Biomechanical evaluation of porous titanium implants (CpTi) fabricated by powder technology

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ABSTRACT
Background: It may be an important prospective clinical use of manufacturing of porous implant for clinical situations, such as cases of limitation in bone height, low bone density. The small segment of porous implant an effective osseointegration allows increasing in contact area provided for small segmented porous provided by its surface configuration. This study was done to Fabricate porous titanium implants by powder technology, as well as the observation of removal torque values of porous titanium implants compared to smooth titanium implants.

Materials and methods: Twenty porous titanium implants (3.2mm in diameter and 8mm in length) were manufactured by powder technology using commercially pure titanium powder of ≤75um particles size, with polyvinyl alcohol powder of 212-300um particle size, as a space holder, by volume ratio (70:30) % respectively. The mixed powder was compacted using punch and die set -specially designed for this study –under 20 bar then sintering at 900°C by the use of argon gas. Twenty smooth titanium implants were prepared of (3.2mm in diameter and 8mm in length) by lathing of commercially pure titanium rod as a control group. The implants were examined by X-ray diffraction (XRD) and scanning electron microscope (SEM), as well as estimation of porosity percentage. For each tibia of the 20 white New Zealand rabbits, one implant of each type (one porous in right, and the smooth in left tibia), were inserted through surgical procedure carried under serial condition. Mechanical test was performed to evaluate the bone-implant interface, after (2 and 6 weeks) healing periods.

Results: Porous implants were obtained successfully by powder technology with 52% porosity and pore size 210um 17±. The porous implant showed significantly higher removal torque values when compared to smooth implants at the two different intervals of examination (2, 6 weeks), this proved to be statistically highly significant, also a highly significant difference was noticed for the means of the torque removal values in the same group at different implantation , with no evidence of clinical features of inflammatory reaction with both.

Conclusion: Powder technology seemed to be particularly advantageous in fabrication of porous titanium. Porous implant show an increasing bone ingrowth compared with the smooth type represented by higher removal torque for both healing periods (2, 6) weeks.

Key words: Porous titanium implant, powder technology, removal torque test. (J Bagh Coll Dentistry 2015; 27(1):18-25).

INTRODUCTION
Lost body structures are replaced by surgical implants gaining the goals of becoming the most promising fields, improving quality of life with the increase in expectancy of population. The most commonly commercially biocompatible material used for the manufacturing of surgical implants are metals, with titanium being the most commonly used metals in the field of biomedicine presenting excellent physical and chemical properties there are two main groups of titanium according to manufacturing process the casting and powder technology (1,2).

At present, however, the fabrication of Ti-based implants through the casting method is limited to a costly, multi-step process of vacuum melting machining, which is costly with the limited use the high melting temperature of Ti (3,4).

The advantage of using powder technology (powder metallurgy is due to its processing route with limited cost) (5).

In powder technology pores can be found from removal of spacer particles with increasing porosity which is crucial for bone ingrowth.

“Bone in growth", is the osseointegration gained by micromechanical interlocking between the bony tissues and porous structure of the implant which representing strong implant-bone bond thus increasing stability and preventing mobility.

These pores can be interconnected three-dimensionally, which in turn provide enough space for the attachment and proliferation of new tissues thus facilitating the transport of body fluids (6,7).

The applications of porous implants being ranged from spinal fixation to hip prostheses, osteosynthetic plates, and dental implants (8).

MATERIALS AND METHODS
Commercially available titanium powder particle size ≤75 um was used. Firstly PVA particles were milled using mill to powder then sieved using two sieves 212um and 300 um. PVA with average particle size (212-300 um) was used. Pilot study was done to find the best percentage for porous titanium implant five percentage were tested after mixing by volume.

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ratio (90:10)%, (80:20)%, (70:30)% and (60:40)% (50:50)% Titanium powder and PVA respectively.

The (70:30) % Titanium-PVA was selected. The pressure of 20 bar was selected as the best amount of pressure to be applied. The condensation time of 60 seconds was selected. Sintering was performed by Carbolite Furnace using argon gas under 900 c. Preparation of samples for the tests by ultrasonic cleaning using: distilled water, acetone solution, ethanol solution, finally distilled water for 20, 20, 20, and 15 min, respectively (9).

