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Surface characterization analysis of failed dental implants using scanning electron microscopy

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Abstract

Objective. To investigate the failure of 15 dental implants (Paragon/Zimmer) in relation to their surface quality. ***Materials and methods.*** The study comprised of 15 dental implants (7 mm D Advent Implant, 3.9 mm D apex design implant), which were followed from surgery to completion of prosthetic restorations. The implants were placed during a 6-year period from 2003–2009 in non-smoking patients (male; 7, females; 5). There were eight upper and seven lower implants. Surface characterization after immersion in SBF of these failed implants was investigated using SEM and EDS compared to that of an unused implant of the same brand. ***Results.*** Results revealed that, following immersion in SBF, the implant surfaces showed new components like Ca^{+}, Na^{+} and Cl^{-}, but in trace quantities. ***Conclusions.*** After SEM observation and EDS analysis, it was concluded that the apatite layer formation could not be verified.

Key Words: *titanium implants, dental implants, surface morphology*

Introduction

In the past 20 years, the number of dental implants has increased steadily worldwide, reaching about one million dental implantations per year [1]. In Sweden, osseointegrated implants became acceptable in 1977 [2] and international acceptance followed the Toronto conference held in 1982. Behind this acceptance were positive long-term clinical results mainly from treatment of totally edentulous patients. Since 1984 more than 80,000 implants have been placed in American patients and 1,200,000 patients worldwide [3]. Endosseous dental implants have created a revolution in the routine approach to dental care for patients missing one or more teeth [4].

Clinical success of dental implants occurs through a series of clinical and biological steps affected by the implant material and its morphology. The majority of biomaterials present in clinical use are interactive materials, i.e. implantable materials designed to elicit specific, beneficial responses, such as growth and adhesion [5]. Titanium metals and its alloys are used in dental and orthopedic implants on account of their excellent corrosion resistance, biocompatibility and osseontegration behaviour [6]. Commercially pure titanium has various degrees of purity (graded from 1–4). This purity is characterized by oxygen, carbon and iron content. Most dental implants are made from grade 4 cpTi as it is stronger than other grades [7]. These materials are known to have a combination of good properties, making them particularly relevant and suited for biomedical applications. Among these properties are their low specific weight, high strength-to-weight ratio and the low modulus of elasticity [8].

Implant design refers to the 3-dimensional structure of the implant, with all the elements and characteristics that compose it. Form, shape configuration, surface macrostructure and macro-irregularities are terms that have been used in the literature to describe the aspects of the structure. Endosseous dental implants exist in a wide variety of designs [9], with the main objective in every instance being the long-term success of the osseointegrated interface and uncomplicated function of the prosthetic replacement. Moreover, the use of updated technologies for implant surface modifications has become a marketing trend in the production of new implants, creating various morphologies as well as chemistries [10]. It is now believed that, apart from

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the rough surface texture, the specific topography of the implant surface influences qualitatively and quantitatively its integration [11]. Topographical features of implants defines the macro level as being in the range of millimeters to tens of microns. This scale is directly related to implant geometry, with threaded screw and macroporous surface treatments giving surface roughness of more than 10 μm [12]. A goal of current implantology research is design of devices that induce controlled, guided and rapid healing. More specifically, in addition to acceleration of normal wound healing phenomena, implants should result in an interfacial matrix with a composition and structure characteristic of bone, and the matrix should have adequate biomechanical properties [13]. Success of dental implants is related to their early osseointegration, geometry and surface topography, surface treatment besides other factors such as surgery technique, host bone quality and load bearing [1]. After implantation, titanium implants interact with biological fluids and tissues with subsequent bone apposition onto the surface of titanium. This direct bone apposition is critical for rapid loading of dental implants [14]. The long-term success of implant therapy is not just dependant on enhanced osseous stability but greater attention is being addressed to the transmucosal dental implant or implant abutment interfaces [15,16].

Despite high success rates, implant failure may occur and is defined as 'the inadequacy of the host tissue to establish or maintain osseointegration' [18]. A significantly higher survival rate as well as a significantly lower incidence of peri-implantitis was identified for dental implants after 10 years of service [17]. Factors affecting failure of implants may be broadly classified as implant-, patient- and surgical technique/environment-related. Implant-related factors could be surface roughness, surface purity or fit discrepancies [18]. The combination of the effects of surface morphology and surface chemistry must be considered in implant design amongst other important factors, as the thickness and structure of the passive layer [19]. The localized corrosion and dissolution of the implant surface can affect the function of the metal alloy surface which can eventually lead to dental implant failure, as inorganic species, body fluids contain different types of biomolecules and cells may attach to the biomaterial surface and affect the surface reactions [20]. The long-term presence of corrosion reaction products and ongoing corrosion lead to fractures of the alloy–abutment interface, abutment or implant body. The combination of stress, corrosion and bacteria contribute to implant failure [21].

