Effect of ultrasound surface activation on the surface roughness of titanium dental implants

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ABSTRACT

Background: In dental implant, the surface treatment is used to modify surface topography and surface energy, resulting in an improved biocompatibility, increased cell proliferation and growth, and accelerated osseointegration process. Objective: the use of ultrasonic surface treatment. To form submicro- and nanocrystalline states on the surface of titanium implant. Results: The implant sample was affected by ultra-sonic energy and heterogeneous surface morphologies were observed. Conclusion: Using ultrasonic treatment caused a change in surface properties due to the mechanical effects of ultra-sonic on the samples surfaces (topography, composition, and roughness) depending on the initial surface properties that result from the primary surface activation procedure.

KEYWORDS: dental implant, ultrasonic surface treatment, surface morphologies

INTRODUCTION

The use of bone-anchored titanium implants has become routine treatment modalities in dentistry. The success of implants is highly dictated by the surface properties of the implant material that influence molecular interactions, cellular response and thereby, bone regeneration. Mesenchymal stem cell involvement, cell-cell communication at the bone-implant interfaces and in particular interactions between the surface oxide and the biological host are the underlying mechanisms of osseointegration[1]. Surface modification is a process that changes a material’s surface composition, structure, and morphology, leaving the bulk mechanical properties intact. In addition, metals with bio functions have been required in the recent past. In dentistry, dental implants require hard tissue compatibility for Osseointegration and bone formation, soft tissue compatibility for adhesion of gingival epithelium, and antibacterial property for the inhibition of biofilm formation. These bio functional properties consist of two conflicting properties: the inhibition and enhancement of protein adsorption or cell adhesion. When a metallic material is implanted into a human body, immediate reaction occurs between its surface and the living tissues. With surface modification, biofunction of surface layer could be improved. For these purposes, many techniques for surface modification of metals are attempted [2]. Chemical treatment of titanium and its alloys are mainly based on chemical reactions occurring at the interface between titanium and a solution [3].

Acid treatment is often used to remove oxide and contamination to obtain clean and uniform surface finishes. A combination of acids is frequently used to pre-treat titanium. In acid etching, the use of acids on metal surfaces is not only to clean the surface but also to modify the roughness. In addition, the acid treatment
was often used to combine other treatment methods to improve the properties of titanium and its alloys. Wen et al. reported that the bioactivity of Ti alloy could be improved by two-step chemical treatments employing (HCl + H₂SO₄) and alkaline solution [4]. And Wanye Tan et al. studied the use of ultrasonic irradiation (UI) along two different routes to obtain several modified surfaces on titanium plates; the first group was first treated by a NaOH solution, and then UI is used to wash them in double distilled water; while the second were modified by a NaOH solution in an ultrasonic cleaner with UI at 50 W. It was demonstrated that the UI energy can easily remove any weakly bound layers (WBL) on the titanium surface, leaving a strongly bounded layer (SBL) [5]. Modification of surfaces using ultrasonic treatment leads to significant changes in surface properties, and most of the effects of interest regarding ultrasonication are related to cavitation. Cavitation causes solute thermolysis along with the formation of highly reactive radicals and reagents, such as hydroxyl radicals and hydrogen peroxide, which induce drastic reactive conditions in the liquid media. In addition, if a solid is present in solution, the sample size of the particles is diminished by solid disruption, thereby increasing the total solid surface in contact with the solvent. In this way, ultrasonication remains unique, since no other method of sample treatment can produce such effects [6, 7]

