 10 minutes. The PA foil was between the two PMMA-plates. Thereafter, the foil was carefully removed, the specimens were air-stored and the contact angles were measured after 50 minutes (60 minutes setting time in all).

Measuring the contact angles

Initial water contact angles were studied on thin unset and set films using contact Angle Goniometer (DIGIDROP GBX) to quantitatively record the average contact angle values (CA) in degrees at drop ages of 1 second and 3 seconds (Fig. 1C, 1D).

The goniometer has a platform to keep the specimen to be tested, a micro-syringe and a camera connected to software that converts the images from the goniometer into a binary image. The video recording of the drops falling from the water outlet is recorded by this inbuilt camera in the Goniometer which later gives the photo images at desired times, say 1 second and 3 second in this particular study.

After loading the material onto the molds for unset and set materials, the specimens were made ready to be kept on the platform of the Goniometer. The platform of the Goniometer was adjusted to get a clear visibility of the inverted image of the drop through the camera. The Goniometer was checked for the availability of distilled water in the outlet. Deionized water was used for the contact angle measurements and applied using a syringe to the substrate. The size of the water droplet was set to be 8 µl. Tissue paper was used to absorb excess water before putting the next specimen under the water droplet. Before recording, the camera was checked for the clear view

and adjusted if required. The recorded image appeared on the screen.

For Unset Specimens

The loaded material on the metal mold was transferred immediately to the contact angle instrument and moved towards the water droplet with a controlled volume on the syringe. The time between the application of the material to the glass slide and the start of the measurement was 30 seconds. As the water droplet of 8- μ L distilled water was about to drop, the stopwatch was set to a time of 1 second first and then for 3 seconds to record the contact angles at these two drop ages at 25 frames per second.

For Set Specimens

The loaded material on the notch was kept in the goniometer under the water drop outlet, 60 minutes after the mixing of the material. As the water droplet of 8- μ L distilled water was about to drop, the stopwatch was set to a time of 1 second first and then for 3 seconds to record the contact angles at these two drop ages at 25 frames per second.

Video recording of the shapes

The measurements were started before the droplet touched the surface to record the whole process of the jump to contact, and the following developments of the contact angle were determined with two sets of pictures each at intervals of 1 and 3 seconds. One second represented the first picture evaluated after the droplet was deposited on the surface, which also served for the jump to contact evaluation. The water was exchanged for each measurement, and the instruments were cleaned with deionized water twice. Every single experiment was conducted fivefold for all the six materials for unset and set specimens (Fig 2 – 7).

The pictures were further processed by the software supplied with the goniometer and were used to investigate changes in the water contact angle during the setting of the dental impression material. The initial hydrophilicity was quantified by the calculation of the respective left and right contact angles of each drop shape of 1 second and 3 second old drops using the analytical software of the contact angle Goniometer.

The observations were subjected to statistical analysis. Mean and standard deviations were calculated and paired samples test was applied to statistically analyze the data obtained.

RESULTS

The “mean value” of contact angles on the comparison between six subgroups of elastomeric impression materials for the unset stage (Table 1 and Graph 1) showed the least value for IMPREGUM (Group A Subgroup a) both at 1 second and 3 second. Thus, IM was found to have the maximum hydrophilicity for the unset stage out of all the elastomeric materials tested. The change in hydrophilicity of Impregum in the unset stage from 1 second to 3 seconds (Table 2) was found to be statistically insignificant ($P=0.110$). However, the hydrophilicity of all the other elastomeric materials (AQ, AF, SP, EX, PS) in the unset stage from 1 second to 3 seconds was found to be statistically significant ($p=0.000$). All unset PVS materials started with contact angles > 85 degrees and showed different kinetics of hydrophilization (i.e. a decrease in contact angle with increasing drop age). In contrast, the unset polyether started with an initial contact angle of 62.45 degrees but lacked distinct hydrophilization.

The “mean value” of contact angles on the comparison between six subgroups of elastomeric impression materials for the set stage (Table 3 and Graph 2) showed the least value for AQUASIL (Group B Subgroup b) both at 1 second and 3 second. Thus, AQ was found to have the maximum hydrophilicity for set stage among all the elastomeric materials tested. The change in hydrophilicity of all the elastomeric materials (IM, AQ, AF, SP, EX, PS) in the unset stage from 1 second to 3 seconds was found to be statistically significant ($p=0.000$).

At a drop age of 3 seconds, the contact angles of set SP, EX reached lower than that of set IM. At drop ages of 1 second and 3 seconds, AQ, AF and SP show a significant decrease of their contact angles (hydrophilization) in the unset stage as compared to the set situation, whereas the polyether (IM) is characterized by a significant increase of contact angle during setting as compared to the unset stage (Table 4 and Graph 3).

Table 1: Paired t test for contact angle values for unset samples (Group A) of all six elastomeric materials tested at 1 second and 3 seconds.

GROUP A UNSET SUBGROUPS		Mean (\bar{X})	n	Standard Deviation (σ)	Standard Error Mean
Subgroup a	Impregum -1sec	62.45	10	4.964	1.570
	Impregum-3secs	61.97		5.121	1.619
Subgroup b	Aquasil-1sec	85.39	10	11.502	3.637
	Aquasil- 3secs	69.44		9.413	2.977
Subgroup c	Affinis- 1sec	85.64	10	14.584	4.612
	Affinis- 3secs	77.20		12.892	4.077
Subgroup d	Photosil- 1sec	103.55	10	1.339	.423
	Photosil- 3secs	99.71		1.199	.379

Subgroup e	Examix- 1sec	93.32	10	3.468	1.097
	Examix- 3secs	90.03		3.056	.966
Subgroup f	Speedex- 1sec	108.11	10	8.241	2.606
	Speedex- 3secs	100.57		15.476	4.894

Table 2: Comparison of “p” values for unset samples (Group A) of all six elastomeric materials tested at 1 second and 3 seconds.

