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BACTERICIDE EFFECT OF POWDER GLASSES SUBMITTED TO Na^{+}/Ag^{+} IONIC EXCHANGE IN IONIC MEDIA CONTAINING DIFFERENT CONCENTRATION OF $AgNO_3$

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Abstract. This work presents preliminary results of powder glasses with bactericide effect obtained by ionic exchange between sodium ions, present in glass composition, and silver ions, present in the ionic exchange medium. The powder glass was submitted to ionic exchange in ionic medium containing different concentration of silver species. The results showed a bactericide effect dependence on $AgNO_3$ concentration in the ionic medium. Agar Diffusion Test on *Escherichia coli* bacteria, EDS analyses was applied to the samples; the results showed there is a critical concentration of silver ions incorporated in the powder glass and a limit to bactericide effect. The tests and analyses reveled that 6 wt% of $AgNO_3$ in ionic medium was the critical concentration.

Introduction

The action of bacteria and fungus in home, industry and hospital environments offers high risk to human health. These microorganisms present potential to proliferate in many environments used by people, representing a big concern in public health programs. In this context, the development and use of antimicrobial materials has been an efficient way to handle risk situations [1-4]. In recent years many works had been developed on biocide materials applied to polymer and ceramic materials [3-5].

Nowadays, glass materials are a special class of biocide materials developed with high interest because its bactericide action is lingering. The ionic specimens with active principle presents in the structure glass is gradually set free during long time, providing a long time of the bactericide action for the products [6]. These materials has been applied to produces coverings for ceramics parts with bactericide property for use at home ambient, at the hospitals ambient, at the laboratory ambient and many others applications. These glasses in the powder form can be applied as additive in many products, for example, as polymeric additive, as additive for inks, as composites additives and ceramic products.

A method to develop biocide properties in these materials consists to incorporate the metallic ions capable to eliminate bacteria (bactericide elements) and/or fungi (fungicide elements) into the glass structure. Some metallic ions present oligodinamic properties and are used by industries in the production of biocide materials. The oligodinamic properties are manifested by the action of these ions on microorganisms, inhibiting their reproduction or causing their death. Some ions present these proprieties: the more studied ions are, silver, titanium, mercury and cupper [7]. Many works report the use of ionic exchange processes incorporating Ag^{+} and Cu^{++} into glass structure [8].

This work presents study about of $AgNO_3$ concentration in an ionic medium used to develop ionic exchange processes between sodium ions, present in glass composition, and silver ions, present in the ionic medium, this one responsible for the glass bactericide properties. A powder glass, used as a substrate, was submitted to ionic exchange treatment and to microbiological analysis after a specific time. EDS techniques was used to evaluate silver incorporation in the glass structure.

Experimental

Glass development

The glass was prepared following composition and methodology developed by Angioletto et al. [9], table 01. The raw materials were first melted at 850 ^{o}C during 30 min with a heating rate of 10 $^{o}C/min$, then at 1450^{o}C during 120 min, same heating rate. In sequence, the obtained glass was crushed bellow 40 μm particle size and submitted to ionic exchange treatment in $AgNO_3$ ion media at different concentrations. Although is know that the particle size affect the exchange area was opted to an only one granulometric distribution to simplify this work. In this work the exchange area was considerate constant.

Table 1 – Glass Formula (Angioletto et all.), all values in wt%.

Al_2O_3	SiO_2	Li_2O	Na_2O
3,0	72,0	5,0	20,0

Ionic Exchange Treatment

The ionic exchange treatment between the prepared glass (containing sodium oxide) and the ionic medium (containing silver ions) was carried out using $AgNO_3$ as silver source and $NaNO_3$ as melted medium (Reagan, 99,9% purity). The powder glass was immersed into the ionic medium and submitted to 430 ^{o}C over 4 hours in a muffle oven. Both processes were carried out in different ionic media, maintaining constant the sodium concentration but varying $AgNO_3$ concentration, 0,430 wt% to 10,750 wt%.

After ionic exchange treatment, the powder glasses were washed in water during 2 days. These procedures are due to dissolve the sodium residues incorporated on glass surfaces during the ionic exchange process. The powder glasses were then dried in a vacuum system and crushed again below 40 μm particle size.

