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Contact Angle Measurement and Wettability of TiO₂ and ZrO₂ Nano Composite Coated on cpTi

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Abstract

Background: Dental implants have become one of the most reliable treatment options for replacing missing teeth due to their high success rates and long-term stability. Commercially pure titanium (cpTi) is widely used as an implant material because of its excellent biocompatibility, corrosion resistance, and its ability to achieve osseointegration with the surrounding bone. However, the biological performance of titanium implants is greatly influenced by surface characteristics such as surface roughness, surface energy, and wettability, which play an important role in protein adsorption, cell adhesion, and bone healing. **Aim:** The aim of this study was to evaluate the effect of a TiO₂- ZrO₂ nano-composite coating on the wettability of commercially pure titanium surfaces by measuring the contact angle. **Materials and Methods:** Commercially pure titanium discs were prepared and divided into two groups: a control group with uncoated titanium surfaces and an experimental group coated with TiO₂- ZrO₂ nano- composite. Surface wettability was evaluated using contact angle measurement by placing a droplet of distilled water on the surface and recording the angle using a goniometer. **Results:** The coated titanium surfaces showed a lower contact angle compared with the uncoated surfaces, indicating improved wettability and a more hydrophilic surface. **Conclusion:** TiO₂- ZrO₂ nano-composite coating enhances the wettability of titanium surfaces by reducing the contact angle, which may improve protein adsorption, cell attachment, and potentially enhance the osseointegration of dental implants.

Keywords: Contact Angle, TiO₂- ZrO₂ Nano- Composite, cpTi

Introduction

Dental implants have emerged as a reliable and predictable treatment option for restoring oral function in partially or completely edentulous individuals, as they provide a



stable foundation for various types of prosthetic restorations (Shaik et al., 2021 ; Prasanth et al., 2024). The long-term success of dental implants primarily depends on osseointegration, which is defined as the direct and intimate structural and functional connection between the implant surface and the surrounding bone tissue (Shaik et al., 2021; Matos, 2021).

Consequently, improving the quality and speed of osseointegration has become central focus contemporary implant research. In recent years, surface engineering has received increasing attention as a critical and natural advancement in implant technology aimed at enhancing implant– bone interactions (Boyan et al., 2016).

Of the range of surface properties, characterization of wettability has proven to be one of the most important intrinsic determinants influencing the biological behavior associated with implant materials. The patent description states that hydrophilic surfaces behavior better than hydrophobic surface in interactions with biological fluids and with proteins as well as cells, which means that a great cell adhesion will be promoted at early stages thus potentially accelerating the osseointegration process (Boyan et al., 2016).

Contact angle measurement is a widely used method to estimate surface wettability and yields useful information regarding the surface energy of solid materials as well as their compatibility with biological surroundings, particularly from the point of view of medical implants. Besides surface properties, one major factor of clinical performance is the variability in implant design. At present, over 1,300 systems of dental implants exist with differences in the form and dimensions, cores and surface materials, screw profiles and other physical characteristics such as shape or modification methods (Khalil & Sakr 2023).

That diversity reinforces the need for rigorous optimising of surface properties to realise the expected clinical benefits. Contact osteogenesis requires appropriate biological and surface-related stimuli to ensure this most-effective due process between with implanted material or device. The surface modification of titanium implant has been demonstrated to be critical in accelerating this process, signifying its role in the development and innovation of implants (Dahiya et al., 2014).

Surface modifications of titanium implants have been correlated to improved biocompatibility, improved bio-affinity with hard tissues as well as faster bone formation historically (Han et al., 2022). This notion of osseointegration has evolved, and current concepts suggest that bone tissue more effectively recognises modified titanium surfaces, allowing for faster accumulation of stable bone gap filling around the implant (Han et al. 2022).