GROUP A	MEAN AT 1 SEC	MEAN AT 3 SECS	p VALUE
Subgroup a (IM)	62.45	61.97	0.110
Subgroup b (AQ)	85.39	69.44	0.000 [*]
Subgroup c (AF)	85.64	77.20	0.000 [*]
Subgroup d (PS)	103.55	99.71	0.000 [*]
Subgroup e (EX)	93.32	90.03	0.000 [*]
Subgroup f (SX)	108.11	100.57	0.000 [*]

***Significant**

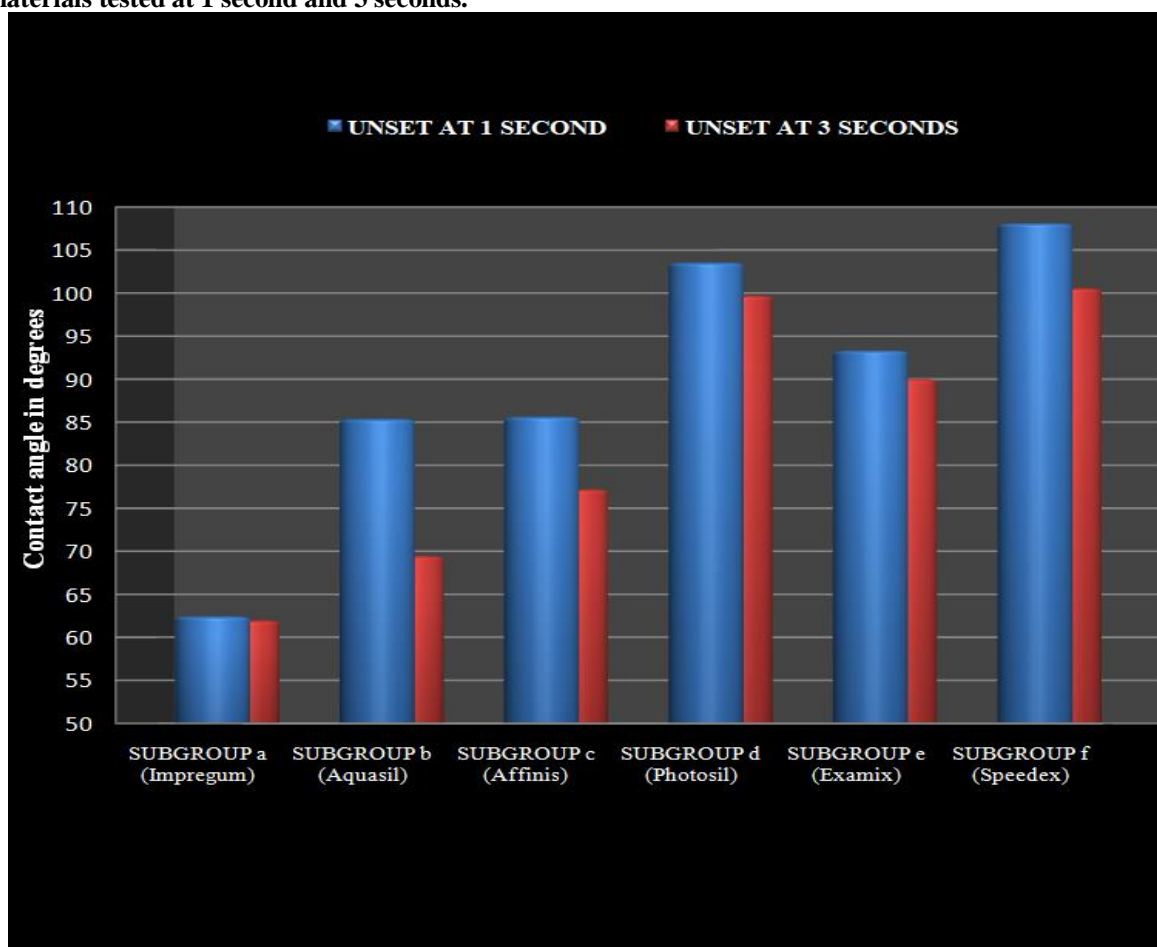
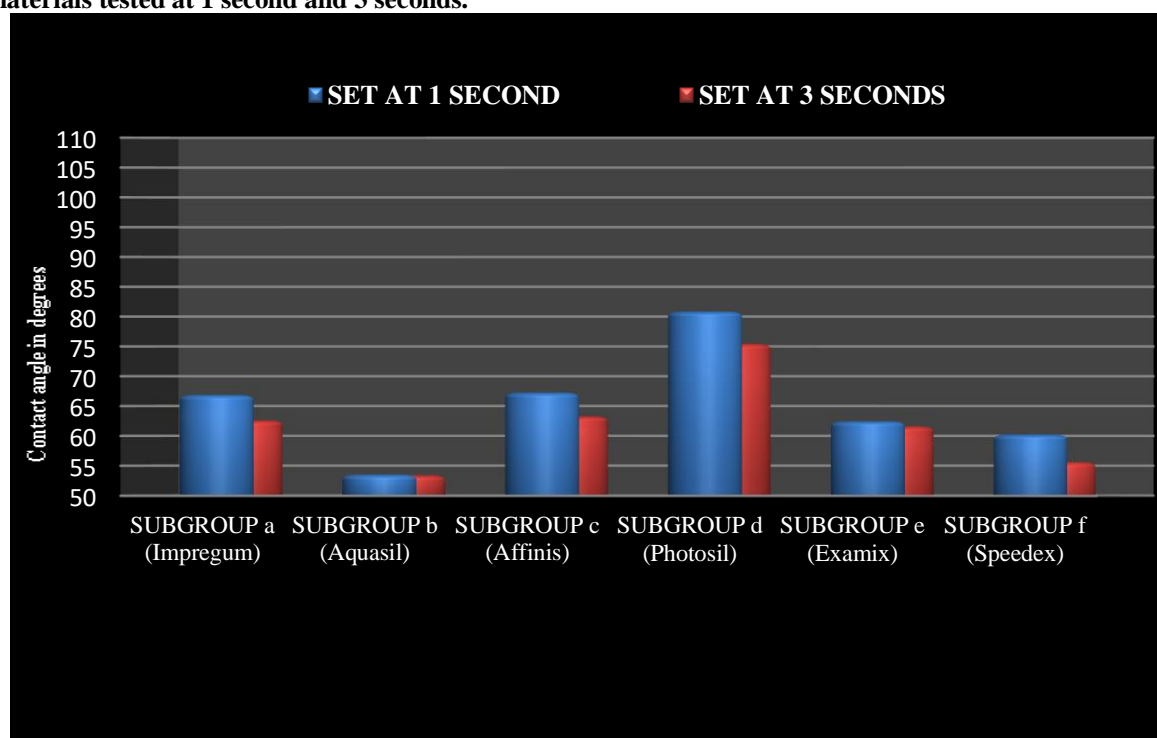
Table 3: Paired t test for contact angle values for set samples (Group B) of all six elastomeric materials tested at 1 second and 3 seconds.

