CHITOSAN BASED COATING ON THE PUR TITANIUM SUBSTRATE / DENTAL APPLICATION

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Abstract:

Chitosan bioactive coatings on bio-inert metal implants have recently received considerable attention. Chitosan has many properties that can be used in the biomedical field. This work aims to develop chitosan layers on titanium substrates with physic-chemical and mechanical properties favorable for an application for dental implants. The extraction is performed chitosan from the shrimp shells*. The chemical composition of the chitosan is carried out by IR. The morphology of the deposited coating is examined by optical microscopy shows that the chitosan coating is uniform. The layer thickness is determined by weighing it varies from 119 to 160 µm.the influence of the roughness of the substrate thickness and studied with the roughnessmeter. The first breakout tests show that this coating adhesion. The hardness test showed that the thickness is uniform and that the layer does not have any stresses at the interface. The biological response of the developed coatings was evaluated by observation of the water wettability and at room temperature.*

1. Introduction:

In recent years, considerable progress has been made in the field of dental implants and care techniques in orthodontics. Many materials have been studied such as titanium, ceramic, zirconium. Titanium and its alloys are widely used as an implant because of their good mechanical properties [1], biocompatibility and corrosion resistance [2]. The response of biological tissue transplantation implants some problems. In fact the metallic implants, such as titanium implant can release harmful metal ions in the body or allergies develop in the oral cavity of the patient. To address the interaction implant / biological tissue, it is necessary to study implant / biological tissue and especially the change of the interface surfaces of the implants. Several techniques for modifying surface properties such as plasma spraying
[3, 4, 5], sol-gel method [6, 7], electrophoresis and electrochemical deposition [8, 9], have been developed to deposit bioactive film on titanium and its alloys. The purpose of this

change is to improve the interaction of the implant with the biological environment and to better protect patients against all infections (allergy ...). The treatment consists of depositing on the surface a layer of implants with a bioactive material. Chitosan, a promising bioactive polymer as implantable material [10]. Indeed, chitosan prevents the growth of fibroblasts, cells that may prevent the integration of the implant in the bone tissue [12, 13]. Chitosan is non-toxic polysaccharide degradation after it is transformed into simple sugars and can be used by normal cellular metabolism [10, 14].

2. Materials and methods:

2.1 Synthesis of chitosan:

The shrimp shells from local resources are washed with water and then treated. Then they are heated in water for 2 h and then dried in an oven at 100 ° C for 4 h. The dried shells were ground to obtain a powder. The obtained powder is demineralized shells with a 1M HCl solution at room temperature for 24 hours, filtered and then the powder is washed with demineralized water until neutralization. The déprotéinisation is performed at room temperature with a solution of 2M NaOH during 24 hours. Chitin obtained is treated with 12M sodium hydroxide NaOH at a temperature of 120 ° C for 6 hours. After filtration, washing with deionized water and desalting and drying the chitosan is obtained at 80 ° C in Fig.1. The aqueous chitosan solution is obtained by dissolving 1 g of chitosan in 50 ml of acetic acid 1% (v/v).

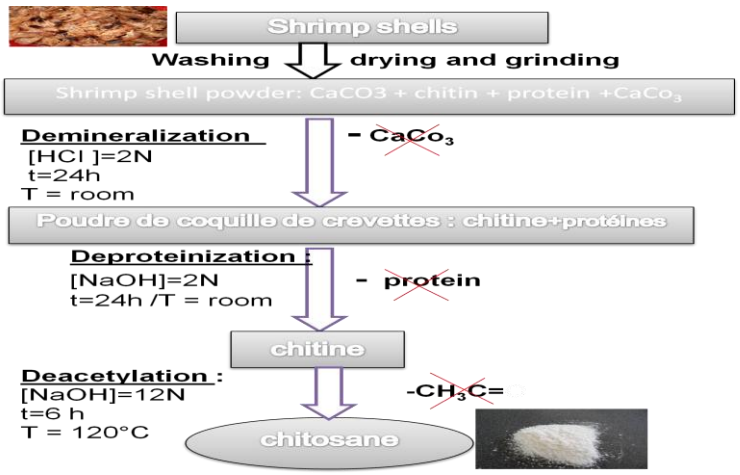

Figure 1: Extraction explanatory Schema of chitosan.

