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STUDING THE NANOMETIC FEATURES OF COMERCIAL PURE TITANIUM AFTER THERMOCHEMICAL ETCHING

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Abstract

Titanium surface modifying for the implants by chemical and physical methods generally used with the aim of achieving better osseointegration. This study examines the effects of thermochemical treatments on titanium's nanoscale surface. The physical properties of the surface and topography of CPTi are influencing the outcomes of implant surgery. Method: Three implants surface prepared: first group untreated CpTi (control), second group surface modified by etching with H_2SO4 acid within 80C° thermal application, and 3rd. group surface modified with HCL and 80C° thermal application during acid etching. The surface characterized by SEM, AFM, and wettability test analysis of data was done by using statistical SPSS v 22 Result: higher roughness 25.1nm shown when etching performed by H2SO4 with 80C heating for 10min, while the surface roughness was 17nm when etched with HCL under same conditions. The wettability test revel increase in hydrophilicity when H2SO4 used for etching and the contact angle reduced to 56.55 degree. Conclusion: thermochemical etching is an effective surface modification technique that can enhance the properties of commercial pure titanium. The modified surface layer results in enhanced surface roughness, and improved wettability. These properties make the material suitable for various industrial and biomedical applications.

Keywords: Acid etching, commercial pure titanium, surface topography, Nano morphology.

1. Introduction

In the field of oral restoration dental implants are broadly utilized, but issues like marginal bone resorption, failed osseointegration, and peri-implantitis continue to limit patient fulfilment and the achievement rate of dental implants in clinical setting (Al Najjar and Al- Adili 2019). The two primary causes of implant failure are inadequate osseointegration and bacterial infection. (Barfeie, Wilson, and Rees 2015)Titanium's exceptional physical characteristics, such as its strong fatigue strength, low elasticity module, and great resistance to corrosion, have made it a popular material for dental implants (Kadhim, Hamad, and Fatalla 2022; Muhammed, Al-deen, and Faculty 1994). When exposed to air, a thick 4-nm layer of titanium dioxide (TiO2) forms spontaneously, giving it additional outstanding biocompatibility (Becker et al. 2000). One of the most significant events in the early stage of osseointegration is the increased calcium deposition caused by this layer, which leads to the primary adsorption of adhesive proteins such as albumin or glycosaminoglycans (Brett et al. 2004; Cho and Park 2003), since the high level of activity in titanium, when the metal is exposed to air, a stable layer of TiO2 forms on its surface instantly. This coating acts as a passive shield to prevent corrosion on the titanium. However, titanium's passive coating becomes unstable in a reducing atmosphere, which can lead to corrosion. To alter the titanium surface, several acids have been use (Cochran et al. 1996), and Biological applications have been made of some of them. Studies have shown that when titanium is sprayed with titanium plasma, sandblasted and acid-etched titanium (SLA) facilitates higher osseous contact earlier. (Cochran et al. 1996)

There have been various innovative surface treatments reported to accomplish these goals in recent years. The foundation of these therapies are either chemistry or physics (Junker et al. 2009). For instance, various treatments, including as blasting, acid etching, electrochemical and thermochemical treatments, plasma spray, and its combinations, have been used to alter the surface roughness and topography of titanium (Ting et al. 2016). The CpTi surface bioactivity has been improved and the period of dental implants osseointegration has been shortened by the combination of acid etching, management in a hydrogen peroxide – hydrochloric acid mixture, and other thermochemical usages [9]. The vast majority of implant surfaces that have been produced and studied, including SLA (big grit sandblasted with acid-etched) (Data and Willse 1995).

In biomaterials research studies this field are rarely reported, despite the attraction of etching CpTi in concentrated acid for creating novel surface alterations for biological uses (Al-Khateeb, Al-Hassani, and Jabur 2023). The previous studies revealed that etching in a concentrated acid lead to a CpTi titanium surface (Barfeie, Wilson, and Rees 2015). This study evaluated the possibility of surface modification of CpTi for topographical purposes and characterized the etching behavior of CpTi in a concentrated acid, for modifying the surface of titanium implants chemical and physical methods have been generally used with the aim of achieving better osseointegration (Cruz et al. 2020). The purpose of this study was to spot the thermochemical etching effect, namely the etching rate for commercial pure titanium dental implant material in concentrated acid with heating and recognize the nanoscale surface topographical changes under different period and spot the size of nano-pore that formed afterwards etching was about 100–500 nm.