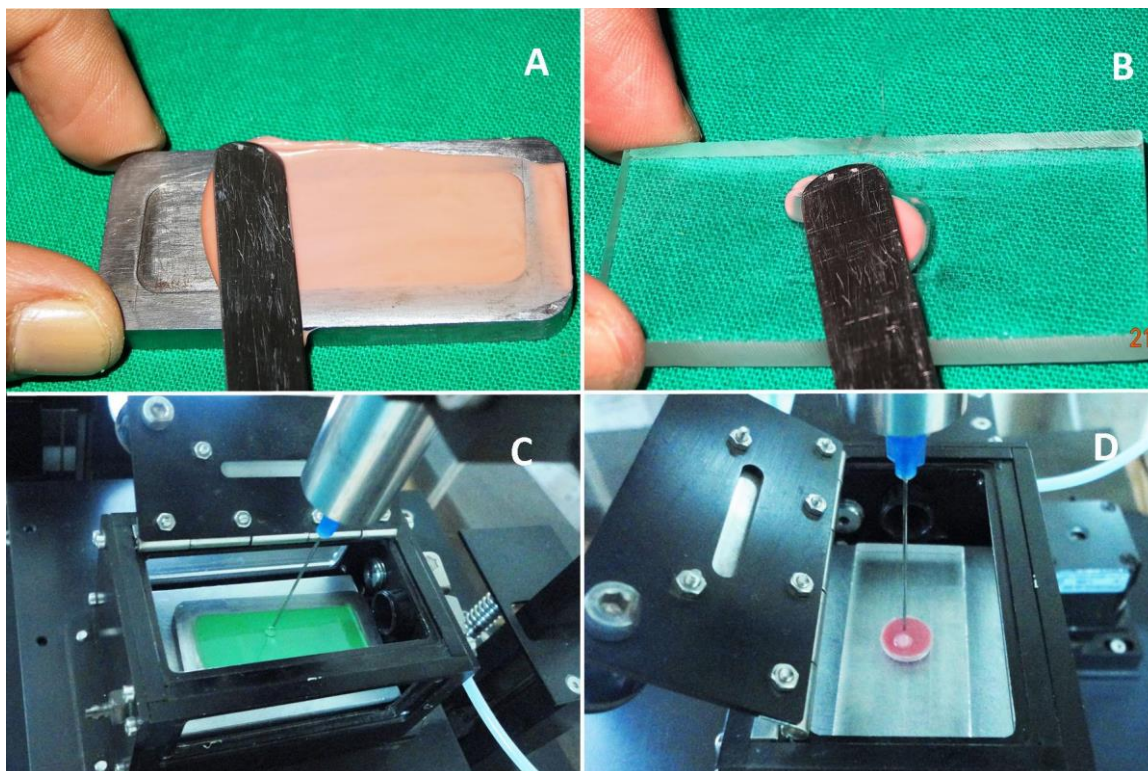
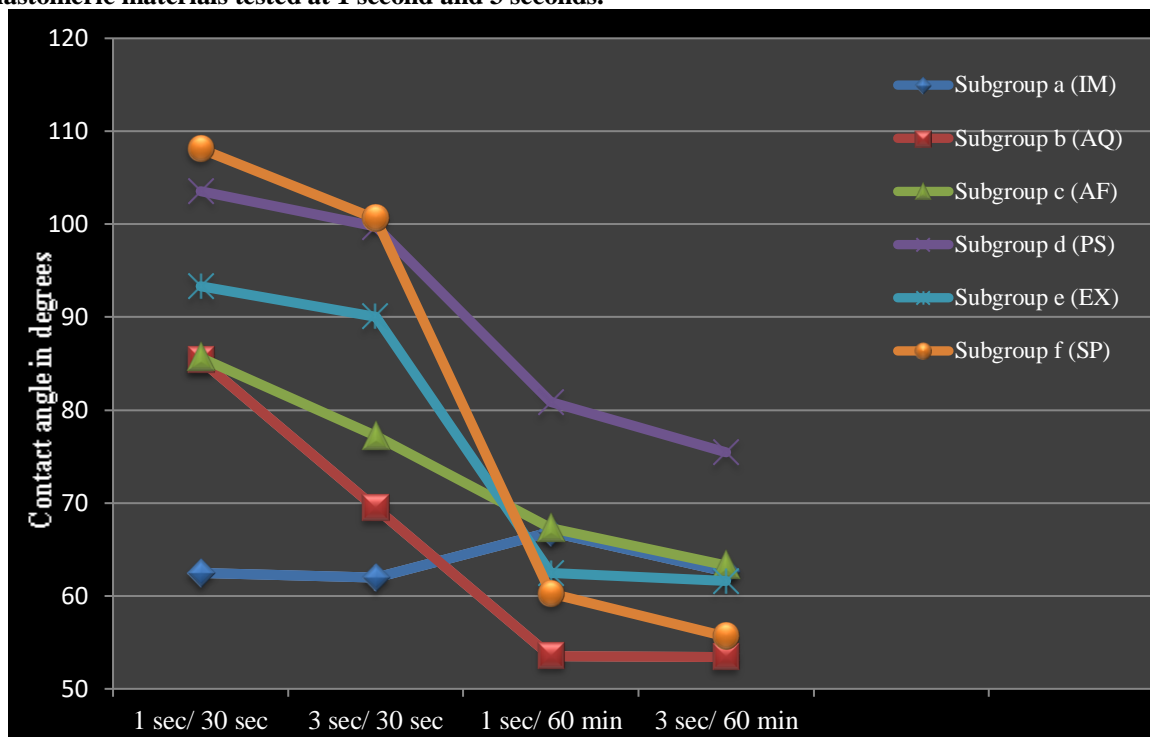
GROUP B SET SUBGROUPS		Mean (\bar{X})	n	Standard Deviation (σ)	Standard Error Mean
Subgroup a	Impregum -1sec	66.87	10	.811	.256
	Impregum-3secs	62.61		.933	.295
Subgroup b	Aquasil-1sec	53.53	10	.048	.015
	Aquasil- 3secs	53.42		.042	.013
Subgroup c	Affinis- 1sec	67.27	10	.955	.320
	Affinis- 3secs	63.28		1.376	.435
Subgroup d	Photosil- 1sec	80.86	10	.622	.197
	Photosil- 3secs	75.45		.682	.216
Subgroup e	Examix- 1sec	62.46	10	.123	.039
	Examix- 3secs	61.61		.145	.046
Subgroup f	Speedex- 1sec	60.25	10	.196	.062
	Speedex- 3secs	55.62		.512	.162