2.2 Preparation of chitosan deposit:

Titanium substrates used in this work are disks with a diameter 12 mm and thickness 3.7 mm. The samples are mechanically polished with abrasive paper and then cleaned with deioinzed water and ethanol.
Using the method implemented in the laboratory, the chitosan coatings are deposited on a titanium substrate layer by layer Fig.a.2 in the LBL coatings as a versatile inexpensive but effective technique for biologically active surfaces [15, 16] in recent years. To obtain a uniform, adherent and stress at the interface without topcoat, the substrate is driven in a rotary motion with a constant angular velocity in Fig.2 Once the layer is deposited, the samples dry at room temperature.

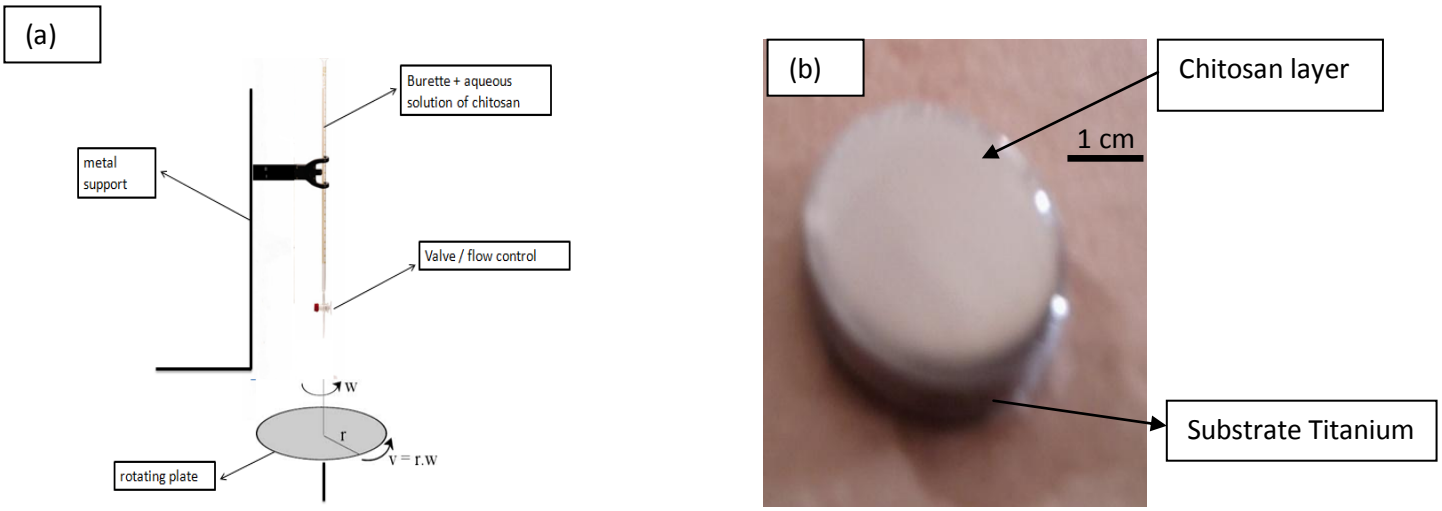

Figure 2: a) Principle methodology deposit on titanium substrate. b) Deposit of chitosan on titanium.

