CONTACT ANGLE MEASUREMENT ON DENTAL IMPLANT BIOMATERIALS

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KEY WORDS

Dental implants
Contact angle measurement
Surface preparation
Wettability
Biomaterials
Implant surface

Wettability may be one of the surface factors to be considered when selecting dental implant biomaterials. Contact angles of dental implant surface preparations influence wettability and tissue adhesion. The contact angles of eight implant surface preparations were determined in this study. Contact angles were measured by a telemicroscope equipped with a protractor eyepiece. Groups 1 to 6 had a Ti_6Al_4V substrate. Group 1 was metallurgically polished, group 2 was blasted with 180 µm Al_2O_3, group 3 was blasted with 710 µm Al_2O_3, group 4 was hydroxyapatite (HA) blasted (125 µm), group 5 had a Calcitite HA plasma-sprayed coating, and group 6 was coated with plasma-sprayed MP-1 HA. Group 7 was metallurgically polished commercially pure (CP) titanium (grade 1), and group 8 was etched CP titanium (grade 1). Contact angles were measured 30 times for each group with distilled water and glycerol, and the determinations were statistically analyzed. Mean contact angles for groups 1 to 8 were 65.5, 65.3, 62.5, 67.9, 46.6, 81.7, 58.5, and 69.0, respectively, when tested with distilled water, and 70.7, 68.3, 81.6, 75.4, 67.1, 70.7, 62.3, and 82.5, respectively, when tested with glycerol. Analysis of variance and Tukey's Honestly Significant Difference test ($p = 0.05$) demonstrated significant differences between group 5 and all other groups when groups were tested with distilled water and demonstrated no significant differences between groups 5 and 7 when groups were tested with glycerol. Surface preparation of implant biomaterials affects wettability. In this study, Ti_6Al_4V coated with Calcitite HA had the lowest contact angles and the best wettability.

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INTRODUCTION

Although many different dental implant biomaterials and systems are used to treat edentulous and partially edentulous patients, available information still does not allow a completely informed selection of these tooth-replacing materials. In 1988 at the National Institutes of Health Consensus Development Conference on Dental Implants, Balkin raised 10 areas with deficient knowledge for future research, including the need for comparative research on various types of implants under the same conditions and circumstances.[1] The determination of the questionable issue of equivalent integration among

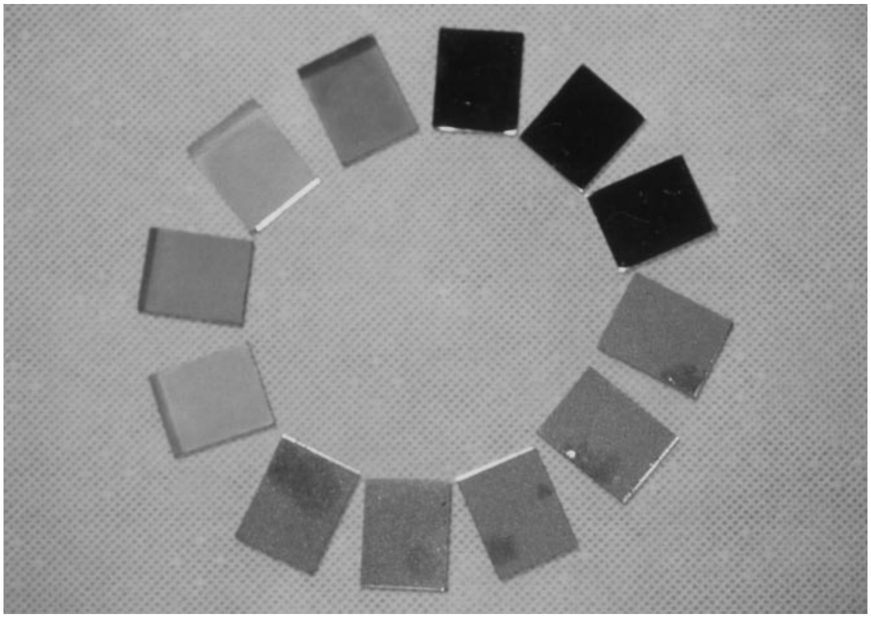

FIGURE 1. The CP titanium (grade 1) and Ti_6Al_4V alloy were prepared with different surface treatments.