Dental implants Surface roughness

Numerous studies have shown that the ratio of osseointegration and biomechanical fixation are effected by the roughness of titanium implants surface (Feiruz and Mahmood 2021). Depending on the size of the features, there are three degrees of surface roughness: macro, micro, and Nano-sized topologies.

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For topographical characteristics, the macro level was determined to be between millimeters and tens of microns. With threaded screws and macro porous surface providing a roughness above 10µm, this scale is directly associated with implant geometry. studies have demonstrated that a high roughness profile, as opposed toward smooth surfaces, can enhance the prosthesis early attachment and long-term mechanical stability (Jafarkhani et al. 2023). The implant surface and bone development mechanically interlocked because of the extreme roughness. High surface roughness, however, carries a significant risk of increasing ionic leakage and peri-implantitis. For these two characteristics, a modest roughness of 1-2 µm may be the limit. (Becker et al. 2000)

The micro topographic profile of dental implants can be definite the surface roughness being in range of 1–10µm. This roughness range maximizes the interlocking between the implant's surface and the growing bone. (Rosa et al. 2012). Theoretically, hemispherical pits measuring around 1.5 m in depth and 4 m in diameter should cover the perfect surface. (Jafarkhani et al. 2023). the use of an implant with a rough surface influenced by the primary clinical reason the poor quality or none favorable volume of the host bone (Farhan, Jassim, and Thair 2020).High loading might be possible in these unfavorable clinical circumstances if there was early and significant bone-to-implant contact. Rough-surfaced, short-designed implants have shown better clinical results than smooth surfaces in situations where there is not enough bone or when there are anatomical constraints. (Conner et al. 2003). Abundant studies have shown that surface roughness in 1-10 µm range resulted in superior bone-to implant contact and greater resistance to torque removal strength than other types of surface topography (Wennerberg et al. 1998). However, the Cochrane relationship has not found in any clinical evidence demonstrating the superiority of any particular implant surface type (Esposito, Ardebili, and Worthington 2014).

Nanoscale surface profiles are critical for protein adsorption and osteoblastic cell adhesion, and they have an impact on the rate of osseointegration. (Brett et al. 2004). However, it is challenging to achieve consistent surface roughness in the nanoscale range using chemical treatments (Nasser and Abass 2020). Furthermore, it is uncertain whether surface nanotopography is best for selectively adsorbing proteins that cause osteoblastic cells to adhere and quickly assemble into bone. A number of techniques have been developed to increase the osseointegration of titanium dental implants by roughening their surface. In these techniques, thermal acid etching is performed on commercially pure titanium as a substrate.

Roughening of implants by acid

Strong acids such as HCl, H2SO4, HNO3, and HF were utilized for etching titanium dental implants. Acid etching creates tiny pits on titanium surfaces ranging in size from 0.5 to 2m. (Zinger et al. 2004). Acid etching has been demonstrated to significantly improve osseointegration. (López-Valverde et al. 2023). dual acid etching of Titanium implants were studied by dipped for a few minutes into a strong HCl and H2SO4 solution that had been heated to about 100 °C to produce a tiny surface roughness This type of surface expedites osseointegration during a three-year period while maintaining long-term success.(Jassim and Shukur 2015. Dual acid-etched surfaces have been proven to improve osteoconductive recovery. This were accomplished by enabling fibrin and osteogenic cells to adhere to the implant surface, resulting in direct bone production (Velasco-Ortega et al. 2021). These investigations proposed the theory that implants subjected to dual acid etching have a unique topography that enables attachment to the fibrin scaffold, encourages osteogenic cell adhesion, and consequently encourages bone apposition (Gautam, Yadav, and Bansal 2022). Acid-etching techniques have recently been developed to stimulate bone growth and cell adhesion. It has also been proposed