3. Measure:

The chemical composition of the chitosan is carried out by IR .The macrographic clichés coatings are obtained by optical microscopy Olympus BX60. The roughness are measured before and after the deposit of chitosan using roughnessmeter RM600 available in the laboratory. The coating thickness of chitosan is derived by taking the mass of the substrate before and after deposition. The adhesion of the coatings is carried out under a load of 500 g and a displacement of 50 microns. This test is performed using a device mounted on a microhardness RWU Indentation test under a load of 500 g and a loading time of 15 s are performed to evaluate the quality of the coating to crack resistance. The biological response of the developed coatings was evaluated by observation of the water wettability and at room temperature.

4. Results and discussion:

4.1 The IR analysis of chitosan:

To determine the chemical composition of chitosan prepared IR analyzes are carried out. The spectra show intense peaks namely 1656 $cm^{-1}$the peak, which is bonded to the amide function which characterizes the chitosan polymer peaks.

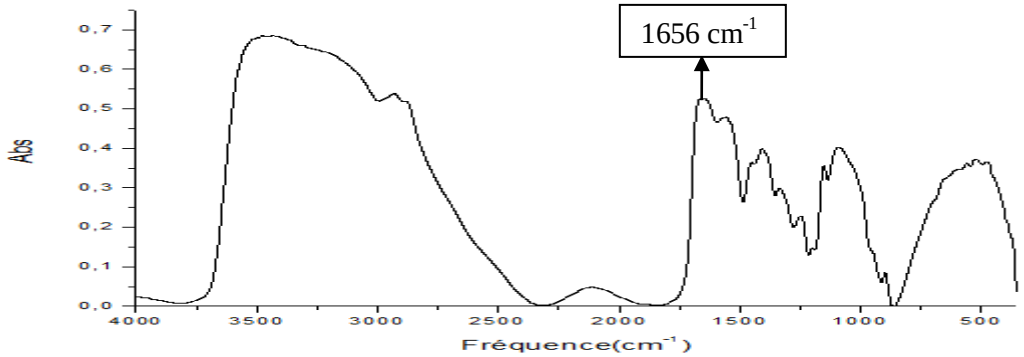

Figure 3: Spectrum I.R chitosan.

4.2 Roughness:

Roughness measurements chitosan coatings are made on the substrate and no deposit on the coating surface. The results are summarized in the following table 1:

Polishing Paper	Ra (µm) (before Deposit)	Ra (µm) (after deposit)
Sample polished paper 320 SiC	0,34	0,25
Sample polished paper 500 SIC	0,31	0,27
Sample polished paper 800 SiC	0,28	0,38
Sample polished paper 1000 SiC	0,25	0,4

Table 1: values of roughness before and after deposit of the samples

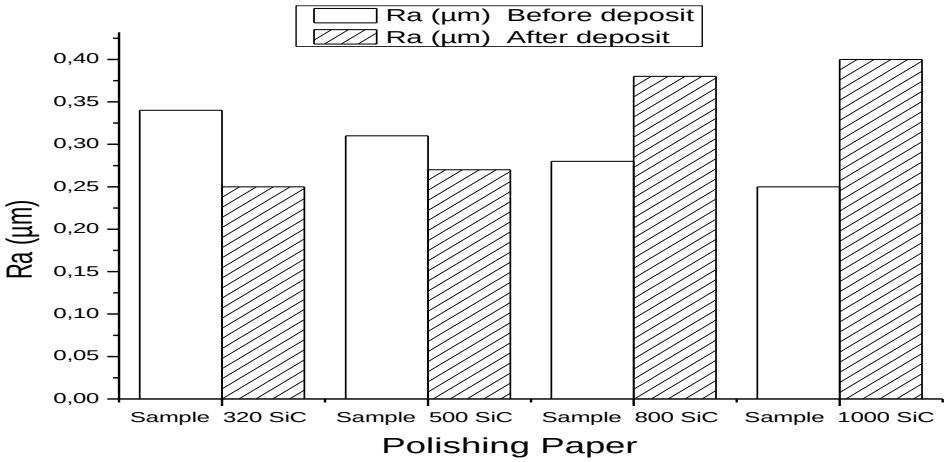

Figure .4: Variation of roughness before and after deposition, depending on the grade of polishing paper.

