



## Effect of Acid and Alkali Etching on Titanium metal Implant Devices for Bone Infections

Baskaran Vaidhyanathan<sup>1</sup>, Sekaran Saravanan<sup>1\*</sup>

<sup>1</sup> Department of Prosthodontics, Saveetha Institute of Medical and Technical sciences (SIMATS), Saveetha Dental College and Hospital, Chennai, Tamil Nadu, India

\*Corresponding Author

S. Saravanan, Department of Prosthodontics, Saveetha Institute of Medical and Technical sciences (SIMATS), Saveetha Dental College and Hospital, Chennai, Tamil Nadu, India.

Email id: [ranklogp@gmail.com](mailto:ranklogp@gmail.com)

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### Abstract

External augmentation is often necessary to support bone formation in critical sized bone defects. There are many metals used for bone regeneration. Titanium (Ti) metals and its alloys are widely used for bone and dental implant devices. The major advantage of the titanium metals is that they show excellent corrosion resistance and good hard-tissue compatibility. Despite this Pure Titanium performs inert in nature biologically in clinical and medicine. To overcome this limitation, Titanium metal can be modified using both acid and alkali etching. Surface morphology is critical for improving Ti metals physiological characteristics. The goal of this research is to evaluate titanium etched performance using intense hydrochloric acid and sodium hydroxide. So, the metal has been modified, surface morphologies were periodically checked and the treatment methods were performed rapidly to discuss the biological properties of the modified metal. The effects on the Acid Etching Treatment and the metal's suitability for use in bone tissue engineering (BTE) technologies were explored. The metals are very porous and have the capacity to freeze, adsorb proteins, and biomineralize.

Keywords: Titanium metals, Acid, Alkali, Sodium Hydroxide.

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### 1. INTRODUCTION

Because of its superior mechanical qualities and biocompatibility, in the fields of dental and bone domains, the Ti metals and their related ions have been used [1,2]. One of the major contributing factors of these issues is its low biological activity. As a result, enhancing the biosynthetic pathway of Ti implants has emerged as an emphasis of Ti medical research [3,4,5]. The surface treatment of those Ti metals will also improve the osteoinduction and osteoconductivity of the bone nature in the dental implants. Mechanical, physical, chemical, and biological approaches are commonly used for Titanium surface intensities [6-9]. Among these approaches, alkali heat treatment (AHT) has recently emerged as an intriguing method for micro-/nano-scale functional alteration of Ti surfaces [10,11]. Although AHT can generate a sodium titanate coating with bioactivities on the surface of Ti, the apatite forming capabilities of the Ti surface by AHT is unsustainable in high temperatures circumstances.

Various approaches, such as further water treatment, acid pretreatment, and acid-base treatment, are being investigated to further explore the potential of AHT and maximise the biosynthetic pathway of the coating [12-14]. Despite their advantages, these strategies still are inadequate for enhancing HA reliability. Acid etching treatment (AET) is frequently employed on Ti before AHT as an efficient preparation.

This AET and AHT Ti has great biological features. AET and AHT, when compared to AHT, can enhance the abrasion and hydrophilicity of the Ti surface. The production cycle of HA on the AET and AHT Ti surface was quicker than on the AHT-Ti surface, and osteocytes demonstrated better cytocompatibility on the AET and AHT Ti surface [15].

Induction heating treatment (IHT) is a method for fast oxidation. Although its use in the surface functionalization of hard metallic implants is uncommon, its applicability hopes in the medicinal area are extensive. When a Ti-based implant is subjected to IHT, the epidermal phenomenon makes the Ti surface to rapidly warm up, making IHT technology acceptable for the area of surface functionalization of metallic alloys [11,16]. The TiO<sub>2</sub> layer can form quickly during IHT, enhancing the amount of TiO<sub>2</sub> on the interface of Ti. TiO<sub>2</sub> has been widely employed as a valuable resource in photocatalytic activity and other disciplines [17,18].

Furthermore, TiO<sub>2</sub> has been proven to always have strong metabolic processes and cytocompatibility; it can generate HA and calcium transportation, and it can certainly increase osteoblast adhesion. As a result, producing TiO<sub>2</sub> oxide coating on Ti surface by IHT is an excellent way for improving its physicochemical functions [19,20]. IHT will also alter the microstructure of the material at elevated of the Ti substrate. AET, to the contrary side, is a viable approach for Ti surface modification. AET can alter not only surface quality but also surface shape and chemical composition, endowing Ti with bone-like micro/nano structure. The impact of AET upon that surface of materials is mostly determined by the acid solution's type and intensity [21,22].