implants of different biomaterials or designs is best established by an improved basic understanding of the bone-biomaterial interface, rather than by trial and error in prolonged clinical treatment.[2]

Structural and functional bonding between living bone and the surface of load-bearing implants is believed to be an important factor for implant success. One major factor in biocompatibility of an implant is its surface quality. The initial interaction between living bone and tissue and implant surface occurs as the surface of the implant biomaterial is exposed to tissue fluids. This produces a layer of macromolecules and fluids, which influences the behavior of cells when they encounter the implant surface. Following these events, a series of cell-material interactions takes place, leading to the release of growth and chemotactic factors, which may modulate cellular activity in the surrounding tissue.[3–5] The surface quality of the implant will depend on its chemical, physical, mechanical, and topographical properties. Several authors have called attention to the importance of careful characterization of the surface properties.[6–8] Among the surface analysis techniques used to study material interactions with host fluids and tissues are infrared spectroscopy, ellipsometry, immunofluorescence assays, profilometry, electron microscopy, scanning auger microprobe analysis, secondary ion mass spectrometry, and contact angle measurement.[9] The determination of slowly advancing equilibrium contact angle profiles of a variety of pure liquids placed against the relevant material surfaces is useful in displaying the operational surface energetic or adhesive properties of the materials.[10]

Contact angle techniques evolved from the method first described by Young in 1805, as cited by Baier *et al.*[11] If a small amount of liquid is placed on a solid surface and it does not spread, a drop is formed. The angle of intersection of a line tangent to the liquid and the surface of the solid that it contacts is the contact angle. This angle is characteristic of the substances in the system because of the surface tension of the liquid and the surface energy of the solid, modified by certain properties such as roughness. Stated another way, a low contact angle indicates good wettability, whereas a high contact angle results in poor wettability. As the contact angle increases, the wetting decreases.[12,13]

The contact angles of eight implant surface preparations were determined in this study to answer the following questions: does surface preparation of implants affect wettability, and which implant surface has the lowest contact angle?

MATERIALS AND METHODS

Two types of metals were used in this study, commercially pure (CP) titanium (grade 1) and titanium alloy (Ti_6Al_4V). SULZERmedica (Sulzer Calcitek Inc., Carlsbad, Calif) and 3i (Implant Innovations, Palm Beach Gardens, Fla) supplied the specimens. The specimens were approximately ¾ × 1–inch rectangles and were rinsed with acetone for 5 minutes and then in isopropyl alcohol for 5 minutes, followed by thorough drying before the contact angle measurements were made. The specimens were handled only with forceps and gloved hands to eliminate the transfer of oil and other contaminants from the skin to the specimens. The specimens were then divided into eight different surface preparation and material groups (Fig 1).

The specimens from SULZERmedica were as follows: group 1, Ti_6Al_4V metallurgically polished surface, final polish with colloidal silica; group 2, Ti_6Al_4V alumina-blasted surface, blasted with 180 μm Al_2O_3; group 3, Ti_6Al_4V alumina-blasted surface, blasted with 710 μm Al_2O_3; group 4, Ti_6Al_4V hydroxyapatite (HA)-blasted surface, blasted with 125-μm HA particles; group 5, Ti_6Al_4V Calcitite HA plasma spray coating surface; and group 6, Ti_6Al_4V MP-1 HA plasma spray coating surface. Specimens from 3i were as follows: group 7, CP titanium (grade 1) metallurgically polished surface and group 8, CP titanium (grade 1) etched surface. The implant material surface was measured 30 times for each group.