The degree of the paper substrate polishing effect on the value of roughness of the coating. The roughness of the samples that coatings of Ra = 0, 31 and 0, 34 µm decreases after deposition for samples of chitosan in the Ra = 0.28 and 0.25 µm, the roughness believed after deposition in Fig.4 .Indeed polishing with grade 320 SiC paper and 500 SiC generates a gross surface substrate deep scratch. The chitosan gel deposited on the surface of these samples is filled these ridges form a coating in samples lisse.par against whose Ra is equal to 0.28 to 0.25 µm and the initial surface is smooth when the chitosan gel is deposited on the surface it spreads it in a time of solidification form streaks on the surface.

Examination of the literature shows that the areas used for growing cells have a roughness of between 0.02 and 6 µm [17].The roughness of the films produced in this work lie in this area.

4.3 Measurement of the thickness of chitosan coating:

Thickness of coating of chitosan is derived by taking the weight of the substrate: the substrate is weighed before and after deposition. Knowing the mass of chitosan deposited layer (m) of the surface of the titanium substrate (A) and the density of the aqueous solution of chitosan (ρ), a first estimate is obtained for the film thickness:

$$e = \frac{Mf - Mi}{\rho \times A} \qquad (1)$$

Thickness of coating of chitosan is grouped in the following Table 2:

Polishing paper	Thickness (µm)
Paper 320 SiC	119,30
Paper 500 SiC	124,17
Paper 800 SiC	126,61
Paper 1000 SiC	160,70

Table 2: The thickness of coatings obtained by superposing layers of chitosan

The trying times on titanium substrate show the thicknesses of the layers on top of the titanium are about 160 µm or less.

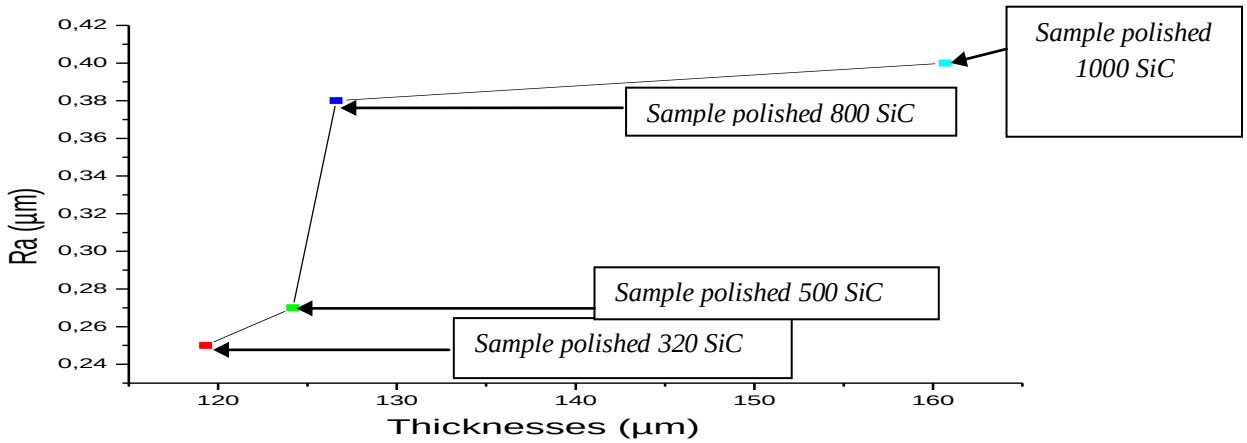

Figure 5: the roughness profile of the coated substrate according to the coating thickness

The thickness of chitosan coating was about 119 and 160 microns. The reason why the layer was thicker chitosan with a molecular weight which was higher. In our case, the final roughness essentially as a function of changing the thickness of the deposit.

4.4 The surface morphology of the deposits by optical microscopy:

The morphology after surface modification shows a homogeneous appearance with no cracks existence of pores. The images that the coating had a macroporous structure in Fig.6. This will be beneficial for the application would in the study. The sample was polished with 1000 SiC has a better condition for implantation.