Hence, the development of sophisticated surface modification techniques such as nano-composite coatings has been a hot topic. Contact angle measurement as a means of evaluating surface wettability is a well-established method of assessing the biological performance of coated titanium implant surfaces. The relationships between the wettability, bioactivity and implant–bone interactions on a surface level are important to optimize the implant surfaces for improved clinical success rates. Consequently, ongoing research continues to explore the fundamental mechanisms of osseointegration and the surface characteristics that contribute to its long-term stability and effectiveness.

Methods

Study Design and Setting

This study was an experimental in vitro comparative study carried out at the College of Dentistry, University of Kufa, Najaf in Iraq (from September 2025 to February 2026). This study aimed to assess the impact of titanium dioxide (TiO₂) and zirconium dioxide (ZrO₂) nanocomposite surface coating on wettability of commercially pure titanium (cpTi), according to contact angle values. Surface wettability is one of the main physicochemical properties affecting protein absorption, cell adhesion and osseointegration of dental implants.

To determine differences in contact angle between untreated cpTi surfaces (control) and cpTi surfaces coated by two proteomic deposition methods—dip coating (DIP) and electrophoretic deposition (EPD)—a comparative laboratory design was applied. A direct immune or biologic control group was not used since the principal aim of the study was to assess and compare physical surface characteristics among differently-treated titanium specimens in a laboratory setting.

Specimen Preparation

Circular discs (10 mm diameter × 2 mm thickness) of commercially pure titanium (cpTi) were prepared. The specimens were then mechanically polished with a stepwise increase in grit size of silicon carbide abrasive papers (400, 800, 1200 and 200) to achieve homogenous surface finish. Next, the discs were ultrasonically cleaned with different cleaning agents (acetone, ethanol and distilled water, with each agent lasting for 15 minutes) to remove contaminants and surface impurities before being air-dried in sterile condition.

Specimens prepared in a random manner were divided into 3 equal experimental groups (n = 10 specimens/group):



1. Control Group: non-treated titanium discs of commercially pure titanium
2. DIP Group: cpTi discs coated with TiO₂-ZrO₂nanocomposite via dip-coating process.
3. EPD Group: cpTi discs electrophoretic deposition coated with TiO₂-ZrO₂ nanocomposite.

Preparation of TiO₂-ZrO₂ Nanocomposite Coating

Coating suspension was prepared using titanium oxide and zirconium oxide nanoparticles (size range: 20–80 nm). The nanoparticles were mixed and dispersed in the ethanol-based solvent medium with magnetic stirring followed by 30 min of ultrasonic agitation to achieve a homogeneous dispersion while avoiding agglomeration.

In case of the dip-coating technique, titanium discs were submerged vertically in vertical manner at a fixed rate, maintained for 60 seconds with its subsequent withdrawal from nanocomposite suspension, and air dried at room temperature before sintering.

In the electrophoretic deposition technique, cpTi discs were employed as working electrode in a suspension including TiO₂-ZrO₂ nanoparticles (diluted particles) dispersed. A direct electric field (20–30 V for 2 minutes) was applied to promote the deposition of nanoparticles on the titanium surface (air dried followed by sintering, booth conditions help with better adherence of coating and also structural stability).

The coated specimens were then subjected to a heat treatment at 450–500°C for 1 h under controlled conditions in order to increase the coating crystallinity and bonding with titanium substrate.

Contact Angle Measurement

A digital contact angle goniometer was employed to evaluate the surface wettability using the sessile drop method. At controlled laboratory temperature (25 ± 1°C), and humidity ≤10% with a micropipette, a standard droplet (5 µL) of distilled deionized water was meticulously placed on each specimen surface.

After placing the droplet in contact with a solid surface, high-resolution digital images of the droplet profile were acquired and analyzed via image analysis software to yield the liquid contact angle formed as defined between the liquid droplet touching towards a specimen (a) surface. Each specimen was measured three times at different locations, and the average value for each

specimen was taken to reduce measurement variance. When lower contact angle values were present, it was observed signifying increased hydrophilicity and wettability.