Table 4: Comparison of “p” values for set samples (Group B) of all six elastomeric materials tested at 1 second and 3 seconds.

GROUP B	MEAN AT 1 SEC	MEAN AT 3 SECS	p VALUE
Subgroup a (IM)	66.87	62.61	0.000 [*]
Subgroup b (AQ)	53.53	53.42	0.000 [*]
Subgroup c (AF)	67.25	63.28	0.000 [*]
Subgroup d (PS)	80.86	75.45	0.000 [*]
Subgroup e (EX)	62.46	61.61	0.000 [*]
Subgroup f (SX)	60.25	55.62	0.000 [*]

***Significant**

GRAPH 1: Comparison of mean contact angle values (in degrees) of unset samples of all six elastomeric materials tested at 1 second and 3 seconds.**GRAPH 2: Comparison of mean contact angle values (in degrees) of set samples of all six elastomeric materials tested at 1 second and 3 seconds.**

GRAPH 3: Comparison of mean contact angle values (in degrees) of unset and set samples of all six elastomeric materials tested at 1 second and 3 seconds.**Figure1: A- Loading of impression material into the mold for unset specimens, B- Loading of impression material into the notch on Plexiglass plate 1 for set specimen, C- Contact angle measurement of unset specimen in Goniometer D- Contact angle measurement of set specimen in Goniometer**

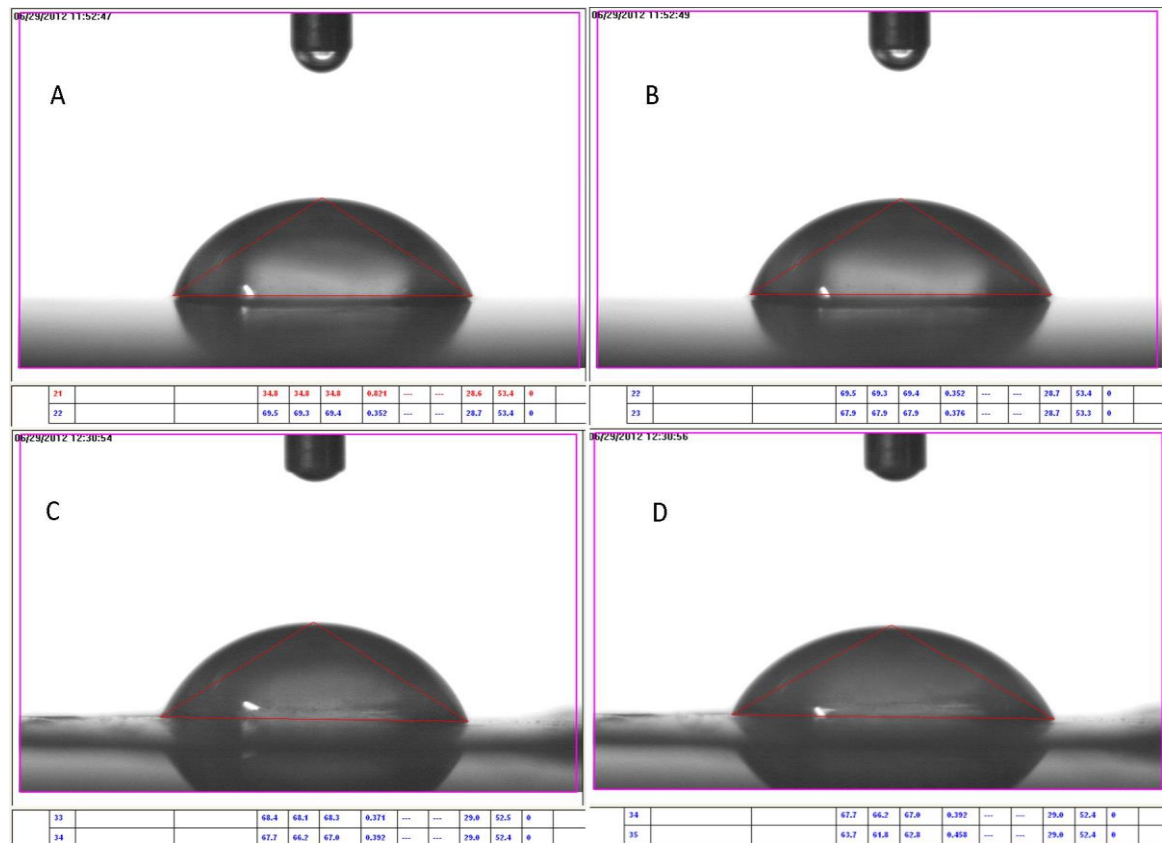


Figure 2: Contact angle measurement for IMPREGUM : A - Unset at 1 sec, B - Unset at 3 sec, C - Set at 1 sec, D - Set at 3 sec