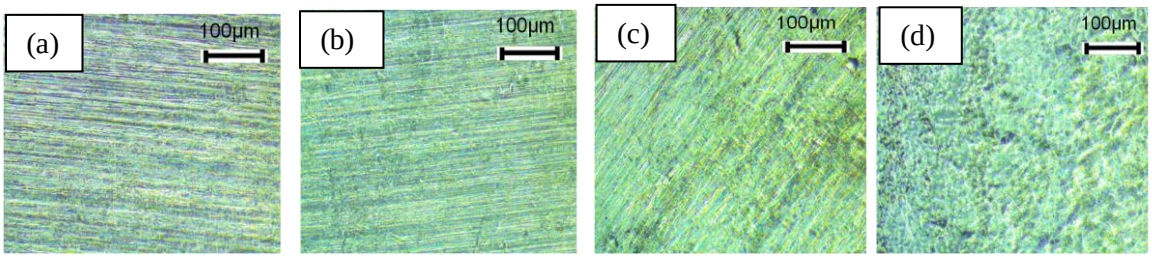

Figure 6: Morphology (optical microscope) for different substrate polished titanium coated a) Polished 320 SiC b) Polished 500 SiC c) Polished 800 SiC. d) Polished 1000 SiC.

First, we present the results of the influence of the surface roughness of the initial substrate of the final coating thickness and surface roughness after the final coating.

4.5 Adhesion:

This test can qualify substrate deposition systems the technique consists in making a scratch on a sample displacement under a point. This tip is diamond. She pulls the surface of the deposit under the effect of a load 500 gF in a movement which is constant. The alteration of the deposit can be observed by optical microscope in Fig.7.Different types of damage may be observed on the surface of the deposit in fig.7 the appearance of cracks represents constraints applied by the frictional forces of the indenter with the coating. In our case, the samples that are more ductile 1000 SiC, 800 SiC and 500 SiC against for sample 320 SiC the effect of crack propagation is observed in Fig.7.a.

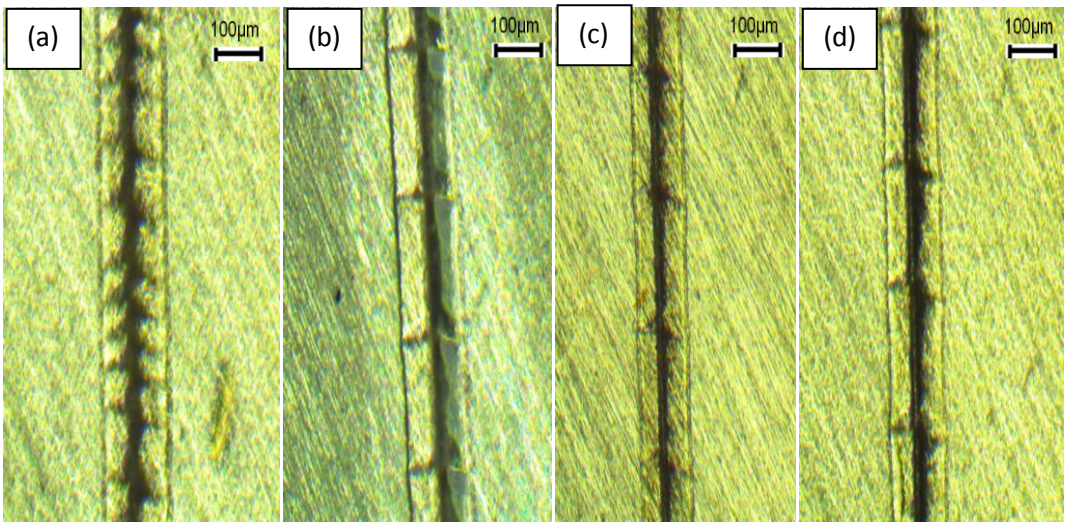

Figure 7: Images by optical microscope scratch marks on the four chitosan coatings. (a) Sample 320 polished SiC. (b) Sample polished 500 SiC. (c) Sample polished 800SiC (d) Sample polished 10000 SiC.