Microbiological Tests

The powder glasses were submitted to microbiological analysis to evaluate their bactericide effects. The Agar Diffusion Test was used for each sample (containing different silver concentration in the glass structure) and applied to *Escherichia coli* bacteria.All tests were conducted at 36 ^{o}C and the bacteria were submitted to 18 hours incubation period. For all samples microbiological tests were carried out to evaluate the ionic medium influence on the production of a biocide glass; the tests were conducted in Petri dishes containing 0,300 g of powder glass.

Analysis of Silver Incorporation in Powder Glass Samples

After ionic exchange treatment, the powder glass samples were submitted to EDS technical to qualitative analysis to determine the silver ion incorporation degree into the glass matrix.

Results and Discussion

Silver ion incorporation into the glass matrix was studied using EDS techniques, Figure 1. The results showed that until 6,00 wt% concentration of $AgNO_3$ in the ionic medium occur the increase of the Ag elements into glass matrices. This concentration correspond a critical concentration of $AgNO_3$ in the ionic medium, where the silver concentration into glass matrices doesn't present the exponential dependence, but a saturation regime, Figure 2.

The saturation regime was expected because the sodium concentration in the matrix glass is limited. The literature reports that the ionic exchange mechanism is dependent of the sodium concentration in the glass matrix [10]. The sodium ion amount in the glass defines a constant flow exchange and, for equal periods of time, guarantees the same ion amount in the glass matrix. Consequently, after an $AgNO_3$ critical concentration has been achieved, there is a saturation regime for ionic exchange and a constant concentration of silver ions in the powder glass is obtained.

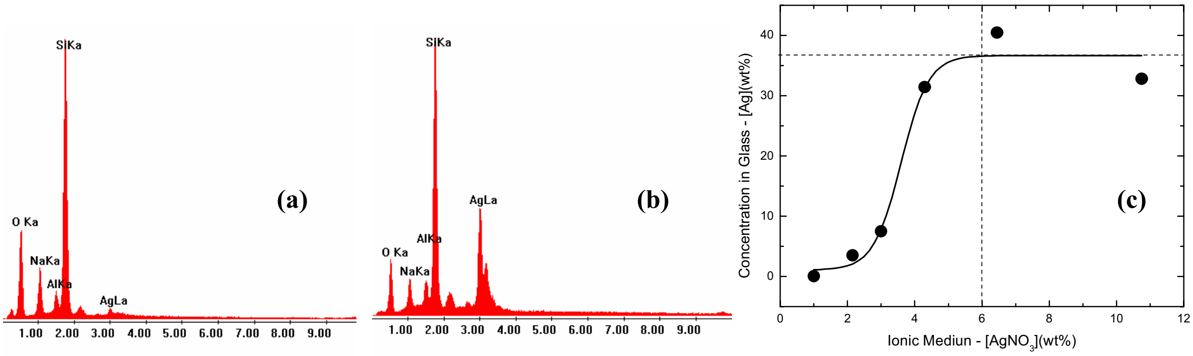

Fig. 1 – EDS spectra of the glass matrices after ionic exchange in the ionic medium containing (a) 0,43 wt% of $AgNO_3$ and (b) 10,75 wt% of $AgNO_3$. (c) The comportment of the concentration of the Ag into glass matrices after ionic exchange with different concentrations of the ionic medium.

The powder glass biocide action was tested using the Agar Diffusion Test to *Escherichia coli* bacteria. The results showed a different bactericide action depending on the concentration of silver ions incorporated in the glass. The glass obtained in medium containing 2,15 wt% of $AgNO_3$ presented a poor effect, while all glasses obtained in media containing concentrations above 6,00 wt% of $AgNO_3$ presented the maximum effects. Figure 2 presents results of Agar Diffusion Test to some samples.