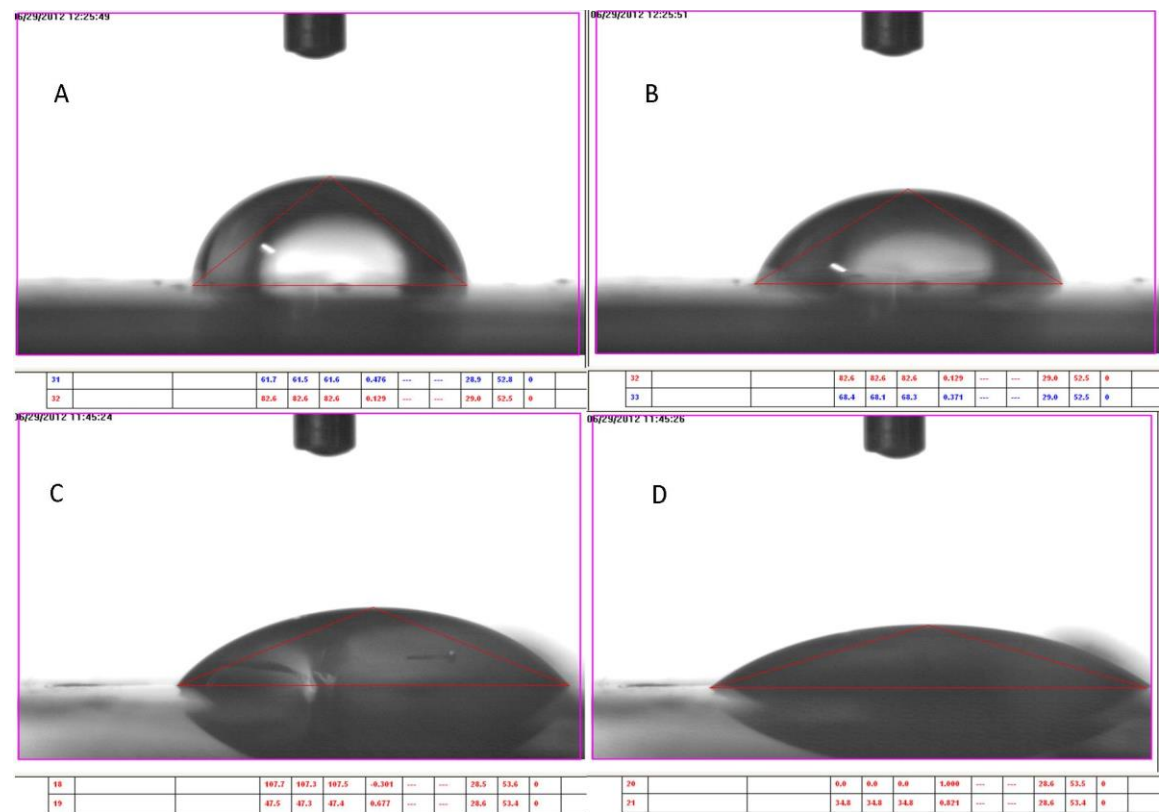


Figure 3: Contact angle measurement for AQUASIL : A - Unset at 1 sec, B - Unset at 3 sec, C - Set at 1 sec, D - Set at 3 sec

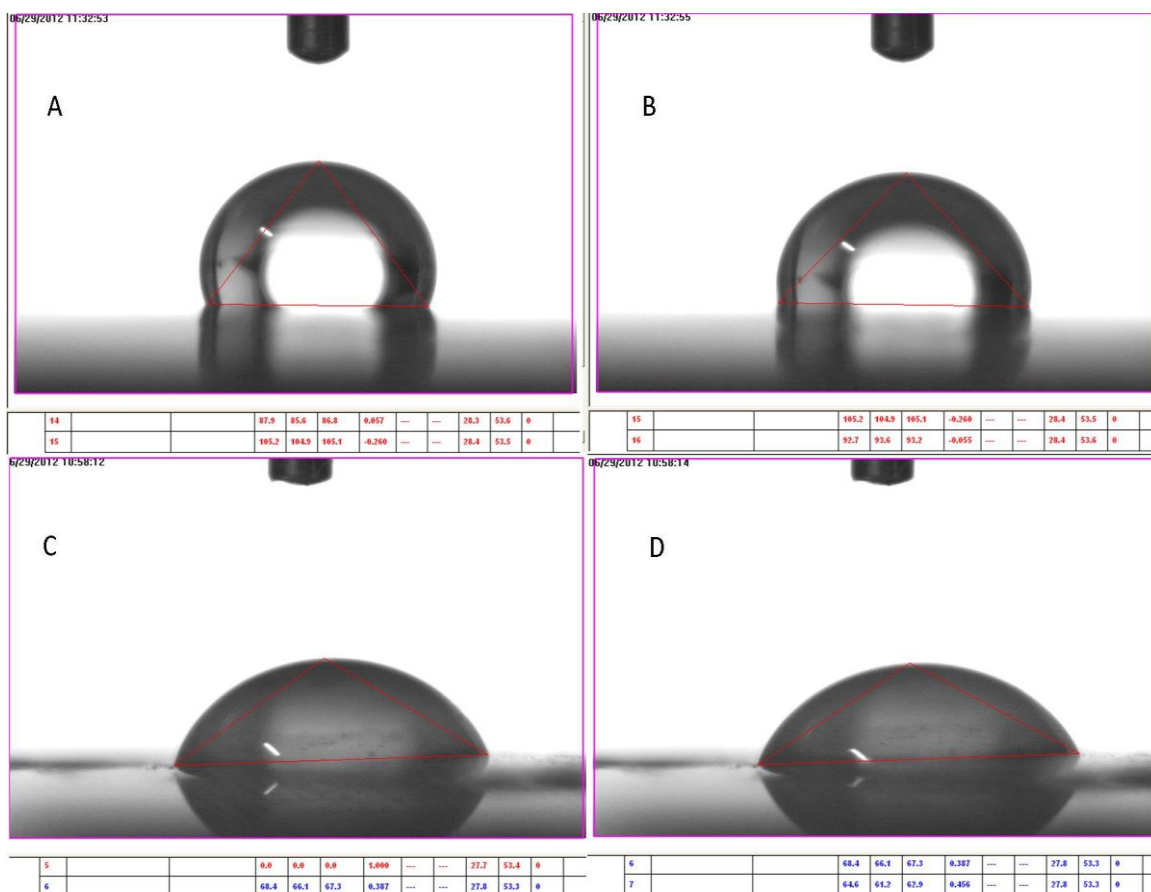


Figure 4: Contact angle measurement for AFFINIS : A - Unset at 1 sec, B - Unset at 3 sec, C - Set at 1 sec, D - Set at 3 sec.

