

Study of a Photodegradant Polymeric Composite Containing TiO₂ and Glass Residue

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Abstract

Photodegradation or photocatalysis is a chemical degradation process that occurs when an inorganic semiconductor is exposed to ultraviolet (UV) light. UV light (wavelength 320 - 400 nm) has enough energy to detach an electron from the last layer of the semiconductor, leading to the conduction band, leaving a hole in the valence band. In these bands, chemical reduction and oxidation reactions occur, respectively. These reactions degrade diverse surface dirt, dissociating them into simpler and less offensive substances such as CO₂ and H₂O. In this work, we studied the potential of photocatalysis of a composite based on a semiconductor encapsulated in epoxy resin, in the degradation of *Staphylococcus aureus*, pathogen with a high degree of hospital contamination, in order to apply it to the construction in hospital facilities. The experiments were carried out with a fabrication of only epoxy resin tablets and tablets with the composite, at various concentrations of the semiconductor and glass powder. Through contamination of these tablets and their exposure to sunlight and the ambient light, contamination on their surfaces was verified. The results indicated potential photodegradation capacity of the composite.

Keywords

Photodegradation, Semiconductor, Epoxy Resin, Composite

1. Introduction

The healthcare-associated infections (HAIs) are vectors of a severe public health problem in Brazil and worldwide. According to [1], there is, besides of financial

and social costs, the dissemination risks of antibiotic resistant bacteria.

It is one of the factors for these infections occurrences the non-efficient cleaning procedures. Studies done by [2] verified that the cleaning, as has been done, just causes the displacement of microbial loads. In [3], a survey was done about *Staphylococcus aureus* surface contamination (on bed railings, bedside tables, doors knobs and nursery floors of the Clinics Hospital of Uberlândia Federal University), and it identified that 50% of the nurseries were contaminated.

Caring for biosafety is another important factor to avoid these occurrences and, some health professionals do not take it seriously. On a technical report, [4] describes biosafety failures found in a São José University Hospital (Belo Horizonte—Minas Gerais, Brazil) as remains of bandage, cirurgical materials and biological waste on the floor, not to mention that those responsible for the sector did not use the obligatory personal protective equipment (PPE). In addition, we verified that the nursing technicians did not do the correctly hands sanitation procedures and touched many patients, favoring the crusade contamination. The students who worked on the hospital wore make up, painted nails and lipstick, which propitiates the microbiota impregnation and diseases transportation.

Taking into consideration the careful procedures related to the use of PPE, the correct hands sanitization and materials disinfections, products that are responsible to disinfect the health services environments are still used. However, [5] said that the use of these products brings unwelcome secondary and dangerous effects such as skin dryness, eye irritation and mucosa; in case of some phenolic products, their use are prohibited in nursery and areas that are in contact with food, due their oral toxicity. Thus, to avoid the use of these aggressive chemical products should mean an environmental and social gain, by means of antimicrobial materials development.

One of the ways to diminish the use of aggressive products and the infecting agents quantities, is the advent of self-cleaning surfaces. The self-cleaning surfaces use incorporated catalysts, which promote chemical reactions on the surface interacting with infecting agents to prevent their actions, perhaps even killing them. This process, in which the catalysts act against the infecting agents, is called photocatalysis or photodegradation. The photocatalysis occurs by an irradiation of a photocatalyst, which is an inorganic semiconductor that has sufficient energy to induce an electronic excitation.

This energy has to be higher than the band gap. The band gap is the distance between the Valence Band (VB) and the Conduction Band (CB). Thereby, with this photonic energy, an electron-hole pair is generated, on which an electron is extracted from the VB, jumping to the CB. During this process, two regions are created, one positive hole (h^+) on the VB and the other with a free electron (e^-) on the CB [6]. These e^- and h^+ reduce and oxidize, respectively, chemical species on the photocatalysis surface, unless they recombine [7].

According to [8], these detached electrons in contact with water and O_2 form oxidizing and reducing regions. Various infectants and dirtiness, both organic

and inorganic, in touch with these regions, suffer electronic degradation, dissociating in simpler and less offensive substances, like CO₂ and H₂O. Some of these materials have already been using this mechanism, with the specific aim of removing infecting agents and dirt, besides other specific functions to which the photocatalysis can attribute.

To improve photocatalytical activity, there are studies about the use of glass mixed to the material and semiconductor. [9] used crushed recycled waste glass, by-product from drinking bottles, in replacement of part of the aggregate of the superficial layers from a photocatalytical concrete. The results showed a significant improvement on the photodegradant action. The authors justified this improvement due to light transmission property of the glass.