In the case of implant technology, new research is continually evolving providing a better understanding of the biologic principles that govern the development of a dynamic interface between the living tissue and an artificial structure [22].

Studies on surface characteristics using photoelectron spectroscopy and electron microscopy among other surface techniques have been seen in the literature [23]. Spectroscopic studies have revealed that the surfaces of the implants consist of a thin layer of oxide covered by a carbon dominated layer. The composition of the surface oxide is shown to be mainly TiO_2. Before implantation, the oxide acquires at its outermost surface an over layer of organic molecules, mainly hydrocarbons which are adsorbed during the process of fabrication and subsequent handling. Hence, detailed surface characterization has become essential for a better understanding of the role of surface properties on implant integration in bone [24–26].

The present work reported a detailed study on the surface characteristics of failed dental implants. A biological test was also implemented in order to verify bioactivity of the samples and the formation of an apatite-like layer on the implant surface. Surface morphology was examined by scanning electron microscopy and chemical composition was determined using energy dispersive spectroscopy (EDS).

Materials and methods

Patient sample

Sixty patients received 78 implants (4.7 mm D Advent Implant 3.9 mm D apex design implant) in a private dental institute during a period of 10 years. Two stage surgical protocol was performed following the manufacturer's recommendations. A sample of 12 patients who received 15 implants was selected with inclusion criteria being that the implants failed within 6 years of their placement (2003–2009). The study population included seven males, five females within an age range of 34–54, who received eight upper implants and seven lower implants.

Failures were detected clinically through measuring peri-implant loss of gingival attachment, bleeding on probing, plaque/gingivitis indices, suppuration and mobility. Further assessment included a peri-implant radiographic examination. Failed implants were stored in their original sterile plastic bottles with a sample holder in order to allow further handling. This procedure was monitored and carefully handled to avoid excessive and subsequent contamination.

Informed consent forms were obtained from the patients explaining that the failed implant will be used for scientific research and complete anonymity of the patients was ensured.

Surface morphology (SEM analysis)

Scanning Electron Microscope (SEM) and Energy Dispersive Analysis (EDS) (Joel, JSM-6380A

Analytical Scanning Electron Microscope with EDS elemental analyzer, Tokyo, Japan) was used for morphological and chemical analysis of the implants, respectively.

The study involved three groups; failed implants were divided into two groups, A and B. Group A comprised seven implants (four upper and three lower), group B involved eight implants (four upper and four lower) and group C was the control group of two new implants of the same brand. Group A and the control group (C) were removed from their sterile packaging; gold sputtered in low vacuum environment (0.1–0.05 mbar) and received a gold layer of up to 1 nm/s. Representative photomicrographs were taken from groups A and C at magnifications of 500, 950, 2700, 6000 and 12,000×.

Surface bioactivity analysis

Surface bioactivity of dental implants was assessed through the immersion of group B implants into a polypropylene flask containing 30 ml of simulated body fluid (SBF) at 37°C for a period of 21 days, after that they were rinsed in distilled water and dried.

SBF solution was prepared using the method proposed by Kokubo et al. [27]. The reagent-grade NaCl, $NaHCO_3$, Na_2CO_3, KCl, $K_2HPO_4 \cdot 3H_2O$, $MgCl_2 \cdot H_2O$, $CaCl_2$ and Na_2SO_4 was dissolved in double distilled water and was buffered at 36.5°C at pH 7.25 with trishydroxymethylamminomethane ($(CH_2OH)_3CNH_3$) and HCl, such that the ionic composition of the SBF was similar with the human body plasma [27]. After the SBF immersion test, group B implants were gold sputtered as mentioned earlier and implant surfaces were analyzed using SEM and EDS to investigate the formation of an apatite-like layer on the implant surface.

Chemical analysis (EDS)

Group B samples examined by SEM were also analyzed by EDS (Joel, JSM-6380A Analytical Scanning Electron Microscope with EDS elemental analyzer, Tokyo, Japan) to verify bulk compositions of the materials. Chemical analyses were conducted on two areas per sample.