Contact angle determinations were

made by placing one drop (0.025 mL) each of two types of liquids, distilled water and glycerol (J.T. Baker Chemical Co., Phillipsburg, NJ), on the implant surfaces. Contact angles were measured by a telemicroscope (Miruc, Japan) equipped with a protractor eyepiece (Figs 2, 3). This type of instrumentation has been used for many years by several researchers.[9,10,12,13] The distance between the tip of the micrometer buret (Gilmont Instruments, Barrington, Ill) and the implant surface was kept constant at 10 mm (Fig 4). All specimens were read in the same environment by one trained investigator. The data were analyzed by analysis of variance and Tukey's honestly significant difference—multiple comparison tests, and their means and standard deviations were calculated. All statistical tests were computed at the p = 0.05 level of significance. The data were statistically analyzed with a SPSSWIN computer package (SPSS Inc., Chicago, Ill).

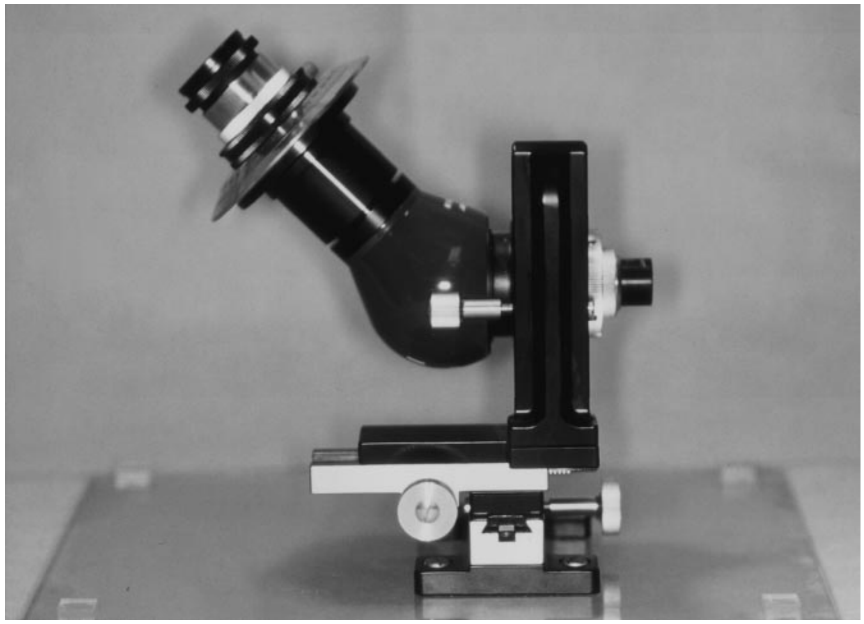

FIGURE 2. Telemicroscope used for contact angle measurements.