Statistical Analysis

IBM SPSS Statistics for statistical analysis. A data were expressed as mean ± standard deviation (SD). Values of the mean contact angle were compared among the three groups with one-way analysis of variance (ANOVA), with Tukey's post hoc multiple comparison test for pairwise group comparisons. P value

< 0.05 was taken as statistically significant, while P value < 0.001 was considered highly significant.

Ethical Considerations

Because this investigation was essentially a laboratory-based in vitro materials study with no human subjects or biological samples, patient consent was not required. The experimental procedures were primarily carried out in accordance with the laboratory biosafety and research standards approved by the scientific committee of the College of Dentistry, University of Kufa.

Results

The Control group exhibited the highest mean contact angle (42.799° ± 0.675), indicating a more hydrophobic surface. The EPD group showed the lowest mean value (24.088° ± 0.844), suggesting a significantly more hydrophilic surface. The DIP group presented intermediate values (27.998° ± 0.835), indicating improved wettability compared to the Control group, but less than the EPD group. Additionally, the relatively low standard deviation (SD) values across all groups indicate: high consistency of measurements, good reliability of the data (table 2). The large F-value (F = 1420.5) indicates that the variation between groups is substantially greater than the variation within groups. This confirms that the observed differences in contact angle are not due to random variation but are a direct result of the different surface treatments applied (table 1).

Figure (1) depicts example sessile-drop images used to determine the contact angle on untreated and nanocomposite-coated cpTi surfaces and visually demonstrates that surface wettability differs between the experimental groups. The control surface (a) showed the lowest contact angle (42°) and was observed to present a fairly spherical droplet profile with relatively low spreading on substrate surfaces, presenting a more hydrophobic and low wettability surface nature. Conversely, EPD-coated specimen (b) exhibited the least contact angle of 24° with the water droplet spreading significantly more on the substrate surface,



indicating significantly enhanced surface hydrophilicity along with superior wettability. Compared with the untreated control (b), the contact angle of the specimen coated via dip coating (c) was intermediate (~28°), suggesting that the wettability increase in the former was less significant than that achieved with electrophoretic deposition. The visual evidence of these observations further corroborates the quantitative contact angle data presented in Table (3.1) that

the incorporation of TiO₂-ZrO₂ into the cpTi matrix greatly alters the surface energy properties of cpTi. The better hydrophilicity, mainly in EPD group can be due to a higher surface homogenization, higher nanoscale surface roughness and higher surface free energy, all three of them have been proved to favor: proteins adsorption, more impressive osteoblast attachment and better biological integration of titanium-based dental implants.

Table 1. A descriptive statistic of contact angle values for all groups

Groups	Mean±SD	F Test	P value
Control (n=10)	42.799 ± 0.675	1420.5	< 0.001*
EPD (n=10)	24.088 ± 0.844		
DIP (n=10)	27.998 ± 0.835		

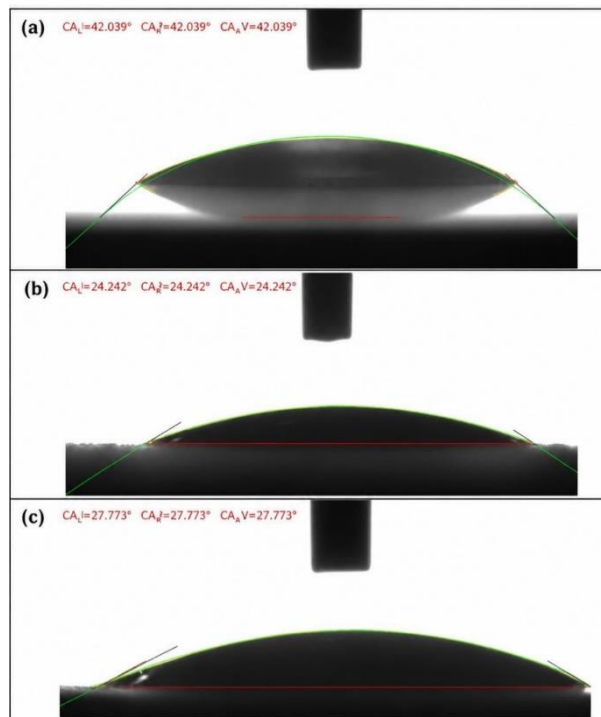


Figure 1. Sessile-drop contact angle measurements for commercially pure titanium (cpTi) surfaces are shown as follows: (a) an unmodified control surface, (b) a surface coated with a TiO₂-ZrO₂ nanocomposite using electrophoretic deposition (EPD), and (c) a surface coated with a TiO₂-ZrO₂ nanocomposite using dip coating (DIP).