The aim of this paper was to study the capacity of a composite containing titanium dioxide (TiO₂), an inorganic semiconductor encapsulated in an epoxy resin, to degrade *Staphylococcus aureus* bacteria with the photocatalysis mechanism. Thus, it was verified that the photocatalytical effect of the composite, when directly exposed to the sunlight and to the ambient light (indirect sunlight and fluorescent lamp light).

The percentages of semiconductors were based on [10] work. It is expected with the results of this paper that the composite shows potential to be used as a coating on health services that, on top of its photocatalyst capacity, also has durability, easy maintenance and is less harmful to the environment.

2. Materials and Methods

2.1. Materials

The composite is formed by epoxy resin, titanium dioxide and waste glass powder. The epoxy resin is a bi component type, being used resin RP 031 with a catalyst RE 043, both from Ariston Polímeros Indústria e Comércio Ltda. The TiO₂ powder used in this experiment was Degussa P25 (Evonik®), and their characteristics are summarized in **Table 1**, according to the manufacturer specifications.

A local glazing provided the waste glass to [5], however in mud form. In the glazing, the glass edges passed through thinning process, which generate the residue but as mud. Therefore, [5] separated and turned into a powder that [11] characterized after. The physical characteristics of the waste glass powder are summarized on **Table 2**.

2.2. Composite Fabrication

The composite samples with different mixing design were made in the laboratory. Because of the tests, the chosen samples diameters were an average size of 1.5 cm with circular geometry and 1.5 mm of thickness. Using a sheet steel stamping waste (in coins form, to give the wanted geometry), a silicon mold was created to fabricate the specimens. The materials were mixed for 5 min, adding, then, the resin followed by the TiO₂ and lastly the waste glass powder. After that, the

Table 1. Properties of TiO₂ used in this experiment.

Crystal form	Percentage of TiO ₂ (%)	Specific Surface Area (BET) (m ² /g)	Particle Size (nm)
Anatase/Rutile	99.5	35 - 65	21

Table 2. Properties of waste glass powder.

Specific mass (kg/m ³)	Specific Surface Area (m ² /g)	Average Particle Size (m)
2480	0.282	19.6

catalyst was added and mixed for 2 min. Afterwards, the mixture was disposed into the mold and left for 24 h to be cured at environment temperature.

The composites had five different compositions, which are described on **Table 3** in epoxy resin mass percentages. To a better differentiation of the samples, colorful aluminum paper was added to the composites for each composition with the exception of reference samples (PN) that was translucent.

2.3. Strains and Growth Conditions

In this work, strains of *Staphylococcus aureus* (ATCC 25923) were used. Both were stemming from the supply of Microbiology Laboratory of Biomedical Science Institute from Uberlândia Federal University. The procedures were in accordance with the [12].

The strains were incubated in Brain Heart Infusion (BHI) broth (DIFCO) for 24 h at 37°C. Then, the cultivated bacteria were transferred to the Mueller Hinton Agar plates in the same temperature and for the same time, for formation of the colony. After the colony growth, they were suspended in sterile saline solution (0.9% NaCl prepared in distilled water) until 1×10^8 CFU concentration approximately or 0.5 Mac Farland scale of turbidity. This contaminated solution was used to contaminate the samples.

2.4. Composite Exposition Conditions

The samples were sterilized in a greenhouse and disposed in a recipient where they were contaminated with 0.1 mL of the *Staphylococcus aureus* suspended in sterile saline solution, and exposed to the sunlight and to the ambient light. The light exposure was divided into groups such as three with *Staphylococcus aureus* for each illumination type.

For every 15 min one specimen of each different addition was removed. A number of four specimens were used to each different concentration. Thus, in 60 min, all the specimens were collected to the analysis, as can be seen on **Figure 1**. The authors arbitrated these periods of exposition and removal.

The collected samples were transferred to a tube with 5 mL of sterile saline solution. Using an ultrasonic vibration, the bacteria that were harvested eluted on saline solution. A total of 0.1 mL was collected from this 5 mL to a Mueller Hinton Agar plate and incubated for 48 h. Afterward, a visual count of the CFU was made on the plate.

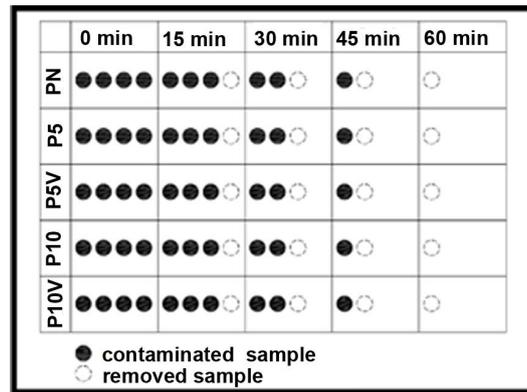


Figure 1. Scheme of sample exposition.

Table 3. Composites composition specification.