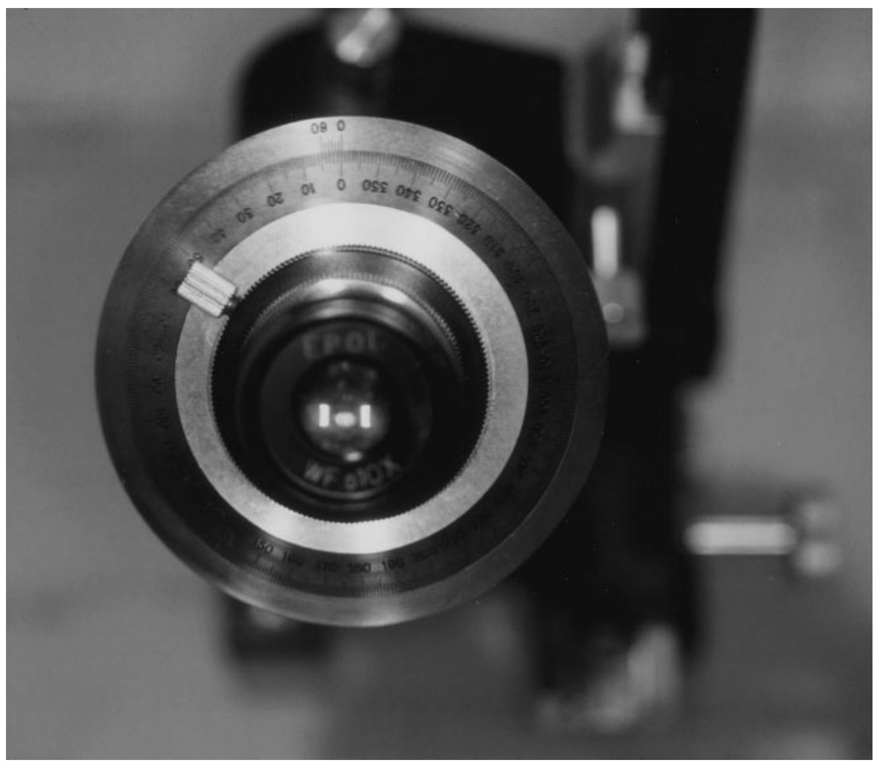

FIGURE 3. Protractor eyepiece.

RESULTS

Tables 1 and 2 show the means and standard deviations for the distilled water and glycerol groups. Group 5 (Ti_6Al_4V Calcitite HA plasma spray coating surface) had the lowest contact angle value when tested with distilled water, and group 6 (Ti_6Al_4V MP-1 HA plasma spray coating surface) had the highest contact angle when tested with distilled water. Group 7 (CP titanium, grade 1, metallurgically polished surface) had the lowest contact angle value when tested with glycerol, followed by group 5. Group 8 (CP titanium, grade 1, etched surface) had the highest contact angle when tested with glycerol.

The differences among the implant materials and surface preparation groups were significant (p = 0.05). Analysis of the data revealed that significant differences were found between group 5 and all of the other groups (groups 1, 2, 3, 4, 6, 7, and 8) when tested with distilled water. There were no significant differences between group 7 and group 5 when tested with glycerol, but group 7 was significantly different from groups 1, 2, 3, 4, 6, and 8, and group 5 was significantly different from groups 3, 4, and 8 when tested with glycerol.

DISCUSSION

An important consideration of this investigation was to evaluate the importance of surface preparation of implant materials. With regard to wettability,

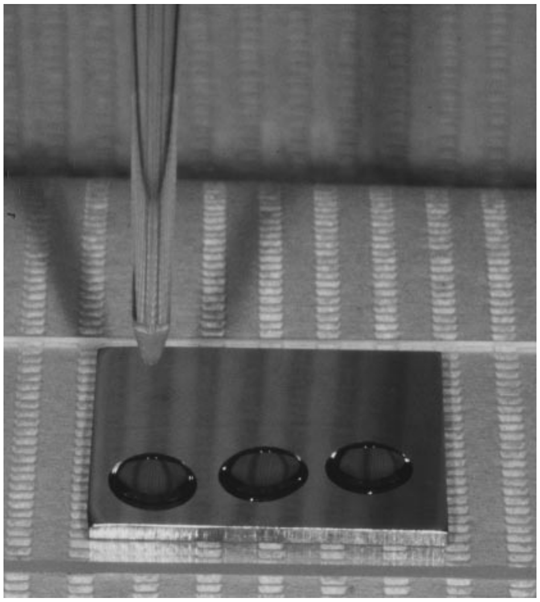

FIGURE 4. The distance between the tip of the micropipette and the implant surface was kept constant at 10 mm.

TABLE 1
Mean and standard deviation (when tested with distilled water)

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean
1	5	65.58	1.98	0.88	63.11–68.04
2	5	65.30	2.21	0.99	62.55–68.04
3	5	62.50	2.84	1.27	58.97–66.02
4	5	67.95	1.25	0.56	66.38–69.51
5	5	46.65	1.48	0.66	44.80–48.49
6	5	81.77	3.15	1.57	76.75–86.78
7	5	58.53	1.60	0.72	56.50–60.55
8	5	69.03	1.30	0.58	67.41–70.65
Total	40	64.22	9.29	1.48	61.21–67.23

TABLE 2
Mean and standard deviation (when tested with glycerol)