Discussion

Firmly evidence indicates that the physicochemical properties of dental implant surfaces represent some of the most important characteristics for their biocompatibility and the clinical success over decades. Notably, surface wettability (which is traditionally characterized by contact angle measurements), being one of the key features to determine the immediate interaction between biomaterials with biological fluids, proteins or host cells shortly after implantation. Less contact angle means higher surface hydrophilicity and thus it helps in facilitate the wetting by blood and tissue fluids, improving protein adsorption which in turn triggers cellular attachment that together causes initiation and rapid osseointegration (Rupp et al 2014). We found that commercially pure titanium (cpTi) surfaces plated with TiO₂-ZrO₂ nanocomposites had greatly improved wettability over uncoated control surfaces, indicating perhaps the first example of how a nanostructured surface treatment can impart positive changes in Titanium behavior in physiological conditions. As a result, they exhibited lower relative activity than their uncoated counterparts Yesigat et al (2006) reported the highest mean contact angle ($42.799^\circ \pm 0.675$) for untreated control specimens, suggesting that these are relatively hydrophobic surfaces when compared to the coated groups. This result is a cohesive finding in respect to the well-established surface characteristics of polished cpTi, where also a chemically persisting oxide film will appear over time on its metallic surface which is biocompatible in general, but may not always be able to provide sufficient hydrophilicity for immediate biological interaction. Additionally, untreated or conventionally polished titanium surfaces are known to have moderate wettability due to limited nanoscale roughness and surface free energy (Le Guéhennec et al., 2007). While they may still support osseointegration, the initial biological response is frequently slower compared to chemically or nanostructurally modified materials.

The EPD group had the lowest contact angle ($24.088^\circ \pm 0.844$), indicating that it was the most hydrophilic of all experimental groups. The dip-coating (DIP) group, on the other hand, yielded contact angle values read at $27.998^\circ \pm 0.835$ with respect to control value showing a statistically significant decrease but also less pronounced than that achieved via EPD (Tab. The results clearly demonstrate that the coating of cpTi surfaces by TiO₂-ZrO₂ nanoparticles can greatly enhance the wettability, and that coating substrate is primarily influenced by the deposition technology itself. Such results have also been described for surface-engineered coatings based on nanoparticles that drastically reduced contact angle values (Lavenus et al., 2011) owing to the influence of their modifications in terms of surface topography and chemistry. Nanostructured coatings provide

micro/nanoscale hierarchical structures that promote capillary spreading of the liquid droplet and increase the surface free energy for enhanced hydrophilicity, such as surface structures with alternating grooves or protrusions.

This improvement in wettability may derives from the intrinsic physiochemical characteristics of titanium dioxide (TiO₂) and zirconium dioxide (ZrO₂). TiO₂ is a well-known hydrophilic and photocatalytic material, with appropriate surface conditions enabling the generation of hydroxyl-rich surfaces that exhibit high attraction to water molecules, thus reducing contact angle (Diebold, 2003). These hydroxyl groups enhance polar surface interactions and enable rapid liquid spreading, On the other hand, ZrO₂ provides advantageous surface energy properties (Hisbergues et al., 2009), superb chemical stability and corrosion resistance as well as mechanical reinforcement. Therefore, incorporation of TiO₂ and ZrO₂ into a nanocomposite coating may exert synergistic effects due both to surface chemistry and topographical roughness producing superior wetting properties when compared with untreated titanium.