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that the wettability of the surface promotes fibrin adhesion. When the osteoblasts made contact with the surface, they were directed by the fibrin attachment. An experimental study found that a hydrophilic surface greatly enhanced bone and implant contact in minipigs when compared to standard sandblasted and acid-etched implants. The chemical treatment may be able to improve implant anchoring in bone by making the implant surface bioactive(López-Valverde et al. 2023). However, chemical reactions may degrade titanium's mechanical properties. Acid etching, for example, may promote hydrogen embrittlement of the titanium, resulting in surface microfractures that may reduce the fatigue resistance of the implants (Kligman et al. 2021). In fact, In experimental studies, titanium has been demonstrated to absorb hydrogen in a biological context. Titanium's ductility is reduced as a result of the brittle hybrid phase that emerges from hydrogen embrittlement. This issue is linked to dental implant fracture mechanisms (Yokoyama et al. 2002).

Material and method

The substrate for surface modification experiments was CpTi discs grade II. These discs were cut from rode by wire cutting machining into small disc pieces (15mm diameter, 2mm) and utilised in this work to test surface properties and in etching studies. The specimen was directly polished to achieve a mirror finish surface using a grinding and polishing machine, with polishing beginning with 240, 320, 400, and 600 grit abrasives. All grinding processes, including abrasive cutting, should be done wet with water to avoid damaging any microstructure components. Wet grinding reduces specimen heating and keeps the abrasive surface from being loaded with metal taken from the specimen being prepared (Elssner et al. 2009). Ultrasonic cleaning in ethanol for 15 minutes, followed by washing in distilled water for 10 minutes, before drying the plates at room temperature, eliminated debris and pollution.

Etching used to create a surface with micro-to-nano-sized pores. A scanning electron microscope used to analyse the surface morphologies of the discs. (Field emission scanning electron microscope, FESEM, JSM-6701F, FEI, USA), AFM and wettability (contact angle) CAM 110, Germany. Plates were classified into three groups: non-modified CpTi (mirror polished from the source), CpTi surface modified by H2SO4 98% concentration etching at 80 Co for 10 minutes, and surface modified by HCL etching with boiling at 80C for 10 minutes.

Characteristic Analysis Following Acid Etching

In this article, the weight loss was tracked for 10 minutes to obtain the etching rates of CpTi at high temperatures and two distinct acids to understand the etching rate of two acids at 80Co temperature on CpTi. The etching rates of CpTi for various etchant solutions 80C° are shown in Table 1. The etching rate of HCl for CpTi was 0.459 mg/min at BT, and the etching rate of H2SO4 for CpTi was 0.637 mg/min at 80Co, as shown in the table. Etching with H2SO4 at high temperature causes greater weight loss, indicating a higher rate of disintegration of the surface structure when compared to etching in HCL.(Alzubaydi et al. 2009)

Table 1 mean values for the weight loss rate and t-test

groups	N	Min	Max	Mean	Std.	Sig
HCL	10	.44	.47	.4536	.00947	0.000
H_2So_4	10	.63	.66	.6447	.00993	

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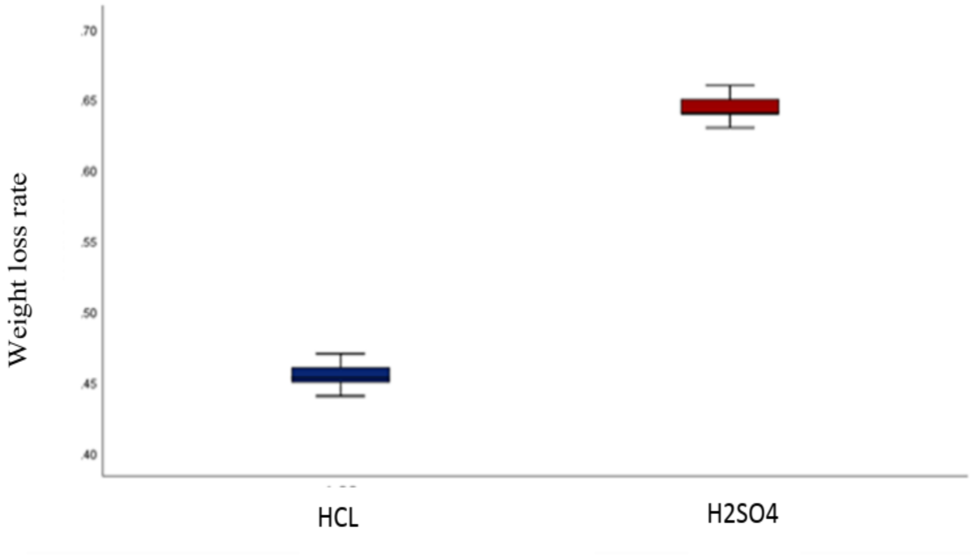