Two implants were placed in a single air tight plastic sheet (one implant from each group) then the implants were autoclaved at (121°C and 20 bar) for 30 minutes, as was performed by Xue XB et al. (10).

![Figure 1-a.stereomicroscope, (b, c implants as appearing under stereomicroscope) b.cylindrical compacted implant before sintering, c. cylindrical compacted after sintering](image)

Examine Implants SEM
SEM and stereomicroscope images observation of the porous and smooth titanium samples was carried on to reveal the micrograph.

X ray diffraction analysis
Phase analysis was employed for CP-titanium powder and porous titanium samples using Shimadzu Lab XRD- 6000 Powder X-ray diffract meter and Cu Kα target radiation . The 20 angles were swept from 20- 80° in step of one degree each time.

Porosity test
The density and porosity of the consolidated samples were measured using Archimedes (9)(11).

Sample distribution before surgery
40 implants were placed into 20 rabbits and were divided into:
- a. Control group (smooth implant): This group includes 10 implants for each healing interval (2 and 6 weeks) implanted in 10 rabbits.
- b. Experimental group (porous implant): This group includes 10 implants for each healing interval (2 and 6 weeks) implanted in 10 rabbits.

Animals and surgical procedures
Twenty New Zealand white rabbits of both sexes weighing 2-2.5 kg were used. The age of the animals was from 10-12 months. Animals were kept in standard separate cages and had free access to tap water, and were fed with standard pellets. They were left for 2 weeks in the same environment before surgical operation. Antibiotic cover with ox tetracycline 20% (0.7 ml/kg) intramuscular injection was given to exclude any infection (one dose/day, for 3 days). All instruments were autoclaved at 121°C and 20 bars for 30 minutes.

The required dose of anesthesia and antibiotic was calculated by weighing each rabbit in a special balance for the animals. Anesthesia was induced by intramuscular injection of ketamine hydrochloride 50 mg (1 ml/kg body weight), Xylazine 20% (0.15 ml/kg body weight). and xylcaine 10% (1 ml/kg body weight). Surgery was performed under sterile condition and a gentle surgical technique. Incision was made on the medial side of the legs about (3 cm) length to expose tibia bone. The skin, fascia the periosteum were carefully reflected.

Drilling was done using round bur with intermittent pressure and continuous cooling with normal saline at rotary speed 1500 RPM and reduction torque 16.1. The enlargement of the hole was made gradually with spiral drill from 2.2 mm 2.9 mm till 3.1 mm.

The operation site was cleaned with copious amount of saline to remove bone shreds; the implants were removed from the plastic sheet and placed in holes with slight tapping pressure until 5 mm was completely introduced into bone.

Suturing of fascia was done with absorbable cat gut suture followed by skin suturing. The operation side was washed with normal saline followed by bandaging. Post-operative care, performed by giving an antibiotic (local and systemic) for 5 days after surgery.

Torque removal test
The animals that categorized for mechanical test were anesthetized with the same type and dose that used in the implantation procedure. Incision was made at the medial side of tibia; muscle and fascia were reflected to expose implants.
The stability of implant checked by the end of head of torque meter, Tibia was supported firmly while performing mechanical test to prevent any movement, which may have an affects on the accuracy of the test. A torque removal test was done by the torque meter to determine the peak torque necessary to loosen the implant from its bed, through the torque meter head manufactured for the measuring purpose of this study.

RESULTS

SEM observations

1. The SEM image observation of the porous titanium samples shows the surface morphology Fig. (2),(3). The pore space structure after space holder removal displays ragged shaped macro-pores inside the sintered material, where the number and the size of spaces can be evaluated. On the other hand a three-dimensional interconnected pores was clearly observed between the pores. Fig (3), (4).

2. The SEM observation of the smooth titanium samples Fig (5)

Figure 2: SEM of porous titanium implant

Figure 3: SEM of porous titanium sample showing the macropore

Figure 4: SEM of porous titanium sample shows the interconnected pores

Figure 5: SEM illustrates topography of smooth titanium implant

Figure 6: X-ray diffraction patterns of TI implant and TI powder

X-ray Diffraction Phase Analysis

The x-ray diffraction pattern of untreated commercially pure titanium powder and the sintered commercially pure titanium implants are shown in Fig (6).