Among the most important conclusions derived from this research is that EPD coating +/- Dipping methods are significantly higher than dip coat formulations. The electrophoretic deposition of metallic coatings over electrodes is primarily based on the phenomenon that charged nanoparticles migrate in an electric field, which allows for relatively fast, and thick deposits with a dense and homogenous structure. This method provides precise control over the thickness of the coating, packing density and surface coverage of particles which could explain the significantly lower contact angle observed in the EPD group (Boccaccini et al., 2010). In contrast, dip coating is predominant on gravitational withdrawal and solvent evaporation processes, which produces small superficial imperfections of the film such as agglomeration of nanoparticles or irregularity on the thicknesses of films. Therefore, although DIP showed a much better wettability than the control for a good surface quality, it did not reach that of EPD in obtaining the most hydrophilic surface.

The statistical data also provides strong evidence in support of these observations. The between groups one-way analysis of variance (ANOVA) was extremely significant (P 999, suggesting that the variance shown across experimental groups separated the probability distribution much more than within-group variability. This further substantiates the thought that more substantial random shifts of the touching angle ranged are an effect of true effects arbitrarily created by a method to treat surfaces employed and never sporadic variabilities in the experiment or small random mistakes. In addition, the low standard deviation in all groups suggests a high level of measurement consistency and reproducibility and augments the present results.

The increased loading of hydrophilic site on the implant has



significant implications for success at the biological and clinical level. Once implanted, the biomaterial surfaces come in immediate contact with blood proteins such as fibronectin, vitronectin, fibrinogen and albumin creating a conditioning layer which dictates following cellular responses. There is faster and more favorable adsorption of adhesion proteins in biologically active conformations on hydrophilic surfaces, which accelerate osteoblasts attachment, spreading, and proliferation (Zhao et al., 2005). Moreover, concerning the hydrophilic implant surfaces, enhanced platelet activation Angiogenesis and early bone matrix deposition have been shown to contribute to accelerate osseointegration (Buser et al., 2004). Thus, the significant decrease in contact angle found especially for EPD-coated samples indicates a possibility of improved biocompatibility which could lead to quicker healing times and better implant fixation in the clinical scenario.

However, a number of limitations warrant consideration. This investigation was conducted in an internal laboratory setting with a controlled environment using distilled water as the wetting medium. The study of contact angle measurement provides useful information concerning surface hydrophilicity, but does not simulate all the variables present in the oral environment (e.g. saliva proteins and bacterial biofilm, mechanical loading and dynamic pH changes), which influence surface behavior. In addition, biological performance is governed by a series of properties including wettability, surface chemistry (also called charge), roughness, crystallinity and coating adhesive strength, corrosion resistance and bactericide. Hence, surface characterization methods like scanning electron microscopy (SEM), atomic force microscopy (AFM), X-ray diffraction (XRD) and energy-dispersive spectroscopy (EDS) should be integrated in future works along with cell culture and in vivo animal studies to provide an overall evaluation of the biological potential of TiO₂-ZrO₂ nanocomposite coatings.

Conclusion

In the present study, surface hydrophilicity of TiO₂ and ZrO₂ coated commercially pure titanium (cpTi) surfaces was assessed by measuring contact angle and reported in terms of wettability. The results showed that the wettability of titanium was improved by the surface coating compared to uncoated control group, as indicated by the significant decrease in contact angle values after surface modification. Epoxy coatings are applied utilizing the research area, which is substantially broader than revealed in this overview and employs a wide variety of coating techniques. Within the investigated methods, electrophoretic deposition (EPD) provided for the greatest degree of wettability improvement and dip-coating remained capable of

attaching a far more hydrophilic topography to polymer surface. Meanwhile, control substrates exhibited the highest values of contact angles, implying less surface wettability and a more hydrophobic nature. Coating of TiO₂-ZrO₂ nanocomposite might successfully change titanium surface to more hydrophilic interface which could contribute to the initial biological events that enhance osseointegration and further clinical outcome of Ti based dental implants.

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