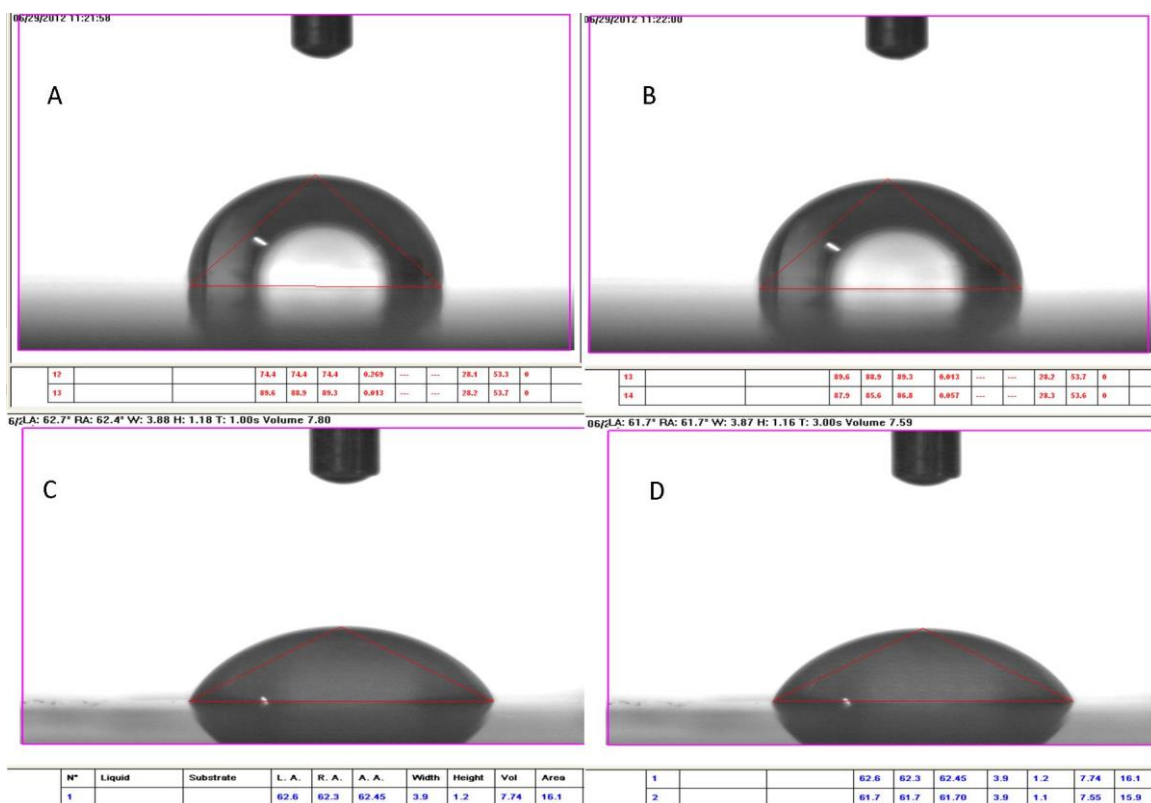


Figure 5: Contact angle measurement for ENAMIX : A - Unset at 1 sec, B - Unset at 3 sec, C - Set at 1 sec, D - Set at 3 sec.

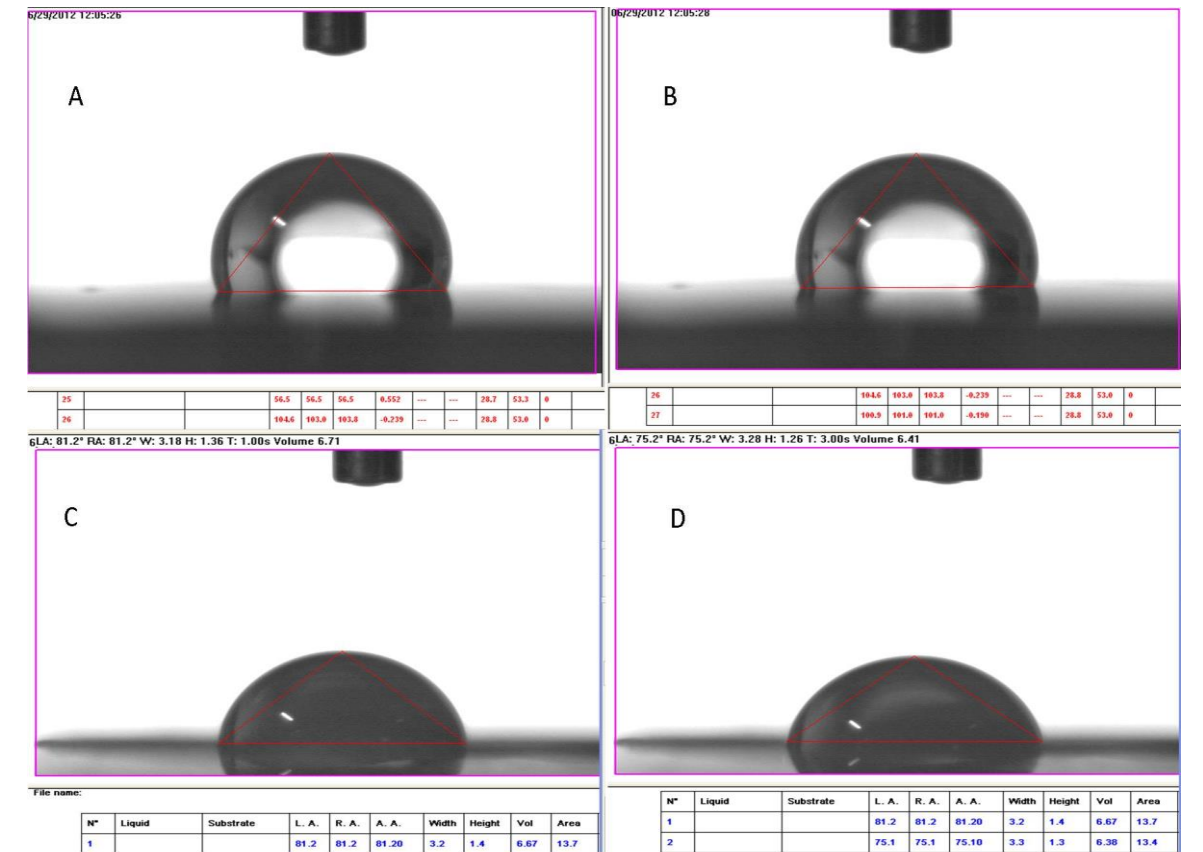


Figure 6: Contact angle measurement for PHOTOSIL : A - Unset at 1 sec, B - Unset at 3 sec, C - Set at 1 sec, D - Set at 3 sec.