In order to explore more things in the etching properties, the Acid and Alkali etching were concentrated more and the experiments were performed accordingly and the results were deeply interpreted and investigated in this paper.

## 2. HYPOTHESIS:

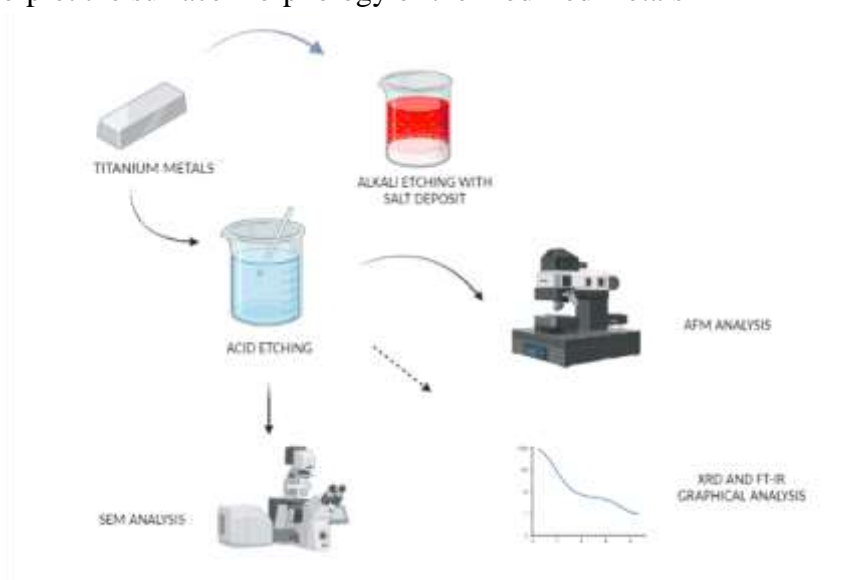
Titanium (Ti) metal possess amino groups (-NH<sub>2</sub>) and hydroxyl groups (-OH) in their backbone which allows for various modifications. It is already reported that Titanium metals has the ability to chelate with Alkali and Acid etching and we hypothesize that, by exploiting this property and chelating with a metal ion by etching method is capable of osteogenic functions will improve the property. In addition to this, the free amino and hydroxyl groups will be involved in metal chelation and unavailable for hydration. This will ultimately lead to an increased mechanical strength and prolong the persistence *in vivo* by controlling its hydrolytic degradation.

Titanium is the selected metal ion of choice which will be complexified with bones. It is found to increase Wnt signaling, osteoblastogenesis via the blockade of nuclear factor kappa B (NF- $\kappa$ B). It is also known for its osteoclasts suppressive activity and promotes its apoptosis. The rationale of our proposal lies in the selection of this particular composite because there is no previous report/study/literature on this combination.

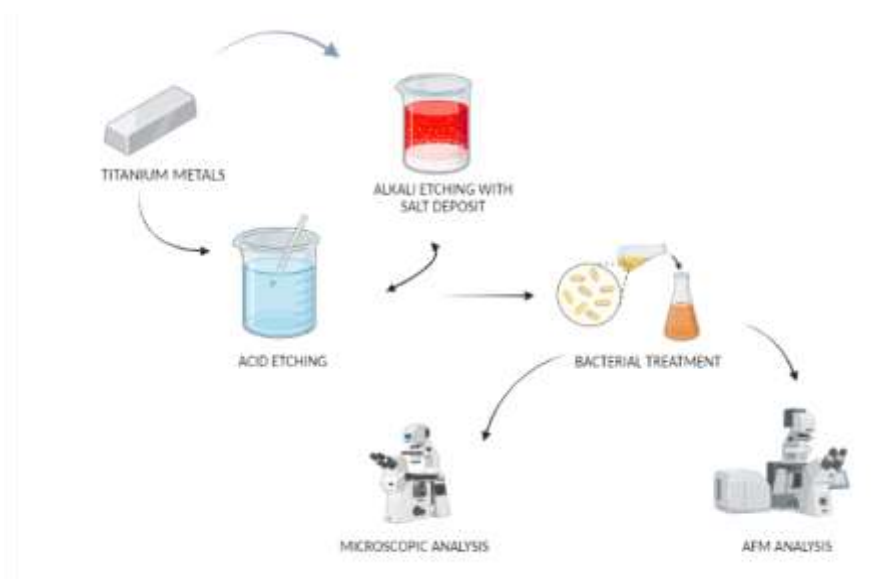
### 3. OBJECTIVES:

To assess our hypothesis, a new composite will be fabricated and tested for bone tissue engineering applications. The following objectives are proposed to test our hypothesis:

1. To modify the metal using acid and alkali etching
2. To characterize the metal using SEM analysis and other characterization techniques
3. To interpret the surface morphology of the modified metals



**FIG 3.1.** ETCHING OF METALS FLOW PROGRESS



**FIG 3.2.** ETCHING OF METALS AND BACTERIAL TREATMENT FLOW PROGRESS

## 4. METHODOLOGY:

### 4.1. MATERIALS AND PRETREATMENT:

The commercial pure titanium metals were taken and grated into square discs of 10x10 mm and 3 mm width. The metals were cleaned properly with acetone followed by ethanol and ultrapure water respectively. Later, It has been incubated at 37°C. Then subjected to the acid and alkali etching

### 4.2. TITANIUM METAL MODIFICATION:

#### 4.2.1. ACID ETCHING:

The cleaned Ti metals were taken properly and it has been treated with acids. The suitable acids for Ti metals are  $H_2SO_4$  and HCl. The metals were treated with 1:1:2 ratio of  $H_2SO_4$ , HCl and ultrapure water. Later the metals were immersed in the acid concentrations at the pH of 4.5 and later treated at 70°C for 1h. Later, the metals were subjected to the characterization techniques.

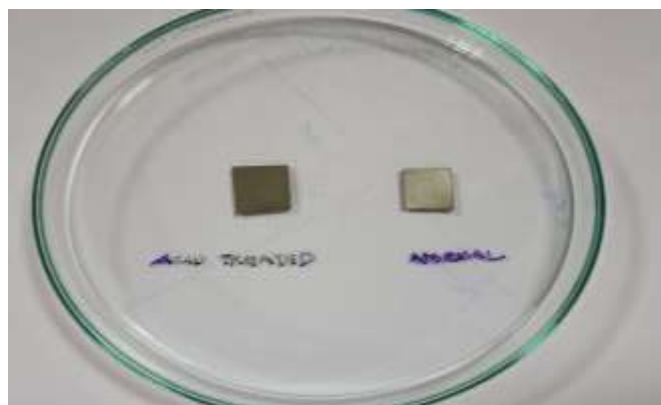


FIG 4.1. ACID TREATED WITH REFERENCE METAL

#### 4.2.2. ALKALI ETCHING:

The cleaned Ti metals were taken properly and it has been treated with NaOH. 10g of NaOH pellets were weighed and it was added in 100mL Double distilled water. The NaOH crystals liberated heat while it was mixing and the metals were immersed in the NaOH Solution at the pH of 12. It was incubated at 60°C overnight and the metals were subjected to the characterization techniques.



FIG 4.2. ALKALI TREATED METAL AND SALT DEPOSIT

#### **4.3. ACID FOLLOWED BY ALKALI:**

The cleaned Ti metals were taken properly and it has been treated with acids. The suitable acids for Ti metals are  $H_2SO_4$  and HCl. The metals were treated with 1:1:2 ratio of  $H_2SO_4$ , HCl and ultrapure water. Later the metals were immersed in the acid concentrations and later treated at 70°C for 1h. Then it was subjected to Alkali treatment with the NaOH crystals, The NaOH crystals liberated heat while it was mixing and the metals were immersed in the NaOH Solution. It was incubated at 60°C overnight and the metals were subjected to the characterization techniques.