Figure 1 box plot or weight loss rate in mg/min for HCL AND H2SO4

3. Results and discussion

3.1 SEM surface features

SEM pictures of the CpTi surface following acid etching (H2SO4 and HCl) were taken to better understand the influence of high temperature etching on the morphology and topography of the etching. SEM micrographs show that the CpTi samples had many uneven projections and a rough surface, as seen in Figure 2. the groups of etched surfaces using H2SO4.and HCl Both specimens were etched for 10 minutes and heated to 80 c°. After etching at a higher temperature, Nano-holes were created. Acid etching with H2SO4 at 80 c°and HCl at 80 c°may produce consistent micrometer-nanometer 3D holes. Accordingly to Diana Patty These holes aid in the deep penetration of osteoblast members and improve implant stability post-implantation due to their consistent surface and significantly deep spiral structure (ewi Ana and Yusuf 2022). In addition, the formation of micro and Nano holes has great effect in increase surface area which play important role in enhance osseointegration(Jafarkhani et al. 2023).

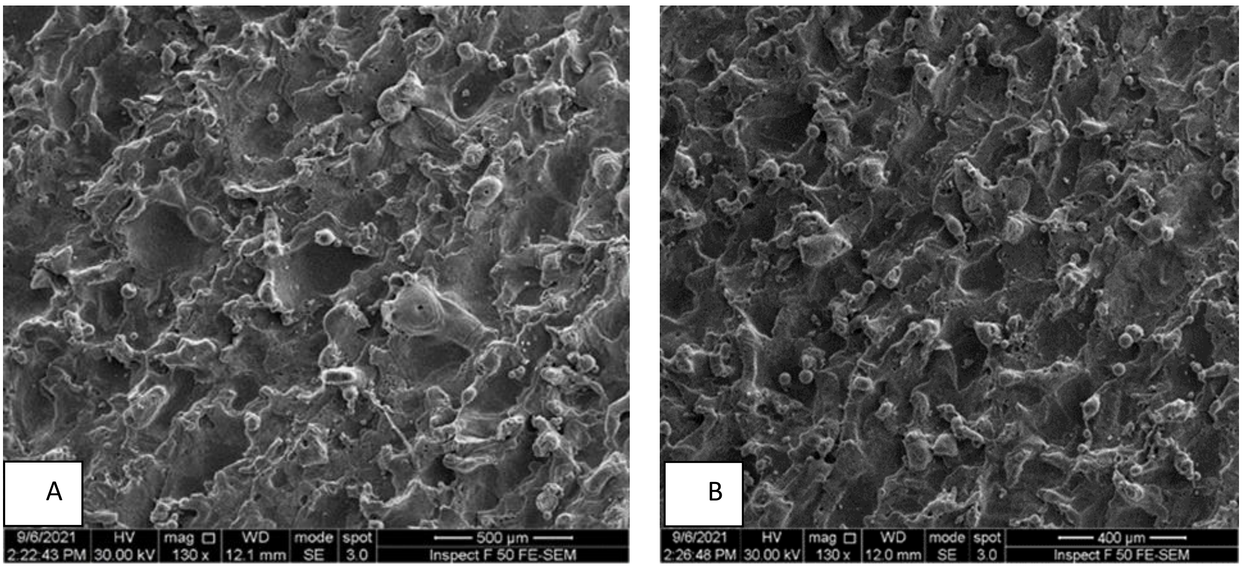

Figure 2 SEM micrographs A. H2SO4 B. HCL magnification 3000kv

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3.2 AFM Surface roughness analysis