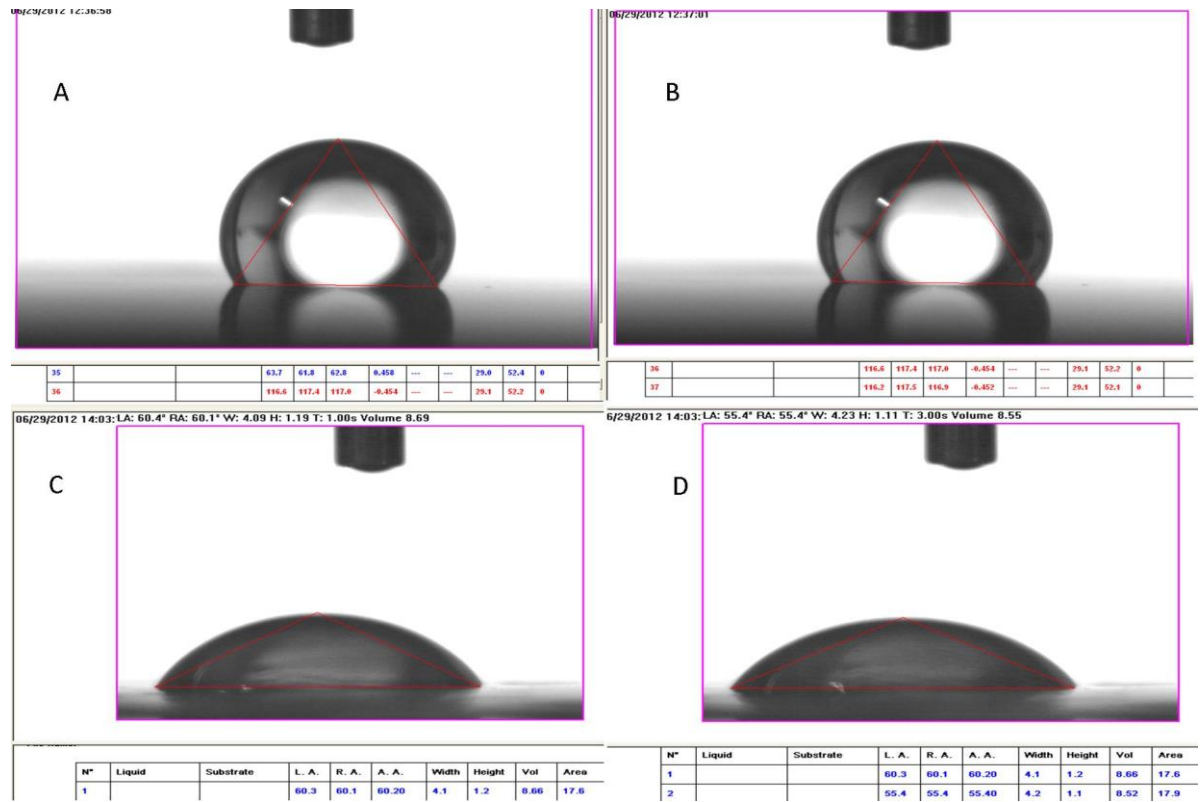


Figure 7: Contact angle measurement for SPEEDEX : A - Unset at 1 sec, B - Unset at 3 sec, C - Set at 1 sec, D - Set at 3 sec

DISCUSSION

A major challenge for a dental impression material is to wet the prepared tooth structure, especially in the area of the finishing line, to obtain a precise impression. Although there is no clear evidence as to which inherent properties of material might specifically affect the wetting ability, hydrophilicity is regarded as a major influencing factor. Besides, the hydrophilicity of the set material is important to avoid entrapment of air during die casting. Hence, the hydrophilicity of an impression material may influence the precision of the impression as well as the die^{5,6}, and thus affect the ultimate clinical success of fixed prosthetic restorations. An impression material needs to flow and adhere to the tooth structure and periodontal tissues, which may be wetted with blood, saliva, and/or water. When these materials are hydrophilic, the water tends to spread and ideally adhere to their surfaces. On the other hand, if the material is hydrophobic, the water creates small droplets, which will finally cause voids in the impression material.

Recent advances have focused on making the impression materials more hydrophilic, thereby allowing the material to make more intimate contact with the oral tissues, to capture better surface details and fewer defects.^{17,22-24} There have been numerous studies on the wettability of cured impression materials.^{25,16,26} However, the wettability of impression materials during the setting has been much less investigated, even though it is this behavior that may be most clinically relevant. Adequate wetting of the material surface during the working time is thus decisive for clinical success in registering fine details.²⁷⁻²⁹ An excellent impression of prepared teeth may be proven useless, if the material used has poor wetting properties.³

In the present study, the hydrophilicity of six commercially available elastomeric impression materials i.e. one polyether (IMPREGUM), four polyvinyl siloxanes (AQUASIL, AFFINIS, PHOTOSIL, EXAMIX) and one condensation silicone (SPEEDEX) in the unset and set stages at two different drop ages and the contact angle values of all these materials were evaluated. The materials taken in the study were those that are commonly used. A total of 120 specimens were made, 60 for unset and 60 for the set stage. This is in accordance with the study conducted by Rupp F et al.³⁰ which compared the initial hydrophilicity of unset and set elastomeric impression materials. Standard dimensions molds were used which were similar to the ones used in the study by Rupp F et al.³⁰ Two different molds were used for unset and set materials. The materials were mixed using an automixing gun and hand spatulation as per the availability of the type of material. Since it was difficult to control the variations, the same operator did all the mixings and placing onto the molds. With automixing gun, the first few centimeters

of mixed paste were discarded to ensure complete mixing.