It is clearly obvious that the strongest peaks of powder were at (100) , (002) , (101) and(102) at 2Ө 35.20 , 38.48 , 40.27 ,and 53.08 respectively which could be indexed for αTitanium (JCPDS file 44.1294).

Figure 6: X-ray diffraction patterns of TI implant and TI powder
Also the pattern shows strong peaks of the sintered commercially pure titanium implants at (101), (101), (002), and (102) at 2θ 40.23, 40.08, 38.18, and 53.23 respectively and this pattern is corresponding to the powder and responsible for αTitanium (JCPDS file 44.1294)

Clinical observation
All animals recovered well after surgery presenting clinically satisfactory postoperative results as an indication of good tolerance for the implantation procedure, with no clinical evidence of inflammation or infection at the surgical site

Torque removal test
The removal torque values of porous titanium implant after 2 weeks of implantation. Where at that interval, a higher torque values was needed to remove porous implants (mean value of 13.77 N.cm) compared to the torque value needed to remove smooth titanium implants (mean values of 8.27 N.cm ) (Figure 7).

Descriptive statistics of removal torque values at 6 weeks after implantation, where higher torque force was required to remove the porous titanium implants (mean value of 18.79 N.cm) compared to that needed for smooth titanium implants (mean values of 13.55 N.cm ) fig (8)

Effect of time on removal torque value
Both coating materials showed increased torque removal force between 2 and 6 weeks of implantation which was statistically highly significant. Figure 9

<table>
<thead>
<tr>
<th>Types of implant</th>
<th>t-test</th>
<th>df</th>
<th>Sig. (2-tailed)</th>
<th>Sig</th>
</tr>
</thead>
<tbody>
<tr>
<td>Porous × smooth</td>
<td>15.77</td>
<td>18</td>
<td>.000</td>
<td>HS</td>
</tr>
</tbody>
</table>

Table 1: t-test for equality of means of torque value for porous and smooth implants at 2 weeks interval

<table>
<thead>
<tr>
<th>Types of implant</th>
<th>t-test</th>
<th>df</th>
<th>Sig. (2-tailed)</th>
<th>Sig</th>
</tr>
</thead>
<tbody>
<tr>
<td>Porous × smooth</td>
<td>15.86</td>
<td>18</td>
<td>.000</td>
<td>HS</td>
</tr>
</tbody>
</table>

Table 2: t-test for equality of means of torque value for porous and smooth implants at 6 weeks interval

T-test was performed for comparing the equality of means for the same group at the different implantation periods. A highly significant differences at p<0.010 between each subgroup of the two periods of examine times.

It was clearly obvious that the torque value needed to remove implants from the bone was increased as healing period increased.
Table 3: t-test for equality of means of torque value within the same group at different time interval 2&6 weeks interval.

<table>
<thead>
<tr>
<th>Type of implants</th>
<th>TIME in weeks</th>
<th>N</th>
<th>Mean</th>
<th>S.D.</th>
<th>T-test</th>
<th>Sig.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Porous</td>
<td>6</td>
<td>10</td>
<td>18.79</td>
<td>.77</td>
<td>14.617</td>
<td>HS</td>
</tr>
<tr>
<td>Smooth</td>
<td>6</td>
<td>10</td>
<td>13.77</td>
<td>.84</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>10</td>
<td>13.55</td>
<td>.70</td>
<td>17.234</td>
<td>HS</td>
</tr>
</tbody>
</table>

S : Significant at P<0.05

DISCUSSION

Around the biomechanical area the resent use of powder technology is of great advantage for the final format of prosthesis production dense or porous and less expensive than the conventional (6,7,12).

1 Part One in Vitro Study

1.1 Selection of powder percentage and particles size

The reason behind choosing the volume percentage of 30% PVA -70% titanium powder, was because implant surface morphology is considered important for ossiointegration, since fibrin clot retention and bone progenater cell migration are related to surface topography is associated .