Samples were examined with an electron beam voltage of 15 kV and a beam current less than 3×10^{-7} A. Differential spectra were collected at two areas for each sample. Atomic concentrations were determined using the relative sensitivity factors for 15 kV. Concentration ratios were calculated using peak height intensities corrected by the appropriate relative sensitivity factors.

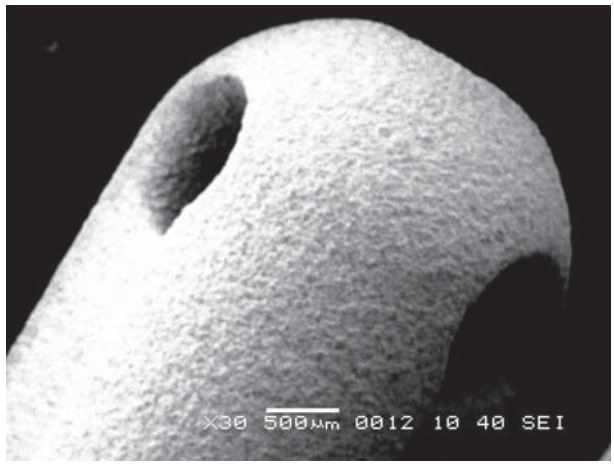


Figure 1. SEM micrograph of a new implant (Group C).

Results

Scanning electron microscopy photomicrographs of the control group (C) showed a relatively homogenous mechanical surface. In lower magnification (500×), the well-contoured threads of the manufacturer are clearly identified along with the porous surface. Higher magnification micrographs showed the irregular striations, which are likely due to the machining process (Figures 1 and 2).

Surface morphology and EDS of non-immersed failed dental implants (group A) showed major constituents of titanium and trace quantities of sodium and chloride (Figures 3 and 4).

The Group B implant shown in Figure 5 is at a lower magnification. Surface morphology and EDS of dental implant after immersion in SBF indicates the appearance of Ca as well as low Ti peaks (Figures 6 and 7) The presence of Ca means that newly formed particles on the coating are comprised of Ca. Some spherical particles accumulated on some areas of the surface, as shown in the micrograph (Figure 8). The Ti-OH groups, which have formed on the surface, are definitely negatively charged and have a chemical

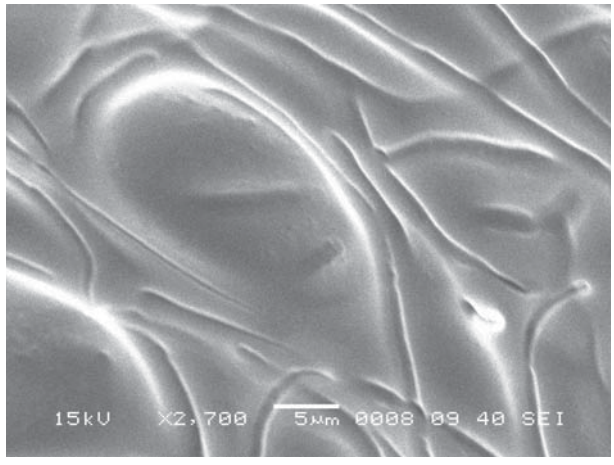


Figure 2. SEM micrograph of a new implant (Group C).

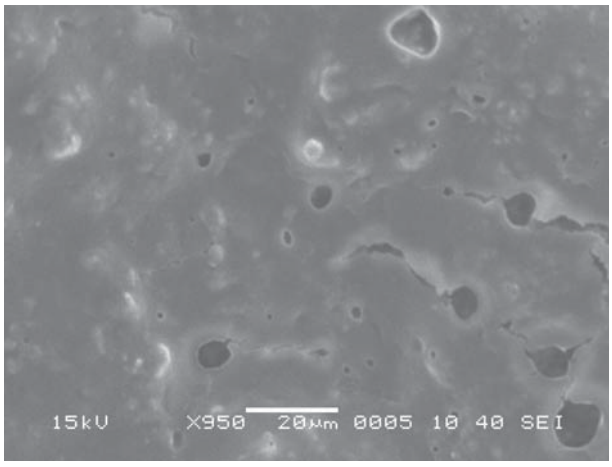


Figure 3. SEM micrograph of a non-immersed dental implant (Group B) showing porous surface of the implant.