Figure 3. Show Mechanical-polishing groves on with the average roughness untreated surface 7.37 nm and considered a smooth surface, the roughness of the treated CpTi by thermal etching with H2SO4 shown in Figure 3. B, several embedded valves with an average roughness of 25.1 nm are seen in the picture, and Figure 3. C shows the specimen that underwent thermal HCL etching had projections and peaks with an average roughness of just 17 nm. Nanoscale measurements of the AFM topographic roughness were made. Figure 4 box plot show that after etching, the results increase in Nano roughness when the surface was treated with thermal H2SO4 etching, as seen from the peaks that appeared on the surface of treated CpTi . the ANOVA test conducted for all groups and the result was statistical significant and multiple comparison Bonferroni between each two groups conducted the results was highly significant between all studied groups , the result accepted with Madrigano et al 2008 that the increase in nanoroughnss related to rate of weight loss because as the rate increase more pits will formed on the surface of CpTi (Madrigano 2008), which has an important role in increase adsorption protein to the surface of the implant and accelerating the process of osseointegration(Im et al. 2023) .

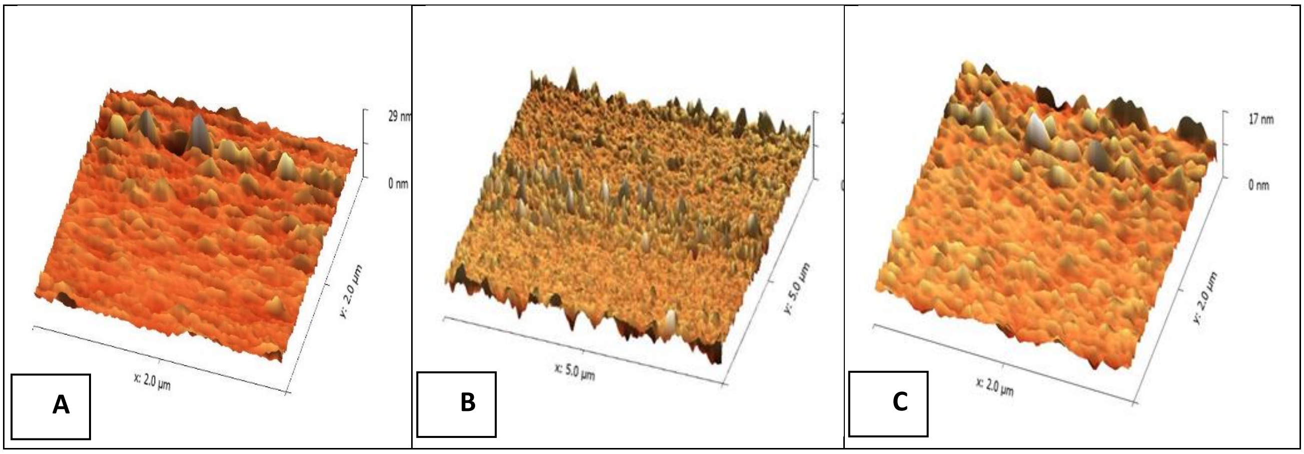

Figure 3 AFM topography for a. non-treated CpTi b. treated CpTi with H2SOU at 120Cc. treated CpTi with HCL at 80c