The use of large particle size for space holder (PVA), and fine particle size for ti, could be due to both a wider PVA particles distribution (which promotes a higher degree of interconnectivity of the pores) and a high average size of space-holder (>200um )which would fulfill the requirements to ensure the growth of bone into the implant (ingrowth); on the other hand, the choice of a titanium powder of small average size would improve the sinter ability of the compact (quality of the neck and lower grain size), helping to offset the loss of mechanical strength inherent in increased porosity.

1.2 Powder Compaction and problems associated with it:

Punch and die set was designed in a way that ensure proper condensation of porous titanium implants .The powder / spacer material mixture was compacted using the hydraulic press with a pressure of 20 bar for 60 seconds, genuinely determined by trial and error in order to get the good quality for producing "green strength " that allowed enough handling strength.

The pilot study showed that when powder / spacer material mixture was compacted at a pressure higher than 20bar with a holding time of more than 60 seconds, the compact became very hard with difficulty in ejecting the pellets from the mold and with a tendency to damage the punch .It was also noted in the pilot study that the compaction pressure should not be used when holding time less than 30 seconds.

It could be understand that when the pressure is too high a considerable proportion of binder would be crushed during compaction this finding coincide with XB Xue et al. (10). While through the compaction of powder before sintering one can improve the mechanica properties (12,13)

The loss of interconnectivity in between the powder particles may be caused by loose packing of powder mixture (14). Generally, higher compaction pressure increased the densification of the Ti powder.

Heat treatment

In sintering (thermal treatment ) the classic melting was substituted , and carried out below the melting point of the metal .In the pattern fig.(6)of the XRD phase analysis showed that in sintered titanium implants, heating was carried on using argon gas to provide a non oxidizing environment ; and this explained by in that the Ti and its alloys may have high affinity towards interstitial elements like oxygen and nitrogen required a non oxidizing environment thus reducing the residual surface oxide in order to improve the metallic contact between adjacent powder particles as stated by Gasser, Nyberg et al. and Ryan et al. and Nouri et al. (34,15,16) On the other hand conventional processing of molten metal to fabricate porous metal is suffering from limited part geometries, and limited control over the size, shape and distribution of porosities , contamination , costly, multi-step process (17).

This in turn can confirmed that in particular, the casting method is unpractical for manufacturing of porous Ti based scaffolds, due to the high melting point and the high affinity of Ti towards oxygen and special refractory materials during the manufacturing process and these support the findings of Ryan et al. (15). These difficulties driven the researchers to a more cost affordable manufacturing methods with minimal waste product (3).

Scanning Electron Microscope

The SEM image observation of the porous titanium samples revealed the micrograph of the porous cylindrical implants upon the removal of the space holder (18). The pore-space structure in the sintered material contains different types of pores; Macro-pores, determined by the number and size of the space holder materials Fig (2, 3and4), also SEM images showed clearly,
interconnected pores. The average pore diameter was about 210μm (± 17), and 52% total porosity. This agreed with Elema et al. (19) who proposed that the pore size should range from 200 to 300 μm for bone tissue in growth in the porous samples; although they did their study about biodegradable porous polymeric implants.

Small pores could favor hypoxia, which can result in the formation of osteocartilaginous tissue, while large richly vascularized pores permit direct osteogenesis and thus resulting in an improved bone implant interface (20). In addition to the presence of pores with more ragged and rough surfaces as seen in fig 3.5 offering larger surface area for bone ingrowth (21).

Both the open porosity and pore interconnectivity are necessary for bone ingrowth, and extensive body fluid transport through the porous implants possible, thus trigger bone growth. It is also known that the pore size itself is less important than the amount of interconnectivity for new bone formation. This agrees with Chen et al. and Nouri et al. (22,23) with the difference in material and technique used.

In the present study the observation of The SEM image can give a good indication of the packing of the powder mixture at a given sintering process.

Porosity
The porous structure of the alloys is important for the growth of bone inside the implant body and thus will improve the fixation and stability and the remodeling between the implant and the human tissue (24); by providing space for cell adhesion and permitting the transport of body fluids and thus leads to acceleration in the proliferation of new vasculature, while providing adequate mechanical properties to withstand stresses during surgical procedure and use (1). This agrees with Ryan et al. and vasconcellous et al. (6,15) but with the difference in material, method that used.