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean
1	5	70.75	1.04	0.46	69.45–72.04
2	5	68.37	3.17	1.41	64.43–72.31
3	5	81.68	4.59	2.05	75.98–87.38
4	5	75.43	1.69	0.75	73.33–77.53
5	5	67.15	1.69	0.75	65.04–69.25
6	5	70.70	1.06	0.47	69.37–72.02
7	5	62.35	2.60	1.16	59.11–65.58
8	5	82.50	2.86	1.28	78.94–86.05
Total	40	72.36	7.08	1.11	70.10–74.63

low contact angles indicate good wetting (the liquid must flow easily over the entire surface and adhere to the solid surface). The ability of a liquid to wet the surface of a solid is influenced by a number of factors such as the roughness of the surface, the cleanliness of the surface, and the surface energy of the solid in relation to the liquid and atmospheric gas. These factors create the driving force for the liquid to spread on the solid surface.[11]

Surface energy is important for wettability.[11] Surface energy is the energy at the surface of a solid, which is greater than its interior energy. The outer atoms are not equally attracted to each other as in the inner layer of atoms. The energy is greatest on the outermost atomic layer because the unsaturated bonds generate surface energies.[14] When a liquid is placed on a lower-energy surface metal, the contact angle will be higher as compared with a higher-energy surface metal.[11]

There was no clear explanation for the different contact angle values on different kinds of implant surface preparations and different implant materials. One or a combination of factors related to contact angle measurements might explain these results, including surface preparation techniques (metallurgically polishing, alumina blasting with different particle sizes, HA blasting, HA plasma spray coating, or etching), properties of different implant materials (different grades of commercially pure titanium or titanium alloys), surface roughness, surface energy, and contamination. Ti_6Al_4V Calcitite HA plasma spray coating surface had the lowest contact angle. This surface preparation exhibited surface roughness, which can improve wettability. In contrast, Ti_6Al_4V MP-1 HA plasma spray coating surface had the highest contact angle. From the information given by the manufacturer, MP-1 was a new surface preparation manufacturing process that increases the content of crystalline HA, which may affect the surface properties.

The surface properties of implants influence the biological response from the body.[15,16] When blood comes in contact with foreign solid surfaces, specific adsorptive interactions can initiate important events, such as the formation of a thrombus and blood coagulation. Two well-recognized processes may occur: the adsorption of proteins leading to coagulation and the adhesion of platelets, at first to the sol-

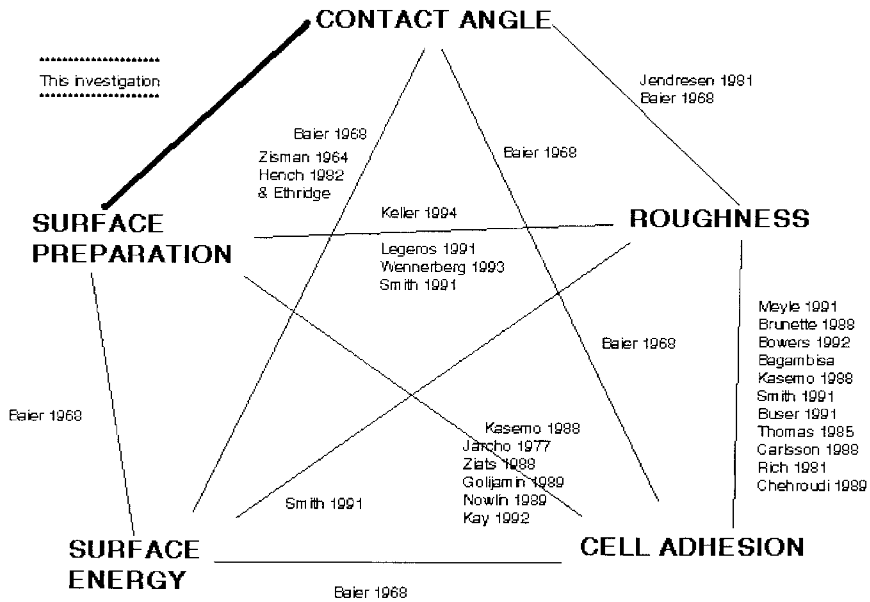

FIGURE 5. The relationship among contact angle, surface preparation, surface energy, roughness, and cell adhesion.