**Experimental:**

Preparation of implant samples involved using two different methods in order to make comparison between them and to study the effect of manufacturing process on the surface roughness which may affect the biological behavior of the implant in the body by elimination of healing period (rapid osseointegration). The first method involved the use of commercial pure titanium rod. The rod was converted to implant screw by machining. The second method included the use of powder technology in order to produce the implant screws with some porosity. The surface state of an implant alters the bone response and fixation. In present stage surface treatments have been done in order to change the surface characteristics such as surface composition and topography. The surface activation stage was divided into two steps primary and secondary. The primary surface activation treatment involved chemical surface treatments (acid and alkali etching) in order to precipitate some elements to make more effective surface. Acid etching in HCl was done because HCl is an excellent decontamination agent, it could easily dissolve titanium salts and not weaken Ti surfaces before alkali treatment. While the purpose of using NaOH was to improve the bioactivity of titanium surface. At first and before the treatment all implant samples were ultrasonically cleaned using ultrasonic cleaner type in ultrapure water for 10 minutes then dried in an oven at 40°C for 15 minutes. After the samples were cleaned they were immersed in HCl acid with concentration 0.5mM for 90 minutes at 40°C after that the samples were washed with ultrapure water and dried in an oven for 15 minutes. Then the samples were immersed again in NaOH with concentration 10 M for 24 hours at 60°C then washed in ultrapure water and dried in an oven for 15 minutes. In order to obtain the etching temperature and maintain the samples at this temperature. After this step the acid and alkaline treatment process were complete and the samples was ready for the secondary treatments. The purpose of using this type of treatment as a final surface activation process is to improve topographical characteristics of the surface and to get a final strong active surface layer that can make the implant more active and safe inside the bone. The ultrasonic surface treatment involved the utilization of the energy resulting from the ultrasonic wave. This energy is considered to be the main source of chemical and mechanical effects on the implant surface by creating Transient bubble collapsing on the implant surface which is act to change the surface topography. The source of the ultrasonic wave used was the ultrasonic cleaner path type (KQ3200E), samples was immersed in ethanol and then placed inside a path of water as a physical medium in which the ultrasonic wave was transmitted. The samples remain in the ultrasonic cleaner for five hours then they were removed and washed in ultrapure water and dried in an oven at 40°C for 15 minutes. After this treatment the final surface topography was obtained and the sample was ready for surface characterization. The characterization was made on implant sample that Organized and categorized in groups as illustrated in table (1).

**Table 1:** sample groups.

<table>
<thead>
<tr>
<th>Group number</th>
<th>Surface treatment procedure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group A</td>
<td></td>
</tr>
<tr>
<td>A1: prepared by powder technology</td>
<td>none</td>
</tr>
<tr>
<td>A2: prepared by machining</td>
<td></td>
</tr>
<tr>
<td>Group B</td>
<td></td>
</tr>
<tr>
<td>B1: prepared by powder technology</td>
<td>Acid and alkaline treatment followed by ultrasound treatment</td>
</tr>
<tr>
<td>B2: prepared by machining</td>
<td></td>
</tr>
</tbody>
</table>

The characterization that have been in order to examine implant samples in terms of the best surface treatment method which produced the preferable surface properties and in order to ensure the safety performance of the implant inside the body in which the implantation process have been done. The characterization includes microstructure characterization, surface chemical composition analysis (EDS), surface roughness (AFM), and in vivo test results (histological analysis).
RESULTS AND DISCUSSION

Microstructure Characterization:

The microstructure observation was done using the scanning electron microscope device to see the effect of surface treatments on surface texture. Figures (1) and (2) show the surface morphologies of master samples (group A). These two samples differ in manufacturing process but both of them are without any surface treatment. There are some inconsiderable morphological differences between samples (A1) and (A2) as a result of the manufacturing process. For sample (A1) produced by powder technology it obvious to have a degree of porosity which result in roughen surface than sample (A2) which was produced by machining. The tool marks created by the turning process made the surface anisotropic with clear directional surface irregularities. Bioactivity of titanium implant surfaces were improved by the utilization of two-step chemical treatment (acid and alkaline etching), which resulted in the formation of sodium titanate hydrogel on the titanium substrate. As found in Jonšov L.[8]. The acid etched of implant surface presents a superficial morphology. From The acid etching of samples with HCl, a uniform initial titanium surface with the formation of TiO₂ oxide layer was observed. During the alkali treatment, the TiO₂ layer partially dissolves in the alkaline solution because of the attack by hydroxyl groups.

\[
\text{TiO}_2 + \text{NaOH} \rightarrow \text{HTiO}_3 \cdot + \text{Na}^+ \\
\text{Ti} + 3\text{OH}^- \rightarrow \text{Ti(OH)}_3^+ + 4\text{e}^- \\
\text{Ti(OH)}_3^+ + \text{e}^- \rightarrow \text{TiO}_2 \cdot \text{H}_2\text{O} + \frac{1}{2}\text{H}_2 \uparrow \\
\text{Ti(OH)}_3^+ + \text{OH}^- \leftrightarrow \text{Ti(OH)}_4 \\
\]

A further hydroxyl attack on the hydrated TiO₂ produces negatively charged hydrates on the surfaces of the substrates as follows:

\[
\text{TiO}_2 \cdot \text{H}_2\text{O} + \text{OH}^- \leftrightarrow \text{HTiO}_3 \cdot + n\text{H}_2\text{O} \\
\]

These negatively charged species combine with the alkali ions in the aqueous solution to produce an alkaline titanate hydrogel layer. During heat treatment, the hydrogel layer is dehydrated and densifies to form a stable amorphous or crystalline alkali titanate layer. It was found that the thickness of the precipitated apatite layer increased continuously with time and the treatment of titanium by a two-step HCl and subsequent NaOH process appears to be a suitable method to enhance the surface bone bonding ability. The using of ultrasound treatment after initial chemical etching caused heterogeneous surface morphologies. From the SEM image of samples B1 in figure (3) and B2 in figure (4), it can be seen more clearly that there are significant changes in the surface irregularity after ultra-sonic irradiation which refers to change in surface morphology. This is due to the mechanical effect of ultra-sonic on the samples surface, in ultrasonic treatment the force is applied by formation of cavitation bubbles which considered being the main source of the chemical and mechanical effects of ultrasonic energy.