#### **4.4. ALKALI FOLLOWED BY ACID:**

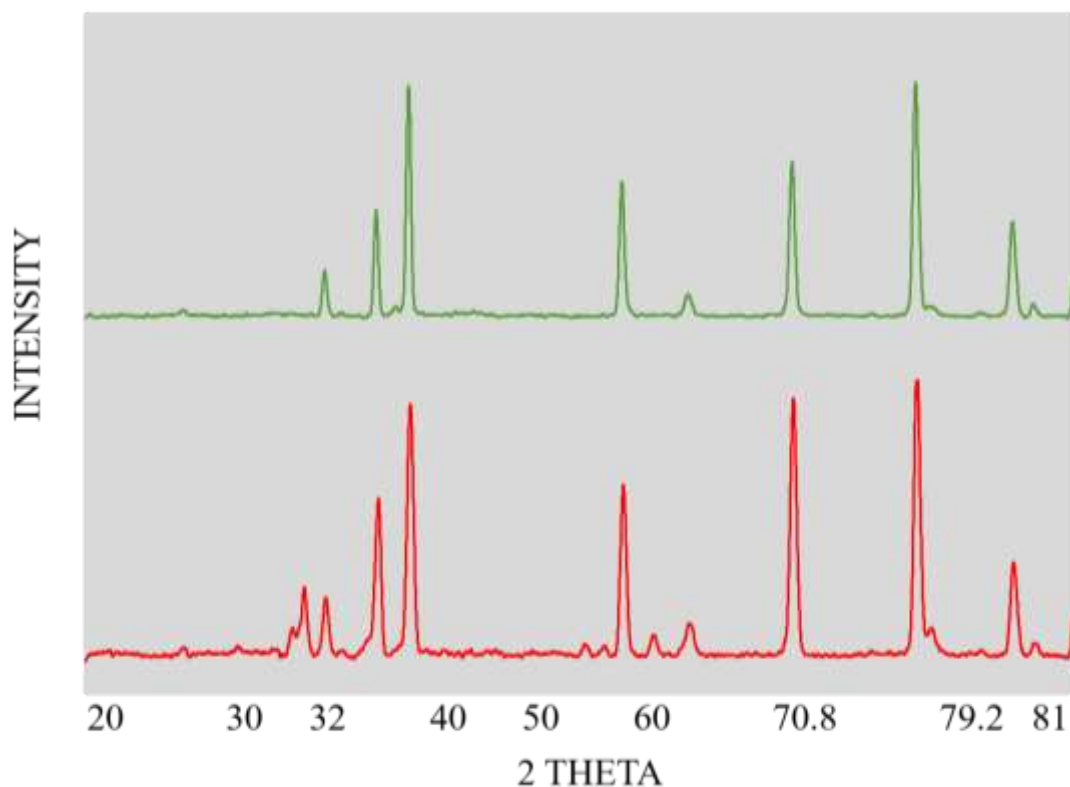
The cleaned Ti metals were taken properly and it has been treated with NaOH. 10g of NaOH pellets were weighed and it was added in 100mL Double distilled water. The NaOH crystals liberated heat while it was mixing and the metals were immersed in the NaOH Solution. It was incubated at 60°C overnight and subjected to acid treatment. The metals were treated with 1:1:2 ratio of  $H_2SO_4$ , HCl and ultrapure water. Later the metals were immersed in the acid concentrations and later treated at 70°C for 1h. Later, the metals were subjected to the characterization techniques.

#### **4.5. Ti MODIFIED METALS TREATED WITH BACTERIA:**

After treating the metals with Acid and Alkali etching, the treated metals were incubated and later treated with *Staphylococcus aureus* bacteria. This bacterium usually occurs in the teeth bones. For the reason, the suitable bacterium was selected and the metals have been treated appropriately. The bacteria were streaked quadrantally and it was incubated at 37°C. Later a small bacterium was picked up from the developed colony and it was grown in broth for 24 h. then the metals were incubated for 24h in the broth and it was magnified in 100X in light microscope.