The indentions marks produced following a microhardness test were examined using light microscopy to determine the effect of stress on the chitosan coatings. As shown in Fig.8. However, it should be noted that the cracks do not extend outside of the footprint, or the cracks reach the surface of the metal by the comparison shows that the values of the depth and thickness of coating each sample in Fig.9. Outside the box, there was no change in the topography of the coating. The lack of change in outside area constraint indicates that the coating can absorb applied stress.

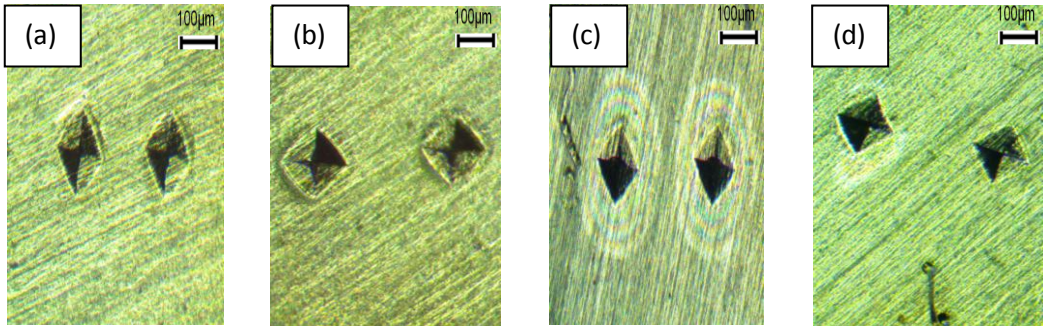

Figure 8: Images by optical microscope Impressions produced on four sample chitosan coatings. (a) Sample 320 SiC. (b) Sample 500SiC. (c) Sample 800 SiC (d) Sample 1000 SiC

The maximum depth obtained using a load of 500 gF was 26µm. The results showed represent mean values of five the indention marks for each sample.