![Angstrom advanced](image-url)

**Fig. 1:** SEM image of sample A1
Surface chemical composition analysis:

The analytical technique that has been used for the elemental analysis or chemical characterization of implant surface before and after the surface treatment was done by using Energy-dispersive spectroscopy EDS.
EDS graph of the master samples (A1, and A2) in figure (5) and (6) show a large peak of titanium element without showing any other peaks of elements which refer to samples with high degree of purity and the manufacturing process will not affect or result in a change of the surface chemical composition. For samples (B1) and (B2) the EDS show wide variety elements peaks as illustrated in figures (7) and (8), which resulted from the dual surface treatment (acid and alkaline followed by ultrasonic treatment). The primary acid and alkaline treatment resulted in formation of sodium titanite hydrogel that caused the appearance of (Cl),(Na) and,(O) peaks and the ultra-sonic treatment caused appearance of carbon peak.

From the results of the surface chemical composition analysis of the ultra-sonic treatment group it was found that using the ultrasonic as a second surface treatment do not change the surface chemical composition resulted from the primary treatments. The change will occur at the surface topography and roughness.

Fig. 5: EDS graph of sample A1

Fig. 6: EDS graph of sample A2
Fig. 7: EDS graph of sample B1

Fig. 8: EDS graph of sample B2

Surface roughness investigation:
This test was done in order to identify the topographic change after the surface treatments as well as the amount of roughness that have been produced after the treatments from micro to Nano scale in two or three dimensions of the surface texture. The test was carried out using Atomic Force Microscope (AFM). Figures (9) and (10) display the roughness values of the master group. It can be observed that sample (A1) which was produced by powder technology process without any surface treatment have higher roughness (700.26 nm) than sample (A2) produced by machining without any surface treatment (527.06 nm). Hence the powder technology process produced samples with higher surface roughness compared to the machining process. After dual surface activation the roughness was raised, first there was large decrease in surface roughness of the samples treated primarily by acid and alkaline etching due to the formation of smooth sodium titanate hydrogel layer on the surface. But the mechanical effect of the ultrasonic energy raised the surface roughness of samples (B1) with roughness (845.36 nm) and (B2) with roughness (531.7nm)in figures (11) and (12),because that there are two types of nanostructure layers formed on the titanium surface after NaOH treatment; the weakly bound layer (WBL) and the strongly bound layer (SBL). The strongly attached to the titanium substrate. The pore diameter of the WBL is about two times larger than that of the SBL. Moreover, the WBL can be easily removed from the SBL by using UI, which may not promote the connection between host tissue and the titanium implant after implantation, and could in fact be detrimental to the surrounding cells due to the release of Na+ from the sodium titanate formed on the titanium surface [8].
Fig. 9: AFM graph of sample A1

Fig. 10: AFM graph of sample A2

Fig. 11: AFM graph of sample B1
Conclusion and recommendations for future work:

1- The using of different manufacturing process (machining and powder technology) produce topographical differences, the topographical change that observed from powder technology method was more than the machined one.

2- Employment of primary surface activation process as chemical acid alkaline etching produced initial surface roughness that aid surface for subsequent treatment by formation of sodium titanate hydro gel layer on samples treated by acid and alkaline etching reduces the surface roughness and produced bioactive surface layer.

3- Large topographical improvement was observed after the using of the second surface activation treatment get a final strong active surface layer that make the implant more active and safe inside the bone.

4- The using of ultrasonic treatment cause change in surface properties due to the mechanical effect of ultrasonic on the samples surface (topography, composition, and roughness)

5- there are two types of nanostructure layers formed on the titanium surface after NaOH treatment; the weakly bound layer (WBL) and the strongly bound layer (SBL) the WBL can be easily removed from the SBL by using UI.

Finally there are recommendations for future such as studying the effect of porosity percentage that obtained from using the powder technology as manufacturing method on surface properties and osseointegration process, Using the powder technology to prepare Ti base alloy for dental application with addition of alloying elements such as Si, Mo, and Cr in order to eliminate the Ti percentage and reduce the cost. Using of ultrasonic treatment to precipitate some nano particles on sample surface by using the particles suspension as a physical medium.

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