



**Cairo University**  
**Faculty of Science**

# **PREPARATION AND CHARACTERIZATION OF SOME ANTIBACTERIAL GLASSES**

*A Thesis*

Submitted in Partial Fulfillment of the Requirements  
For  
The degree of  
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**Chemistry**

*By*

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## تحضير وتوصيف بعض أنواع من الزجاج المضاد للبكتيريا

رسالة مقدمة

كجزء متمم للحصول على درجة

ماجستير العلوم فى

الكيمياء

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## ABSTRACT

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**Title of thesis:** "Preparation and Characterization of Some Antibacterial glasses"

**Degree** : M.Sc. Thesis (Chemistry), Faculty of Science, Cairo University, 2009.

### **Abstract**

This work was carried out to investigate the antibacterial effects of some undoped and silver-doped  $P_2O_5$  - CaO -  $Na_2O$  glasses against *S.aureus*, *P.aeruginosa* and *E.coli* micro-organisms using agar disk-diffusion assays. Glass forming regions in the ternary  $P_2O_5$  - CaO -  $Na_2O$  and in the quaternary  $P_2O_5$  - CaO -  $Na_2O$  -  $Ag_2O$  systems were determined. Density, molar volume, dissolution of glass in water, pH changes of water during glass dissolution, and concentrations of silver ions released from silver-doped glasses into water during their dissolution were determined. The structures of some glasses were studied by XRD, FT-IR, and UV-VIS spectroscopy. The tested silver-free and silver-doped glasses demonstrated different antibacterial effects against the tested micro-organisms. For silver-free glasses, an increase in inhibition zone diameter (zone of no bacterial growth) was seen with the increase in the glass dissolution rate and with the decrease in water pH. Silver-doped glasses showed an increase in inhibition zone diameter with increasing  $Ag_2O$  content. An increase in concentrations of silver ions released from silver-doped glasses into water was seen with increasing time of glass dissolution and with increasing  $Ag_2O$  content. The dissolution rates of  $P_2O_5$  - CaO -  $Na_2O$  glasses decreased with increasing CaO content and slightly decreased with gradual replacement of  $Na_2O$  by  $Ag_2O$ .

**Keywords:** Antibacterial glasses; Silver-doped phosphate-based glasses; Glass dissolution; Controlled release; Silver ions; Antibacterial effect.

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ملخص البحث :

تختص هذه الرسالة بدراسة التأثير المضاد للبكتيريا لبعض التراكيب الزجاجية فى النظام  $P_2O_5 - CaO - Na_2O$  و  $Ag_2O$  و المحتوية على ٠ و ٠.٥ و ١ و ٢ مول % من  $Ag_2O$  وذلك على بعض أنواع من البكتيريا مثل *S.aureus* و *P.aeruginosa* و *E.coli* باستخدام طريقة (Agar disk-diffusion). وقد تم تحضير العديد من التراكيب الزجاجية فى النظام الثلاثي  $P_2O_5 - CaO - Na_2O$  و فى النظام الرباعي  $P_2O_5 - CaO - Na_2O - Ag_2O$  باستخدام طريقة الصهر التقليدية وذلك بهدف تحديد مدى التراكيب الكيميائية فى هذه الأنظمة والتي تعطى عند صهرها زجاجا. تمت دراسة بعض الخواص مثل الكثافة و الحجم المولارى و ذوبان الزجاج فى الماء و تغير الرقم الهيدروجيني للماء أثناء ذوبان الزجاج. بالإضافة الى تقدير تركيزات أيونات الفضة الذائبة فى المحاليل المائية. كما تم دراسة التركيب البنائى لبعض التراكيب الزجاجية وذلك باستخدام حيود الأشعة السينية و إمتصاص الأشعة تحت الحمراء و الأشعة المرئية و فوق البنفسجية. وقد بينت نتائج إختبارات المزارع البكتيرية أن جميع التراكيب الزجاجية المختبرة ( المحتوية و غير المحتوية على  $Ag_2O$  ) لها تأثيرات مضادة للبكتيريا وذلك بدرجات متفاوتة تبعا لنوع البكتيريا ( $S.aureus > P.aeruginosa > E.coli$ ) و تركيب الزجاج و تركيز  $Ag_2O$  به. و قد لوحظ أن التأثير المضاد للبكتيريا للتراكيب الزجاجية غير المحتوية على  $Ag_2O$  يزيد مع زيادة معدل ذوبان الزجاج و أيضا مع زيادة النقص فى الرقم الهيدروجيني. وبالنسبة للتراكيب الزجاجية المحتوية على  $Ag_2O$  فقد لوحظ زيادة التأثير المضاد للبكتيريا بزيادة تركيز  $Ag_2O$  فى الزجاج. كما تبين من دراسة ذوبان الزجاج فى الماء أن معدل الذوبان للتراكيب الزجاجية فى النظام الثلاثي  $P_2O_5 - CaO - Na_2O$  يقل بزيادة تركيز  $CaO$  و أيضا يقل معدل الذوبان للتراكيب الزجاجية فى النظام الرباعي  $P_2O_5 - CaO - Na_2O - Ag_2O$  بزيادة تركيز  $Ag_2O$ . وقد أوضحت قياسات الإمتصاص الذرى لمحاليل الذوبان أن تركيز أيونات الفضة فى المحلول يزيد بزيادة كلا من وقت ذوبان الزجاج و تركيز  $Ag_2O$  فى الزجاج. كما بينت دراسة إمتصاص أطيااف الأشعة المرئية و فوق البنفسجية ظهور قمة إمتصاص عند طول موجى ٢٣٠ نانو متر تقريبا و التى تبين أنها ناتجة عن الإنتقالات الإلكترونية  $[4d^9 5s^1 \rightarrow 4d^{10}]$  لأيونات الفضة.