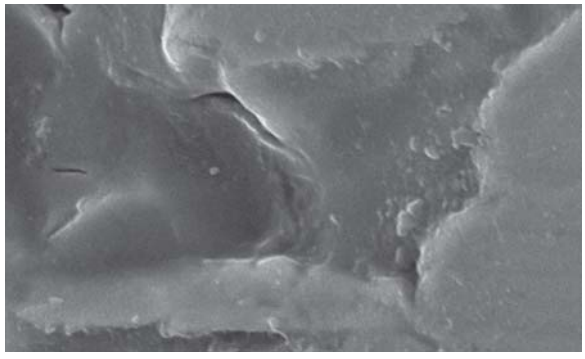
attraction for Ca_2+ and Na_2+ ions in the SBF and form amorphous Na-Ca-Ti-O compounds. As the Ti-Oh groups increase, Ca and Na ions continue to accumulate and positive ions combine with negatively charged phosphate ions to form amorphous Na-Ca-Ti-O-P compounds. XPS studies would be needed to understand more of the nature and formation of these corrosion products.

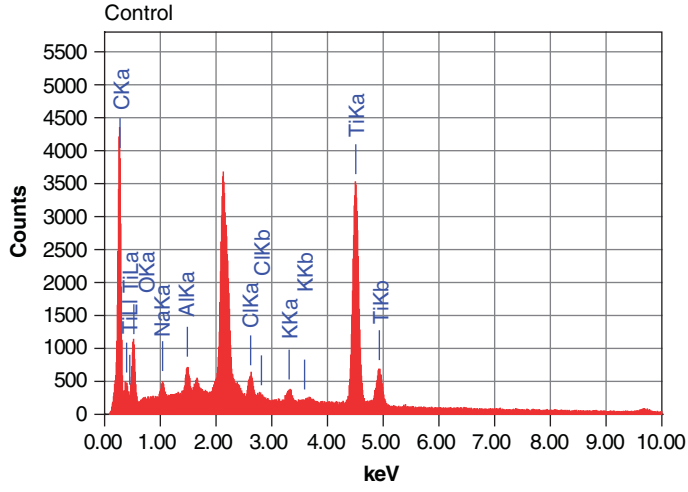
All retrieved implants consisted of different degrees of organic residues, appearing mainly as dark stains. Control samples were essentially free of macroscopic contamination, whereas failed implants contained varying amounts of tissue residues. All surfaces consisted of titanium oxide and varying amounts of additional elements, with C dominating in most cases. Na, Ca, K and Cl were detected measurably.

Discussion

This study was conducted to investigate the surface characteristics and chemical composition of failed dental implants before and after immersion in SBF. The study involved advent dental implants as they were widely used in the institute where the study was conducted. Although titanium and its alloys have an excellent reputation for corrosion resistance and biocompatibility, failures of implants retrieved after use in patients call for attention to the problem of surface stability. Therefore, the material should present superior corrosion resistance in contact with body fluids.

SEM was used to interpret the spectroscopic appearance of implants, while EDS was used to detect elements with atomic number higher than that of sodium in a surface thickness of 1000 nm [28]. SEM images showed different surface morphologies for each group and this might be attributed to the different conditions in which these implants were tested. One of the key parameter for implant osseointegration is roughness of the dental implants. The current study has not addressed roughness of the tested implants and it is highly recommended to conduct studies to evaluate this issue [29].





ZAF method standard less quantitative analysis
Fitting coefficient: 0.4562

Element	(keV)	mass%	Error%	At%	Compound mass%	Cation K
C K	0.277	37.79	0.13	56.87	23.3725	
O K	0.525	24.61	1.05	27.80	11.4840	
Na K	1.041	1.13	0.33	0.89	0.9898	
Al K	1.486	1.26	0.22	0.85	1.5616	
Cl K	2.621	1.63	0.25	0.83	2.3385	
K K	3.312	0.98	0.35	0.45	1.4127	
Ti K	4.508	32.60	0.56	12.30	39.7859	
Total		100.00		100.00		

Figure 4. Surface morphology and EDS of non-immersed failed dental implant (group A) showing major constitutes of Titanium and trace quantities of sodium and chloride.