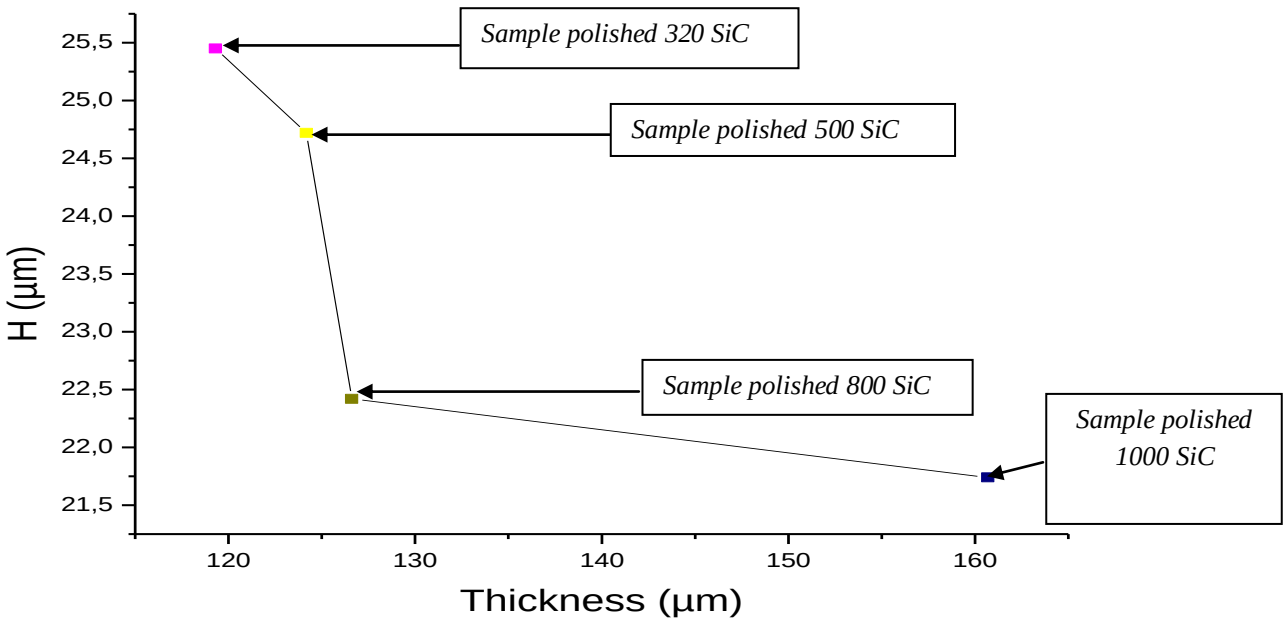

Figure 9: Depth profile of indentation marks depending on thickness of revetment

4.6 Biological evaluation by the developed coating deionized water:

This test system described substrate deposition deionized water. Another parameter of interest is to study the permeability of the surface. It is accessed by a drop of water deposited on the surface. A porous structure may facilitate the incorporation of inorganic ions, very important in the interaction between the implant of titanium and the organic medium is shown in the results.

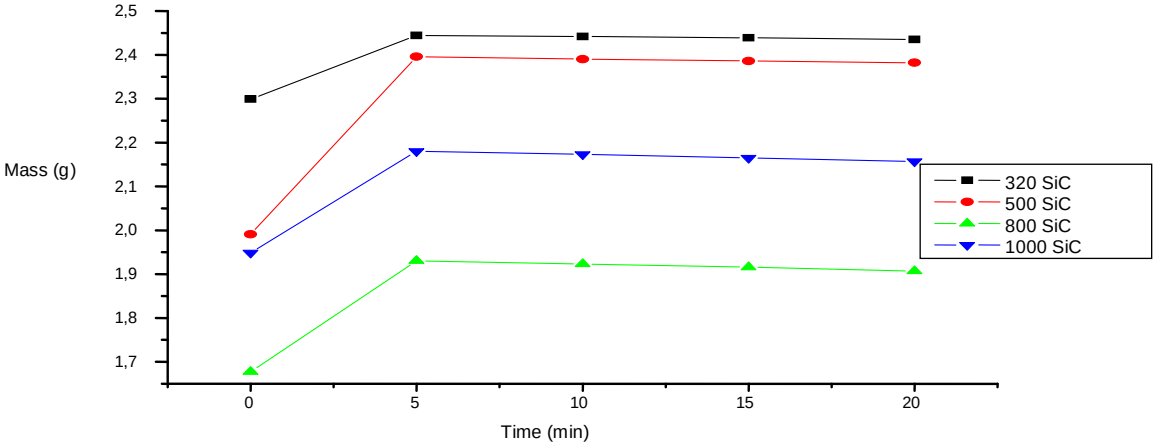

Figure 10: Influence of the drop of water on the coated substrate for 20 min.

The coatings were studied by measuring the influence of biological fluids by mass taken, as shown in Fig. 10 by increasing contact time it is observed that the substrate coated by chitosan returns almost to its original mass has a certain time so that the beneficial hydrophobic coating for applying transplantations.

5. Conclusion:

The purpose of this study was to evaluate a chitosan coating on the substrate of pure titanium layer by layer method under the conditions described so far have a rigorous structure throughout the four samples , the influences by the coating roughness initial substrate, and the number of layers deposited a thick layer can be an effective chemical barrier against the release of metal ions, potentially toxic , from the implant in the same vein, one can also think that the protection the substrate against corrosion.
Surface conditions of chitosan coating are highly desirable in the sample polished by paper 1000 SiC , as the ultimate goal of this research is to improve the bond between the coating and the metal as protection against all patient infections.

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