For contact angle measurement, distilled water was used as the liquid medium as it is the component that would come in contact with the impression material in the oral cavity and during the pouring of impression with gypsum products. It also forms an easily observed contact angle on most materials. 1 second and 3 seconds were used as drop ages in the study. When the contact angle measurements on solid surfaces are evaluated, usually equilibrium contact angles are measured. However, earlier studies have shown that there are remarkable differences in the wetting behavior of impression materials, i.e. the initial drop shape directly after contacting the material surface and after a lapse of time; which seems to have clinical relevance. This was the reason to look in detail at the non-equilibrium drop-shapes and contact angles at two different points of time in the unset and set stage. It has also been found in the previously conducted studies that no significant changes were found in the drop shapes and contact angles after a time-lapse of 1, 2, 3, 10, or 20 seconds from the time of initial contact of the liquid.³⁰ There is no standard procedure, however, to characterize this initial wettability, and that is why 1 second and 3 seconds were taken as the drop ages after the initial material contact to characterize the different kinetics of hydrophilization of the impression materials under research.

The equipment used for contact angle measurement was the contact angle goniometer. This equipment has been proved to be very efficient for contact angle measurements in various studies conducted earlier.^{2,31} The result showed that the increasing order of hydrophilicity for the tested elastomeric materials is:

In the UNSET stage:

IM > AQ > AF > EX > PS > SX

In the SET stage:

AQ > SX > EX > IM > AF > PS

Thirty seconds after mixing (i.e. in the unset stage), the polyether (IM) was found to be the most hydrophilic material with the lowest contact angle. This can be explained by the fact that polyether contains polar oxygen atoms, which have an affinity for water, as stated by Craig and Powers.³² Polyvinylsiloxanes proved to be less hydrophilic than polyether. Reduced hydrophilicity of the examined polyvinyl siloxanes can be explained by the fact that these impression materials contain hydrophobic, aliphatic hydrocarbon groups surrounding the siloxane bond.^{33,34}

In the set stage, the polyether's hydrophilicity was ranked between the very hydrophilic PVS materials (AQ) and the more hydrophobic remaining PVS (AF, PS, EX) and condensation silicone (SP). Also, it was found that addition silicone (AQ) showed the maximum hydrophilicity in the set stage. This is contrary to the fact that polyethers are the most hydrophilic elastomeric impression materials ever.

This study also shows that the hydrophilicity of polyether (IM) reduces as it sets and that of addition silicone (AQ) increases with the setting.

A remarkable difference was observed in the contact angle values at 1 second and 3 second between the most hydrophilic (IM: 62.45, 61.97) and the most hydrophobic (SX: 108.11, 100.57) elastomeric materials in the unset stage respectively. However, the difference in contact angle between these two materials was not as remarkable when compared in the set stage (AQ: 53.53, 53.42; PS: 80.86, 75.45) at 1 second and 3 second respectively. This can be attributed to the surfactants and other additives that are released from the impression materials in the set stage. The surface energy of water is very sensitive to impurities and surfactants and gets lowered in a significant way from its original value of 72.6 mN/m upon contact with an elastomeric impression material in unset and set stage, which can be attributed to the release of surfactants and other additives.^{35,36}

Among the various impression materials studied, the hydrophilicity of condensation silicone (SX) improved considerably during setting. Although, it showed the least hydrophilicity at the unset stage but the value started decreasing within 3 sec and was considerably less after setting and was found to be just next to AQUASIL. Thus, it can be stated that for the condensation silicone that although completely moisture free teeth and gingival sulcus will be a necessary requirement but since the hydrophilicity improves in the set stage, it can be expected that the impression may be poured without entrapment of the air bubbles. Among the different PVS impression materials, PHOTOSIL had the least hydrophilicity in the unset stage followed by ENAMIX, AFFINIS and then AQUASIL. However, after setting, the hydrophilicity of ENAMIX impression material improved and was next to AQUASIL.

It is pertinent to mention here that the hydrophilic nature of any impression material is largely based on the composition of the material. While the polyether impression material has intrinsic hydrophilic property due to presence of polar oxygen atoms, PVS and condensation silicone on the other hand require addition of surfactant to improve this property. The differences observed in the hydrophilicity amongst various addition silicones used in the study can be due to the manufacturer variations in the composition and the quantity of surfactant used.

According to Craig et al, the molecules of the surfactants contain a polyether as a hydrophilic element and a component that is compatible with silicone. It is believed that there is a diffusion of these surfactant molecules into the liquid phase, altering the surface tension of the liquid that comes in contact with the impression material.³² Previous studies have indicated that the term hydrophilic when referring to addition silicones, is probably related to their ability to be poured with gypsum.³⁷⁻⁴⁰ There is no scientific evidence or attempt to study the ability of the

polyvinyl siloxanes to flow into a wet (by water or organic fluids) sulcus and reproduce it accurately. It can be concluded from this study that polyvinylsiloxanes (AQ) have considerable hydrophilicity during the setting to wet the tissue surfaces in the oral cavity and reproduce surface details after pouring also. The differences observed in the hydrophilicity amongst various addition silicones used in the study can be due to the manufacturer variations in the composition and the quantity of surfactant used.

The studies utilizing the Indian brands of materials are scarce in the literature. The composition of the materials used in the present study is entirely different which can change the hydrophilic nature of the materials to a great extent and ultimately affect the impression making procedure. Use of such commonly used materials in both set and unset stage for the present study is strength of the study.

The limitations of the present study are that all the values gained in the study were subjected to the experimental setup and hence can only be used for the comparison of materials studied under similar conditions.

CONCLUSION

Within the limitations of this study, the following could be concluded:

1. There is no correlation between the hydrophilicity data obtained in the unset stage and the set material. Polyether impression material (IMPREGUM) was more hydrophilic in the unset stage than in the set stage. Vinyl polysiloxanes showed a stepwise development of hydrophilicity in the set stage, which was not observed in the unset stage.
2. AQUASIL has a considerable amount of hydrophilicity in the set stage among all the elastomers tested.

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