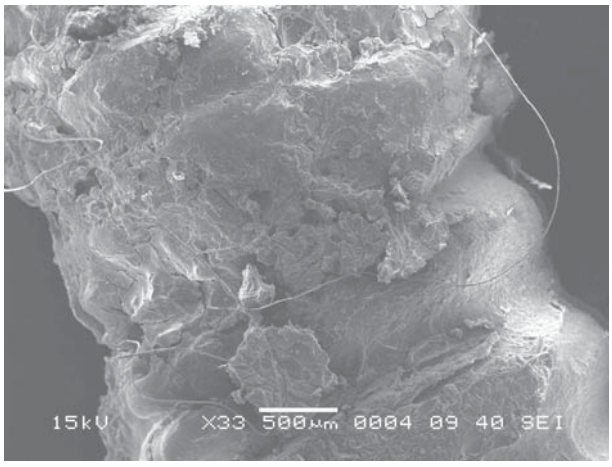
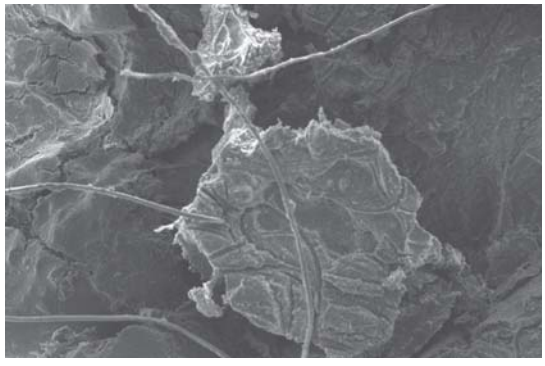
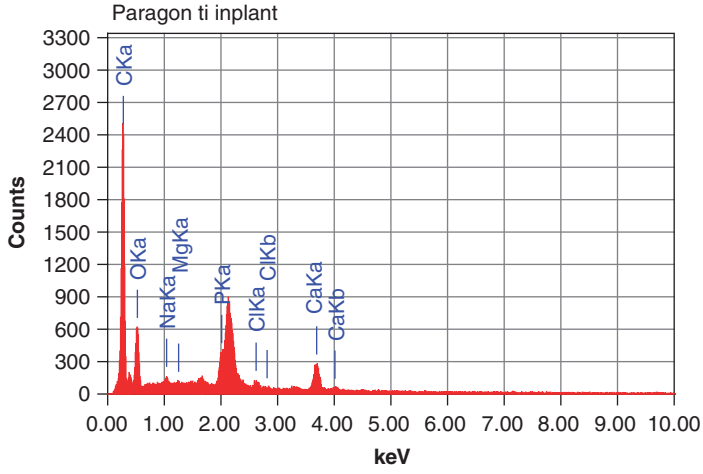


Figure 5. SEM micrograph of a failed implant after immersion in SBF (Group B).

The difference in the contaminants of the oxide layer is difficult to explain as the results reinforce the idea that in reality the surface is dynamic and capable of interacting with its surrounding [28]. Rapidly increasing numbers of publications are supporting the use of simulated body fluid to test the bioactivity of material. The test that has been conducted still leaves a lot of room for improvement. Theoretical arguments and facts supporting these statements are provided, together with possible improvements of the proposed bioactivity test.

New components like Ca, Na and Cl have been revealed after SBF immersion. A vast number of different interactions and reactions take place in the interface area, all of which define the relationship of the implant and surrounding surface. When a solid implant material is surgically implanted, protein adhesion to the biomaterial surface takes place immediately [30]. Energy dispersive spectroscopy analysis showed the presence of Ca, P, Ti and O elements. Analysis revealed that the surface passive TiO_2 layer reacts with SBF solution. The surface passive TiO_2 layer reacts with the hydroxyl ions (OH-) in the SBF and the Ti–OH groups formed on the surface from the above reaction are negatively charged and have a chemical affinity for Ca2+ and Na+ ions in the SBF and combine selectively to form amorphous Na-Ca-Ti-O compounds. As the amount of Ti–OH groups on the surface increase, the calcium and sodium ions can continually accumulate on the surface and as a result the surface gradually gains an overall positive charge. The positively charged surface combines with negatively charged phosphate ions to form amorphous Na-Ca-Ti-O-P compounds [31]. Therefore, the understanding of protein adhesion and biomaterial interaction is extremely important, but little is known about adsorption and exchange of proteins on such surfaces. The combination of effects of surface morphology and surface chemistry has been directed toward defining the nature of interactions between the oxide layer and the surrounding tissue. However, further studies are required to understand the exact nature and formation of corrosion products.