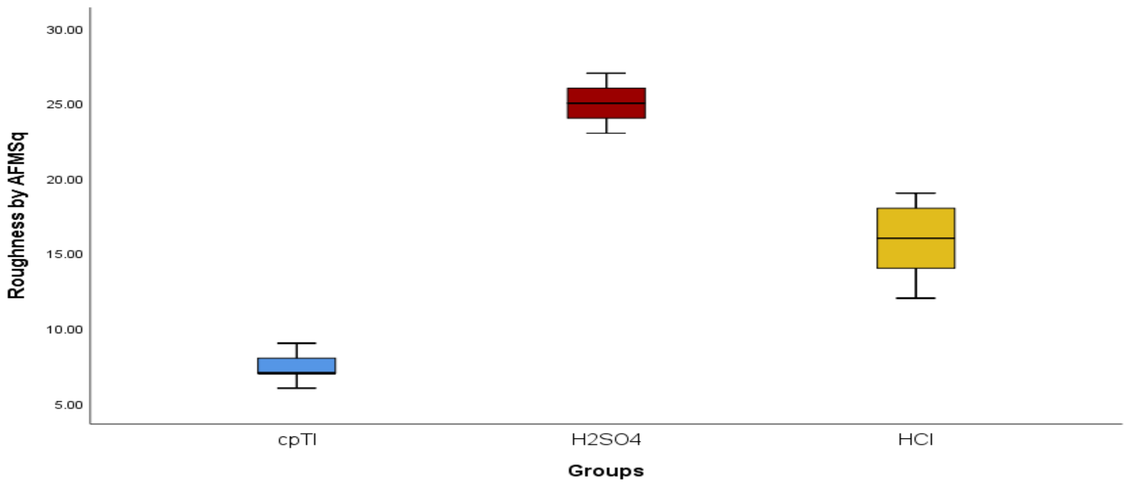

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Table 2 mean values for roughness by AFM Sq and ANOVA test

Goups	N	Min	Max	Mean	Std.	ANOVA test
cpTi	10	6.00	9.00	7.4000	1.07497	267.41
HSO4	10	23.00	27.00	25.1000	1.19722	0.000
HCl	10	12.00	19.00	16.7000	2.49666	

Figure 4 Box plot for data of roughness by AFM Sq

Table 3 Multiple Comparisons bonferroni for data of roughness by Sq AFM

(I) VAR00002	(J) VAR00002	Mean Difference (I-J)	Sig.	95% Confidence Interval	
				Lower Bound	Upper Bound
cpTi	H2So4	-17.70000*	.000	-19.6590	-15.7410
	HCl	-8.48889*	.000	-10.5016	-6.4762
H2SO4	HCl	9.21111*	.000	7.1984	11.2238

3.3Wettability

The contact angle, which is the angle between the solid surface at the three-phase contact line and the tangent to the liquid vapor interface, is a regularly used metric to describe wetting. (Katyal et al. 2021) (Feiruz and Mahmood 2021). Investigation of the wettability for different etching acids conducted. Figure 6 shows mean contact angle 76.77 degrees for none treated smooth CpTi suggests moderate hydrophobicity; H2SO4 etched surface with 80C° heating Surface has a mean contact angle of 56.55 degrees indicates a relatively high level of hydrophilicity; HCL etched Surface at 80C a mean value of contact angle of 64.32 degrees . In table 4 ANOVA test conducted for all studied groups the p value indicate highly significant and multiple comparison Bonferroni test done between each two groups and the results was significant difference. The increase in hydrophilicity of surface related to increase in Nano roughness of the CpTi that etched by H2SO4 because of increase Nano pits this agreed with Im, et al 2023 (Im et al. 2023).