Code	Color	Percentage of addition	
		TiO ₂	Waste Glass Powder
PN	Translucent	0%	0%
P5	Pink	5%	0%
P5V	Green	5%	5%
P10	Black	10%	0%
P10V	Purple	10%	10%

3. Results and Discussion

In **Figure 2**, it can be seen that the composites produced, cured and differentiated by color. The different compositions corresponded for each color as specified on **Table 3**.

As said previously, the samples were contaminated by a saline solution containing 10^8 CFU/mL, that is, 100 million bacteria per mL. Thus, each applied 0.1 mL contained 10 million bacteria. When the specimens were transferred to a tube with 5 mL of sterile saline solution, they were diluted resulting in 2 million of bacteria per mL. The removal of 0.1 mL of this 5 mL to the Mueller Hinton Agar resulted into 200,000 CFU/specimen. Knowing the theoretical amount of CFU that remained alive after exposition, the practical amount of CFU remained was accounted for.

3.1. *Staphylococcus aureus* Sunlight Exposition

Three expositions were made to the sunlight with *Staphylococcus aureus*. It can be observed at **Table 4** that the major reduction to all expositions was performed by P10V sample, which in all cases with 60 min of exposition have had no CFU. This performance was followed by P10 sample, but not as good as P10V. A surprising phenomenon was presented by reference sample (PN), which at First and Replica exposition has zero CFU. The samples with 5% of TiO₂ - P5 and P5V - also acted against *Staphylococcus aureus* but with no clear performance as P10V.



Figure 2. Cured samples.

Table 4. *Staphylococcus aureus* CFU quantification after sunlight exposition.

Code	Period of exposition											
	First				Replica				Rejoinder			
	15	30	45	60	15	30	45	60	15	30	45	60
PN	2	15	4	0	C ^a	11	2	0	C	C	C	C
P5	C	141	1	1	C	147	30	1	C	324	285	2
P5V	C	326	0	10	C	308	1	0	C	C	C	C
P10	C	C	C	3	C	C	16	0	C	C	0	0
P10V	3	0	0	0	14	1	0	1	C	C	C	0

a. In the table, “C” means countless. This occurs when the number of CFU is higher or equal to 400.

Observing all the results, it is not perceived a big difference between the samples’ performances. Despite the P10V presented itself effective in 60 min, all the others showed good results too. One of the reasons seems to be the one proved by [13], that there is an obvious relation between the increase of sunlight exposure with the decrease of the CFU numbers.

3.2. *Staphylococcus aureus* Ambient Light Exposition

The second stage was with ambient light exposition and three expositions were made (First, Replica and Rejoinder). As the same way noticed to the sunlight exposition, here in ambient light exposition, P10V had the best performance followed by P5V, as it can be seen at Table 5. About the others specimens (PN, P5 and P10), the results were inconclusive. The results presented in the rejoinder to all specimens could not be explained.

Note that the samples that worked against the bacteria were those that possess waste glass powder. For this experiment, one more control was used.

Table 5. *Staphylococcus aureus* CFU quantification after ambient light exposition.

Code	Period of exposition											
	First				Replica				Rejoinder			
	15	30	45	60	15	30	45	60	15	30	45	60
PN	C ^b	C	C	C	C	C	C	C	C	C	C	C
P5	C	C	C	C	C	C	C	C	C	C	C	C
P5V	C	C	C	224	C	C	384	244	C	C	C	C
P10	C	C	C	C	C	C	C	C	C	C	C	C
P10V	50	65	62	49	281	71	106	88	C	C	C	C

b. In the table, “C” means countless. This occurs when the number of CFU is higher or equal to 400.

Samples were exposed to the dark, to see if the photocatalysis would work and the results was countless for all the specimens.

Considering the condition “C” countless as the presence of more than 400 CFU, for the sample P10V, the quantity of colony-forming unit decreased at least, for the worst case (ambient light), 30% in 15 min and 78% in 60 min.

Comparing the obtained results, the composites were better on contamination reduction when exposed to the sunlight than to the ambient light. As was said previously, the sunlight has the natural capacity to kill bacteria. The possible explanation to that is the irradiated wavelength by the sunlight, which kills the bacteria, however no specific study was done.

The waste glass powder, as identified by [9], seemed to have contributed to the photodegrading, taking into account that, in 45 min, the samples with this compound presented better results than the samples with the same semiconductor concentration without the waste glass powder.

4. Conclusions

According to the methodology and conditions adopted in this work, it was verified that the composite formed by epoxy resin, titanium dioxide and waste glass powder, in most of done experiments, achieved the goal to degrade bacteria.

The results suggest that the ideal concentration of the waste glass powder and titanium dioxide compositions is around 10% in mass of resin, in view of the composite, which presented the best results, was that formed by 10% of titanium dioxide and 10% of waste glass powder addition.

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