id and then to each other. The developing platelets form a thrombus, which adheres to the solid surface. Concerning the interaction of isolated cells from a variety of tissues with foreign surfaces, there seems to be general agreement that adhesion and spreading of cells can be a direct function of the solid surface energy.[11] The relationship among surface factors, including surface preparation, roughness, surface energy, contact angle values, and cell adhesion to biomaterial surfaces are presented in Fig 5. Each relationship was supported by a number of studies, which are referenced in the diagram (Fig 5).[3,4–7,10,11,17–35] The relationship between contact angles and surface preparations was determined in this investigation. The understanding of surface factors, cell adhesion, and their relationships are mandatory for better understanding of the bone-implant biomaterial interface.

Baier[36] examined the biomaterial interface characteristics of subperiosteal implants embedded in the dermal fascia on the back of rabbits. He found that the bioengineering correlation of biomaterial to tissue response could be evaluated by the use of contact angles or surface energy monitored by critical surface tension, and he concluded that changes in surface contact angles have direct effects on the biology and adherence of tissue to the implant surface. The chemical activity and surface energy of material surfaces is also influenced by surface topography. Numerous studies have demonstrated an influence of topography on cell migration and attachment.[18,19,23,37] Cell shape was influenced in many functions, and the proliferation of cells (based on surface topography) may be a controlling factor in dental implant success. Implants that are machined show varying degrees of roughness, which may be modified by subsequent etching, blasting, or coating treatments. Other preparative treatments, such as heat and thermal etching, may also influence the microarchitecture. The grooves, pores, and pits may favor bone adaptation and mineralization.[6,7] In several studies, rough surfaces were found to produce better bone fixation as compared with smooth surfaces,[20–22] and osteoblastlike cells exhibited greater tendency to attach to rough titanium surfaces.[20,38,39] Cells flattening and spreading have also been observed on both porous and dense hydroxyapatite.[21]

The optimization of implant surfaces in relation to surface energy and chemistry remains to be clarified. The effect of surface roughness on osteoblast metabolism or differentiation is also still unclear, and many studies on coatings (especially HA) have led to conflicting conclusions as a result of a lack of understanding of the actual surface composition.[35] Little information has been published on the precise surface composition, surface roughness, method of surface preparation, and cleanliness of commercially available implant systems.

CONCLUSION

Within the parameters of this study design, the following conclusions may be made:

- The surface preparation of implants affected wettability.
- Titanium alloy (Ti_6Al_4V) with a Calcitite HA plasma spray coating surface had the lowest contact angle and good wettability when tested with distilled water.
- Titanium alloy (Ti_6Al_4V) Calcitite HA plasma spray coating and CP titanium (grade 1) metallurgically polished surfaces did not have significantly different contact angles when tested with glycerol. A CP titanium metallurgically polished surface had significantly lower contact angles as compared with Ti_6Al_4V alumina-blasted surface, hydroxyapatite-blasted surface, MP-1 HA plasma spray coating surface, and CP titanium (grade 1)–etched surface.

ACKNOWLEDGMENTS

Presented at the International Association for Dental Research, 77th General Session, Vancouver, British Columbia, Canada, March 13, 1999. Supported by an Advanced Education Program Research Grant, Temple University School of Dentistry. Material support for this study was provided by Sulzer Calcitek Inc (Carlsbad, Calif) and Implant Innovations (Palm Beach Gardens, Fla).

The authors are grateful to Drs David C. Appleby, Brian Smith, Eugene Whitaker, and Kourosh Zarrinnia for their constructive criticism and suggestions. Thanks and appreciation are extended to Dr Sudarat Kiat-amnuay for her support and encouragement during the preparation of this paper.

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