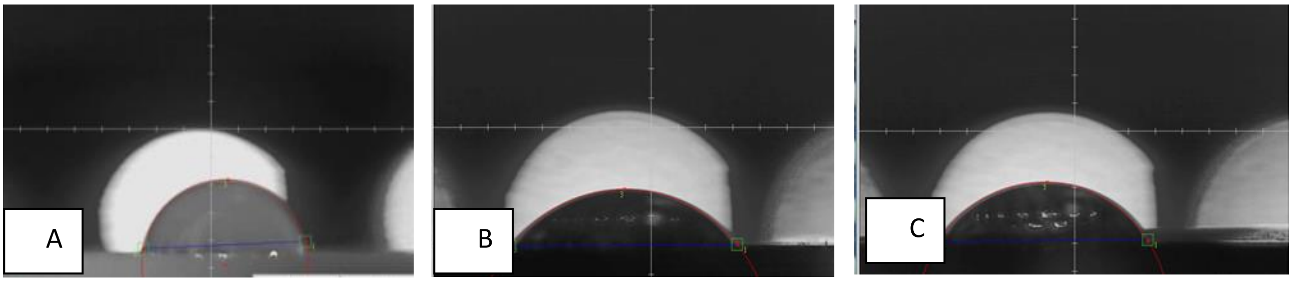

Figure 5Wetability test A. NON-treated CpTi B. H2SO4 C. HCL Treated CpTi

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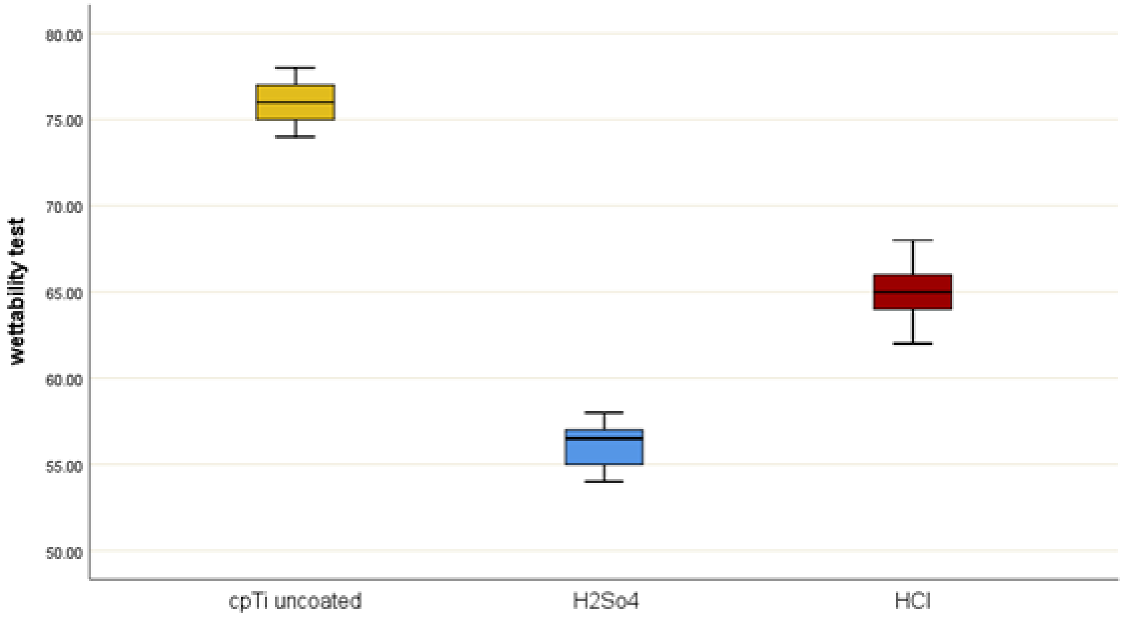

Figure 6 mean value of contact angle for all studied groups

Table 4 Mean value for contact angle measurements for all studied groups

groups	N	Min	Max	Mean	Std.	ANOVA
cpTi	10	74.00	78.00	75.800	1.31656	398.677
H2So4	10	54.00	58.00	56.200	1.22927	P-value
HCl	10	62.00	68.00	65.400	2.01108	0.000

Table 5 multiple comparison Bonferroni test for comparison between mean of each two studied group for contact angle measurement

(I) VAR00002	(J) VAR00002	Mean Difference (I-J)	Sig.	95% Confidence Interval	
				Lower Bound	Upper Bound
cpTi	H2SO4	19.60000*	.000	17.8221	21.3779
	HCl	10.57778*	.000	8.7511	12.4044
H2So4	Hcl	-9.02222*	.000	-10.8489	-7.1956

4. Conclusion

Surface roughness increases when commercial pure titanium is etched thermochemical. The development of pores and micro cracks on the titanium's surface is what causes the roughness. The surface's increased roughness enhances cell and tissue adhesion, which qualifies it for use in biomedical applications. After thermochemical etching, the surface energy of commercial pure titanium was increased, which enhances its wettability. The improved wettability of the material allows for better adhesion of coatings or biomolecules to the surface. This is particularly useful in the biomedical field where surface modification of titanium is required for better cell adhesion and proliferation.

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