The total porosity percentage of the fabricated porous implants after porosity test was within 52% as used in this study which could be an alternative for clinical use, for the reason that increased porosity may permit the growth into pores and subsequent mineralization. Many authors have been suggested that the percentage of pores preferable for Ti samples is between (25-66%). However, samples reaching till 80% porosity have also shown bone formation (25-27).

On the other hand the percentage of the open porosity was 33% while the percentage of closed porosity was 19%. Pores are usually surrounded by pore walls and disconnected from each other in closed –cell porous implant structure, while in open-cell porous implant structure, pores are connected to each other, thus ensuring fixation of implants as new bone tissue grows and integrate into the this is in agreement with Banhart and Shehata Aly et al. (28,29).

Part Two in Vivo Study
Implant Preparation Prior to Surgery
In this study the size of the holes created in the bone were (3.1mm) which was smaller than the diameter of the implant(3.2 mm) and this in turn would result in a better surgical fit, and as a consequence, force-fitting stress increases installation torque and initial stability and This agree with Skalak and Zhaoin and Waheed (30,31) with the differences in material, method, technique, and shape used in this study.

Mechanical Test
The removal torque value (RTV) is the torsion force required to remove an implant and this value represent the critical torque threshold where implant contact was destroyed. This would indirectly provide information about the amount of bone -implant contact for a given implant. Such testing was carried out on experimental animals model, where the rabbit tibia are the most frequently bone components cited in literatures Alnajar et al and Gonzalez et al. (32,33).

The increase in the amount of cortical bone in contact with the implant required greater removal torque forces where the surface of the implant is often porous thus increasing bone/implant interface which consequently will increase the bony ingrowth into the surface irregularities of the implant (34).

Tables (1 and 2) demonstrates t-test for equality means of the removal torque values of the porous titanium implants and the smooth titanium implants at the two implantation testing periods (2 and 6 weeks). It showed statistically highly significant difference; which indicates minimum removal torque values associated with smooth implants group, while the maximum removal torque values were associated with porous implants group thus suggesting that the pore structure for the porous implants provide more surface area and space for bone ingrowth as well as mechanical interlocking between the implant and bone.

The surface area and contact surface configuration are important parameters for implant stability. When there is little or no mechanical interlocking between the implant surface and bone, any excessive loading may
cause rupture at the bone-implant interface. This mechanical interlock should enhance the strength of the bone-implant interface. As well as the force needed to extrude the bone through the porosities may be much higher than the bone mechanical strength itself. This agrees with Wazen et al. (35) with the difference in the material, method.

Implant porosity promotes positive results in bone neoformation in vivo since it facilitates the transport of body fluids, aids in the spread of cells into the implant. Improving the implant stability over time is gained through increase in contact area between bone tissue and implant this in turn promoting the proliferation of bone tissue through a mechanism which is not usually observed on flat or rough surfaces, on the other hand the process of osseointegration is accelerated as claimed by Bottino et al., Vanconcellos, Wazen et al. and Faria et al. (6-8, 35,36) with difference in the material, method technique and shape of implant used in this study.

The removal torque method selected in this study is used for the first time shows the correlation between the force necessary for removal of the porous implants and the degree of bone implant integration and it focuses on interfacial shear properties.

The amount of integration in RT method may be affected by implant geometry and topography as stated by Waheed and Alnajar (31,32) but on the other hand the material and the technique and shape are not the same and are used for the first time.

Table (3) showed the result of t-test for equality for means of removal torque value within the same group at the different implantation periods shows a highly significant difference, which means that the minimum torque value was seen within 2 weeks of implantation periods, while the maximum value was observed in the 6 weeks implantation periods for both the porous and smooth groups.

It was noticed in this study that the torque value significantly increased with time for both the porous and smooth implants. These results may suggest increased holding power and anchorage of implant with time due to progressive bone formation around the implant during healing period and consequently improved mechanical capacity due to maturation of bone with elapsed of time.

REFERENCES