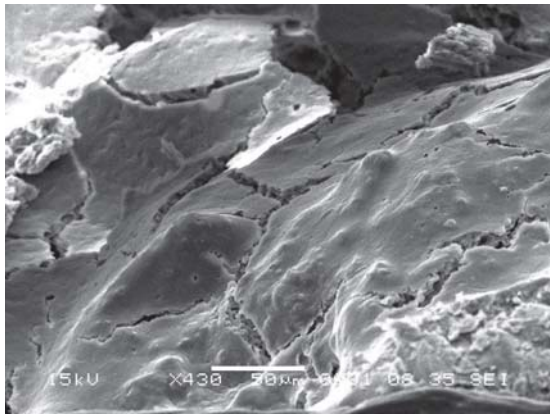


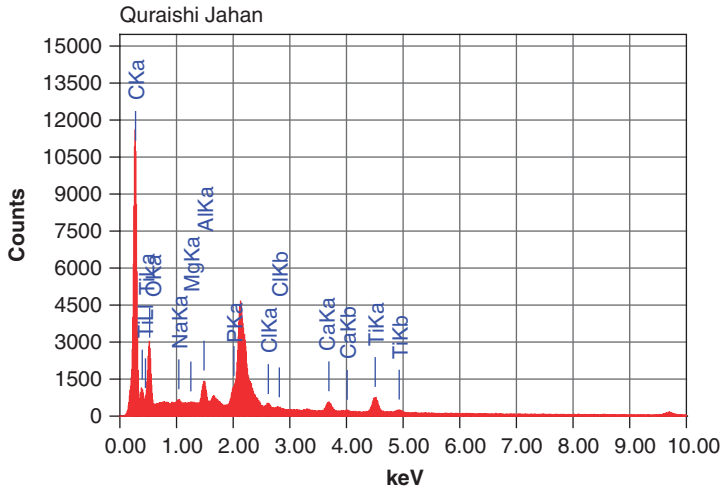


ZAF method standardless quantitative analysis
Fitting coefficient : 0.5792

Element	(keV)	mass%	Error%	At%	Compound mass%	Cation K
C K	0.277	58.67	0.15	67.68	41.2603	
O K	0.525	34.02	1.02	29.46	19.5013	
Na K	1.041	0.93	0.45	0.56	0.7802	
Mg K*	1.253	0.23	0.37	0.13	0.2363	
P K*						
Cl K	2.621	0.95	0.38	0.37	1.1540	
Ca K	3.690	5.19	0.63	1.80	6.4036	
Total		100.00	100.00			

Figure 6. Surface morphology and EDS of dental implant after immersion in SBF (group B) showing traces of titanium.





ZAF method standardless quantitative analysis
Fitting coefficient : 0.5431

Element	(keV)	mass%	Error%	At%	Compound mass%	Cation K
C K	0.277	57.85	0.14	67.68	40.0229	
O K	0.525	32.79	1.01	28.80	19.2539	
Na K*	1.041	0.46	0.40	0.28	0.3959	
Mg K*	1.253	0.07	0.33	0.04	0.0772	
Al K	1.486	1.97	0.28	1.03	2.3167	
P K*						
Cl K	2.621	0.68	0.34	0.27	0.8575	
Ca K	3.690	1.65	0.56	0.58	2.1445	
Ti K	4.508	4.54	0.79	1.33	4.7898	
Total		100.00		100.00		

Figure 7. Surface morphology and EDS of non-immersed failed dental implant (group B) showing minor constitutes of Titanium and trace quantities of sodium and chloride.

Conclusion

After SBF immersion, the implant surfaces showed new components like Ca, Na and Cl, but in trace quantities, therefore the apatite layer formation could not be verified.

The infirmity of the dental implant testing is done because the work is relevant and there are very few studies of implant surface properties.

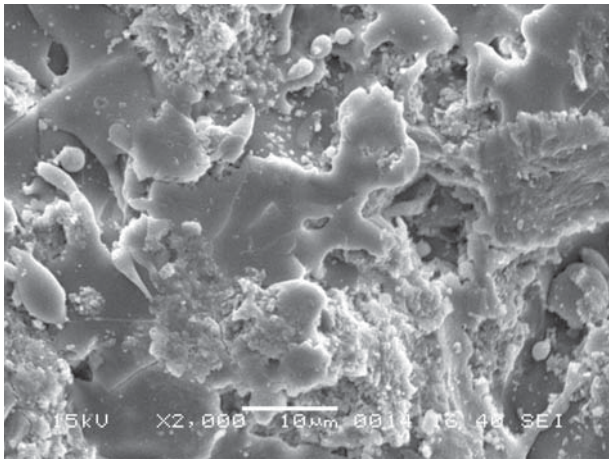


Figure 8. Surface morphology of non-immersed failed dental implant (group B).

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Declaration of interest: The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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