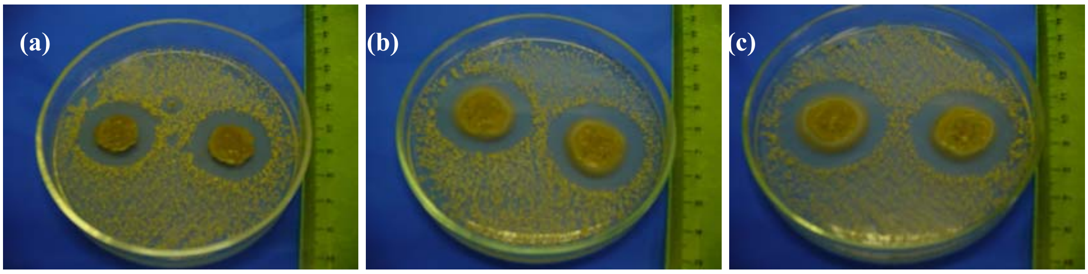

Fig. 2 – Agar Diffusion Tests for powder glasses after ionic exchange in the ionic medium containing different concentrations of AgNO3: (a)2,15 wt%, (b)4,30 wt% and (c)10,75 wt%.

In order to evaluate the bactericide action at the Agar Diffusion Tests, the bactericide action (A_{bac}) was defined by total areas of the health halo, considering a perfect circle. Figure 3 shows the bactericide area versus $AgNO_3$ concentrations in the ionic medium. The results present the dependence of bactericide action with silver ion concentration in the powder glass. Therefore, there is a critical concentration to occur bactericide effect, i.e. 6,00 wt% of $AgNO_3$ concentration in the ionic medium.

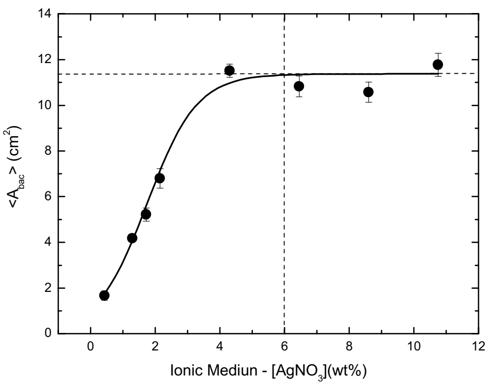

Fig. 3 – Bactericide action by Agar Diffusion Test for powder glasses submitted to ionic exchange between Na+ and Ag+ ions in the ionic medium containing different concentrations of AgNO3.

These results were expected because the incorporation of silver ions in the powder glass is limited by the concentration of sodium ions in glass matrices. The bactericide effect depends on the absorption of silver ions by the bacteria and on the number of free ions present at the external medium. The number of free ions present at the external medium defines the number of bacteria that will be eliminated. When the amount of silver ions in the glass powder increases, then a major quantity of free ions are available to the external medium, increasing the number of bacteria that can be eliminated. As a result, it was observed that an increase in the bactericide area at Agar Tests depends on the concentration of $AgNO_3$ in the ionic medium.

The dependence of the bactericide areas with $AgNO_3$ concentration is coherent with the incorporation results of silver ions in the glass matrices obtained by EDS. For $AgNO_3$ concentrations above 6wt% the bactericide areas present a saturation regime because the concentration of silver ions incorporated in the powder glass is constant (saturation regime for ionic exchange). These results show that the number of free silver ions liberated depends on the concentration of silver ions incorporated in the powder glass. Therefore, the liberation of silver ions to the external medium occurs by diffusion mechanisms and presents a saturation regime.

The exponential behavior of the bactericide area until 6 wt% of $AgNO_3$ shows a bactericide effect subordinated to diffusion mechanisms necessary to transport silver ions into the external medium that increases with silver concentration in the glass matrices. Also, the bactericide effect presents an $AgNO_3$ critical concentration in the ionic medium. This result was also expected because the number of silver ions incorporated in the glass matrices was limited by the number of sodium ions in the glass during ionic exchange. So, consequently, the number of free silver ions will be constant, limiting the bactericide effect.

Conclusions

The concentration of $AgNO_3$ in the ionic medium and sodium ion in glass matrices influences the mechanisms developed during the ionic exchange between Na^+ and Ag^+ and, consequently, the bactericide effect of the biocide glass. The amount of sodium in the glass defines a critical concentration of silver ions in the glass matrices and a critical value to bactericide efficiency of the precursor glass.

The biocide glass presented good bactericide properties. So, controlling the $AgNO_3$ concentration in the ionic medium makes possible to produce a glass with controlled bactericide effects for different bacteria.

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