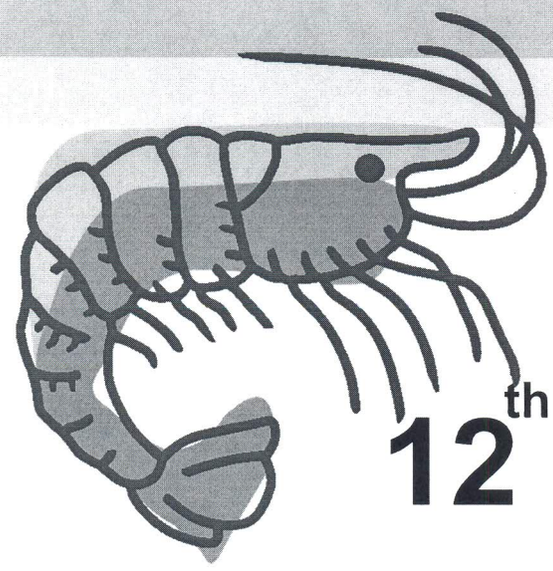
SIAQ

Sociedad Iberoamericana de Quitina



# 6th Iberoamerican Chitin Symposium

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# 12th International Conference on Chitin and Chitosan

**VI SIAQ / XII ICCC**
**September, 02-05, 2012**
**Fortaleza / Brazil**

# ABSTRACTS

# 6th Iberoamerican Chitin Symposium

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# 12th International Conference on Chitin and Chitosan

## September 2nd to 5th, 2012

## Fortaleza, Brazil

## Iberoamerican Chitin Society Board

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## CHITOSAN IMMOBILIZATION ON $SiCl_4$ COLD PLASMA TREATED GLASS SURFACE

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The modification of surfaces by cold plasma has been employed extensively for a large range of substrates such as textiles, polymers, ceramics and metals [1]. Depending on the applied power, the plasma can generate a macromolecular network that will retain or modify the structural characteristics of the starting material. The cold-plasma treatment for the surface-implant of reactive functionalities is a technology suitable for biocompatibility improvement of inert materials [2]. The activation of a surface such as glass, has many technological interests. A biocompatible surface can lead to potential uses as an organophilicity substrate, with especial interest in organics immobilization. The immobilization of chitosan can be relevant to many biochemical applications such as in biosensor or filtration device [2].

In the present work flat surfaces, from commercial soda-lime-silicate glass slides, were treated in $O_2$ and $SiCl_4$ cold plasma environment. After plasma glow the surfaces underwent subsequent chemical reactions (derivatization), by inserting reactive functional groups under gas atmosphere. At the end of the process, medium molecular weight chitosan was attached to the surface. Each step of surface tailoring was followed by X-ray photoelectron spectroscopy (XPS), and the main elements change or implanted were identified along the treatment.

For that commercial glass slides (Med Glass) (24 x 60 x 1 mm) underwent plasma treatment in a RF capacitive rotating chamber reactor (13.56 MHz) excited to 100W. The reactor was cleaned with $O_2$ and argon at low pressure filled with silicon tetrachloride $SiCl_4$ and the plasma ignited. Detail of experimental procedure can be find in [3]. Chemical derivations were carried out after plasma exposure by keeping the samples inside the evacuated reactor and allowing chemical penetration under controlled flow. The whole treatment sequence can be summarized as: $SiCl_4$ plasma (4 min) → Ethylene diamine (1 h) → Oxalyl chloride (1 h). XPS (Perkin-Elmer) was used to evaluate composition changes in all steps of functionalization. Immobilization of chitosan was done according to the following procedure: each batch of functionalized sample was kept immersed in aqueous solution of 2g/l chitosan fo r5 sec. Fig. 1 schematically presents the desired chemistry sequence.

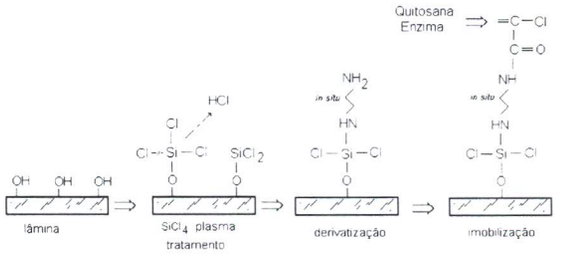


**Fig 1** - Schematic sequence of probable reactions between silanois groups and $SiCl_4$ plasma. Futher chemical derivatization using ethylene diamine tetra acetic acid and oxalyl chloride forming structures suitable for chitosan covalent attachment

To check the final biocompatibility all treatment steps were surveyed using XPS, which showed typical spectrum of a soda-lime to glass surface, with core-level lines collected for the O 1s, Si 2p, Si 2S, Na 1s and adventitious C 1s. Plasma-modified material indicates changes in composition near the surface with the present of high relative C and Si atomic concentrations, with significant reduction on oxygen density on surface, being such peaks characteristics of SiCl4 interaction. The relative element concentrations is presented in Table 1.

**Table 1** - Relative atomic concentration (%) according to XPS survey.

| | Elements (%) | | | | | |
|---|---|---|---|---|---|---|
| **Materials** | **Na** | **C*** | **Cl** | **N** | **O** | **Si**** |
| Glass | 8.6 | 2.2 | 4.6 | 2.3 | 52.9 | 30.3 |
| Plasma $SiCl_4$ | 12.1 | 8.1 | 9.2 | 2.9 | 35.2 | 32.5 |
| Reaction 1 ($C_{10}H_{16}O_8N_2$) | 11.9 | 19.2 | 9.5 | 2.3 | 27.4 | 29.7 |
| Reaçtion 2 ($C_2O_2Cl_2$) | 8.2 | 8.2 | 4.3 | 1.3 | 67.7 | 10.3 |
| Immobilized chitosan | 0.6 | 28.6 | 0.6 | 8.6 | 58.9 | 2.7 |

*(C 1s+C 2s); **(Si 1s+ Si 2p)

It has been demonstrated that from imnplanted SiClx functionalities an extremely reactive surface is attained. These functionalities acts as sites to the formation of subsequent covalent layers allowing strong organic attachment. From these results, it appears that the glass-chitosan complex developed through the present work can be used as high performance biocatalysts for various chemical processing applications, particularly for use in harsh conditions.

## REFERENCES

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