**الكلمات الدالة:** الزجاج المضاد للبكتيريا ؛ زجاج فوسفات الفضة ؛ أيونات الفضة ؛ التأثير المضاد للبكتيريا ؛ ذوبان الزجاج.

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## List of symbols and abbreviations

<b>W.H.O</b>	World Health Organization.
<b>DNA</b>	Deoxyribonucleic Acid.
<b>G +</b>	Gram-positive.
<b>G -</b>	Gram-negative.
<b><i>E.coli</i></b>	<i>Escherichia coli</i> .
<b><i>P.aeruginosa</i></b>	<i>Pseudomonas aeruginosa</i> .
<b><i>S.aureus</i></b>	<i>Staphylococcus aureus</i> .
<b>MRSA</b>	Methicillin-Resistant <i>Staphylococcus Aureus</i> .
<b><i>C. albicans</i></b>	<i>Candida albicans</i> .
<b>ppm</b>	Part per million.
<b>ppb</b>	Part per billion.
<b>NASA</b>	National Aeronautic Space Administration.
<b>ASTM</b>	American Society for Testing Materials.
<b>PBGs</b>	Phosphate-Based Glasses.
<b>CRGs</b>	Controlled Release Glasses.
<b>Q<sup>n</sup></b>	PO <sub>4</sub> group with <i>n</i> = number of bridging oxygen atoms.
<b>BO</b>	Bridging oxygen.
<b>NBO</b>	Non-bridging oxygen.
<b>NMR</b>	Nuclear Magnetic Resonance.
<b>T<sub>g</sub></b>	Glass transition temperature.
<b>XRD</b>	X - Ray Diffraction.
<b>FT-IR</b>	Fourier - Transform Infrared spectroscopy.
<b>UV-VIS</b>	Ultraviolet - Visible spectroscopy.

## Symbols and abbreviations

<b>ICP-OES</b>	Inductively Coupled Plasma - Optical Emission Spectroscopy.
<b>SBF</b>	Simulated Body Fluid.
<b>SEM</b>	Scanning Electron Microscopy.
<b>XPS</b>	X-ray Photoelectron Spectroscopy.
<b>ESEM</b>	Environmental Scanning Electron Microscopy.
<b>HBSS</b>	Hank's Buffered Saline Solution.
<b>PGFs</b>	Phosphate-based glass fibres.
<b>SHMP</b>	Sodium Hexa-Metaphosphate ( $50\text{Na}_2\text{O} - 50\text{P}_2\text{O}_5$ ).
<b>EDX</b>	Energy Dispersive X-ray.
<b>MAS -NMR</b>	Magic Angle Spinning - Nuclear Magnetic Resonance.
<b>XANES</b>	X-ray Absorption Near Edge Structure.
<b>HEXRD</b>	High Energy X - Ray Diffraction.
<b>CFUs</b>	Colony-Forming Units.
<b>CDFE</b>	Constant depth film fermenter.
<b>2D-NMR</b>	Two-dimensional NMR.
<b>TEM</b>	Transmission Electron Microscopy.
<b><i>C.difficile</i></b>	<i>Clostridium difficile</i> .
<b>FAAS</b>	Flame Atomic Absorption Spectrometry.
<b>M<sub>a</sub></b>	Mass of glass sample in air.
<b>M<sub>L</sub></b>	Mass of glass sample in liquid.
<b>DIN</b>	Deutsches Institut für Normung (The German Institute for Standardization)
<b>ISO</b>	International Standards Organization.
<b>MIC</b>	Minimum Inhibitory Concentration.
<b>ATCC</b>	American type culture collection.

## Symbols and abbreviations

<b>GFR</b>	Glass-Forming Region.
<b>D<sub>r</sub></b>	Dissolution Rate.
<b>a.u.</b>	arbitrary units.
<b>IZD</b>	Inhibition Zone Diameter.
<b>T<sub>m</sub></b>	Melting temperature.
<b>CLD</b>	Cross-Link Density.
<b>δ</b>	Bending vibration mode.
<b>ν<sub>s</sub></b>	Symmetric stretching vibration mode.
<b>ν<sub>as</sub></b>	Asymmetric stretching vibration mode.
<b>ATP</b>	Adenosine Tri-Phosphate.
<b>RNA</b>	Ribonucleic Acid
<b>NADH</b>	Nicotinamide Adenine Dinucleotide Hydrogenase.

## APPROVAL SHEET FOR SUBMISSION

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