### **5. RESULT AND DISCUSSION:**

### 5.1. MICROSTRUCTURE AND SURFACE MORPHOLOGY:

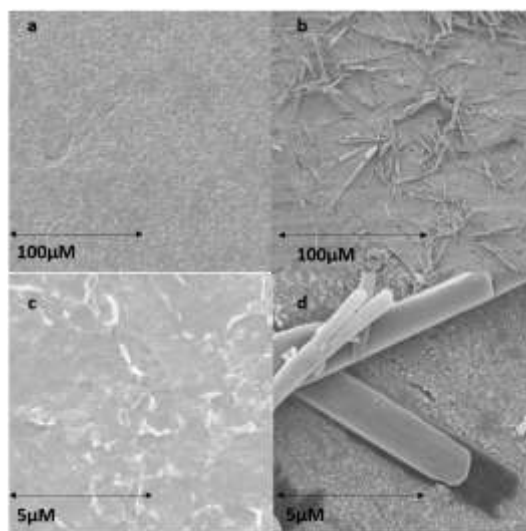


**FIG 5.1.** XRD ANALYSIS OF THE METALS

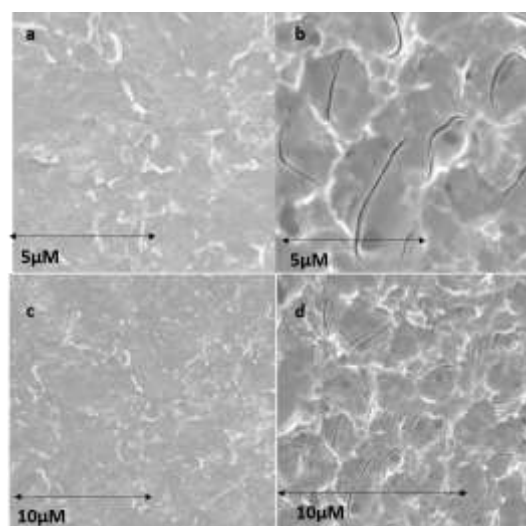
The green pattern indicates reference and red pattern indicates alkali treatment of Ti metals. The most similar peaks show that there is no change in the amorphous nature of the metal after treatment. But, the excessive peaks at the 30 2theta deg and 65 2theta deg indicates that the deposition of Na was successful and it forms NaTi gel. This indicates that the NaOH was deposited frequently at the favorable temperature 60°C. Also, XRD indicates the Stress factor and the deposition of the salts at 65 2theta deg with the less variable peaks and interprets that the NaTi gel was formed on the surface was very much thinner and cannot be given easily in the terms of XRD. In this case the gel formed in the metal because of alkali etching has been discussed on SEM characterization, was gradually thin in the formation, that cannot be easily detected in the XRD Patterns.

The figure 5.2 was observed with the scale of 100 micrometers and 5 micrometers. Comparing with the reference, the deposition shows that the Ti metal was coated with sodium properly and it also formed NaTi gel with major blunts shown at fig b After AHT. The metals were easily allocated with the formation of sodium particles in it. Later in fig d it also shows the blunt space was crucial for roughening the pattern of the titanium metal. The common property of shifting of Na ions with the Ti ions has been taken place successfully because of the high impression of NaOH in Ti metals with the help of heat fixation. The heat fixation method suggests that the major impact of the surface roughness has been created. The figure 5.3 was observed in 10 micrometer and 5 micrometer scale. This study clearly shows that the

strong acids were reacted with metals to the extent. From the analysis, the excessive cracks were formed and pores were created due to the high temperature of the acid concentration ( $\text{H}_2\text{SO}_4$  and  $\text{HCl}$ ) and also due to the rapid increase in the temperature ( $70^\circ\text{C}$ ). This property is because of addition of the two acids and taking it in the high degree. This shows the mechanism formation of shift of Ti ions to the  $\text{HCl}$ . This formation is taken place with the latent heat of vaporization; hence the formation of excessive cracks has been seen. These cracks show that the metal surface has been modified properly, hence the modification of the surface morphology proves that the roughness of the surface is much higher than the normal Ti metal. NaTi Gel formation have the major criteria of better biological activity also in the high humid environment.



**FIG 5.2.** SEM ANALYSIS OF METALS WITH REFERENCES (a), (c) AND METALS WITH ALKALI TREATMENT (b), (d)



**FIG 5.3.** SEM ANALYSIS OF METALS WITH REFERENCES (a), (c) AND METALS WITH ACID TREATMENT (b), (d)

## 6. CONCLUSION:

For the quick biological signaling of Ti surfaces, a reinforced treatment approach of acid and alkali is used. The AET-treated Ti has a coarser and more consistent permeable network model than the references, which results in good corrosion and wear resilience. The work of the AHT samples is mainly of NaTi and magnetite  $TiO_2$ , resulting in a great capacity to increase hydroxyapatite production. Furthermore, the long-term sustainability of Hydroxyapatite generated by AET and AHT is greater than that of conventional Ti metals, demonstrating that AET and AHT are superior surface activation treatment methods for obtaining a more suitable biological coating. Hence this type of techniques can be suggested for the bone tissue engineering applications.

### Author Statements:

The raw/processed data required to reproduce these findings cannot be shared at this time due to legal or ethical concerns

### CREDIT AUTHOR STATEMENTS:

**BASKARAN VAIDHYANATHAN**- Investigation, Writing- Original draft preparation, **POORANIAMMAL BALU**- Investigation, Writing- Original draft preparation, **SEKARAN SARAVANAN** and **GNANADHAS DIVYA PRAKASH** - Conceptualization, Resources, Supervision, Funding acquisition.

### CONFLICTS OF INTEREST:

The authors declare that they have no conflict of interest.

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