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EFFECT OF THE TEMPERATURE IN THE ANTIMICROBIAL ACTION OF THE BACTERICIDE WOOD POLYMER COMPOSITE - BWPC

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Abstract. These work present studies about influence of the high temperature at the antimicrobial properties of the Bactericide Wood Polymer Composites (BWPC) after exposition during different times at 110 ^{o}C. The composite was formulated containing Polypropylene (PP) as matrix polymeric, wood powder of the species *Pinus* and Triclosan additive as bactericide agent. The BWPC was exposited during different times in the ambient and submitted to microbiological tests. The Agar Diffusion tests were applied with two kind bacteria, the *Echerichia coli (EC - Gram Positive)* and *Staphylococcus aureos (SA - Gram Negative).* The FT-IR and TGA techniques were utilized to available of the temperature effect in the chemical structure of the composites BWPC. The studies showed a strong dependence of the bactericide action of the composites with exposition time at 110 ^{o}C and a constant bactericide action after 100 hours of exposition.

Introduction

The increase of the industrial competitiveness and the need to develop products with new joined properties has been important factors for the development of materials with antimicrobial properties. The need of use of these materials is growing, being that many microorganisms can be noxious to human health. In the development of bactericidal materials with satisfactory efficiency many works have been accomplished involving several classes of materials, as well as polymeric bactericide materials [1]. However, such materials don't only present a high application potential in this industrial segment, but also in segments that involve products with medical applications, deontological, pharmaceutical and other applications, allowing be still that used as matrix bactericide polymeric for materials composites. In the last two decades, many development's works were realized of composites the base of polymer and wood elements [2-4]. Considering a polymeric composite, the wood mass is substitution element of the polymeric resin, that in the great majority, has a relatively high cost, while the wood residue can be found with low cost.

The high technological growth lived by the humanity in the last decade has been requesting of the materials many special properties. The application of the WPC materials comes increased significantly, being now very used by the segments of the building site, furniture's industries, wooden shoe's industries and many others. Potentially, due to easy processing, such materials with bactericide propriety can be applied in the making of products of home use, public sanitariums, ambient of laboratories and of hospitals and many others. In the scenery of the public health, seeking to contribute for the prevention of diseases originating from of bacterial infections, the incorporation of special properties, as well as bactericides in such materials, it can present an efficient form and relatively cockroach of minimizing the appearance or the evolution of bacterial focuses in objects of great use.

The application of the composite polymer/wood, with bactericide properties (BWPC - Bactericide Wood Polymer Composite), for the fabrication of bactericide products can be a cheap alternative for use of the great part of the society. Still, in the powder form, the bactericide composite can be applied in aviaries or in others ambient as a cheap alternative for the prevention of bacterial diseases in animals domestic or created in grand scales.

This work presents studies about the influence of the high temperature in the antimicrobial properties of the Bactericide Wood Polymer Composites (BWPC) after different times of the exposition at 110 ^{o}C. Such information are important for the thermal characterization of the material and to know the bactericide behavior of the BWPC during its exposition at different ambient with high temperature. This information will be useful to define the applications and processing conditions for a product with BWPC.

Experimental

The Polypropylene was utilized as composites matrix and wood flour as composites elements. The wood *Pinus elliot* specimen was utilized with size of the particle between 28 to 100 mesh. The wood was submitted to drying during 100 minutes at 110 ^{o}C. The mixture between polypropylene, dry wood flour and the antimicrobial additive it was made with a homogenate system at ambient temperature. The antimicrobial additive utilized was a commercial additive that it contains a composition of Triclosan (CIBA Chemical). The BWPC was prepared containing 50 % of wood flour, 49,5 % of polypropylene and 0,5 % of antimicrobial additive.

The BWPC was processed in the extrusion system Oryzon-OZ-E-EX-L22, with 100 rpm and with four thermal regions controlled with temperature of 165 ^{o}C, 165 ^{o}C, 170 ^{o}C and 175 ^{o}C, respectively. For the microbiological tests six cylindrical samples of BWPC were made with diameter of 0,5 cm and 0,5 cm of height, for each exhibition period. The samples were exposed in atmosphere with controlled temperature of 110 ^{o}C during different periods of time. After exposed in different times the proof bodies were submitted to microbiological tests of the type Diffusion in Agar, using the types of bacteria *Echerichia coli* (*EC*- ATCC 25922) and *Staphylococcus aureos* (*SA*- ATCC 25923)

For evaluations of the effect of the temperature and time of exposition in the structure of the Triclosan and of the composite were applied the techniques of TGA and FT-IR. The Thermal stability and thermal transitions of bactericide additive was observed by thermogravimetric analysis using a Shimadzu TGA-50 from 25 °C to 350 °C (1 °C.min^{-1}) and a IR spectra were obtained on a Perkin-Elmer FTIR model 1 GPC FTIR spectrophotometer for range 400 – 4000 cm^{-1}. For eath time of exposition were made six proof body, with time of exposition between 0 and 720 hours.

Results and Discussion

Microbiological Tests

For evaluation of the bactericide action were utilized a methodology that it applies the bactericide area. The illustration of this method is presented in the Fig. 1. This way, many studies can be accomplished associating the numeric values of this area with the bactericide action and with the interest factors. This study considers the $\mathbf{A_{bac}}$ being the bactericide action of the BWPC composites sample.

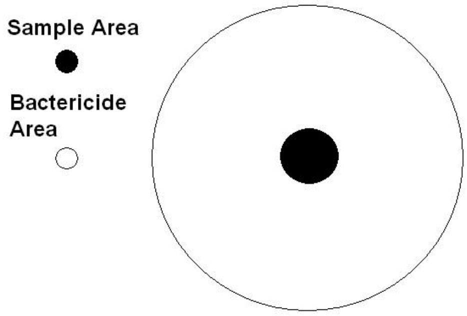

Fig.1 - Illustrative scheme showing of the bactericide area and sample area.

The Fig. 2 presents results of microbiological tests of type Agar Diffusion to samples of the Bactericide Wood Polymer Composites (BWPC) exposed at ambient temperature and samples of the BWPC exposed at 110 ^{o}C during 720 hours.

The results show that the exposition of the BWPC at high temperatures affect the bactericide action for all types of bacteria, being the exposition time a important factor for to define its bactericide action.

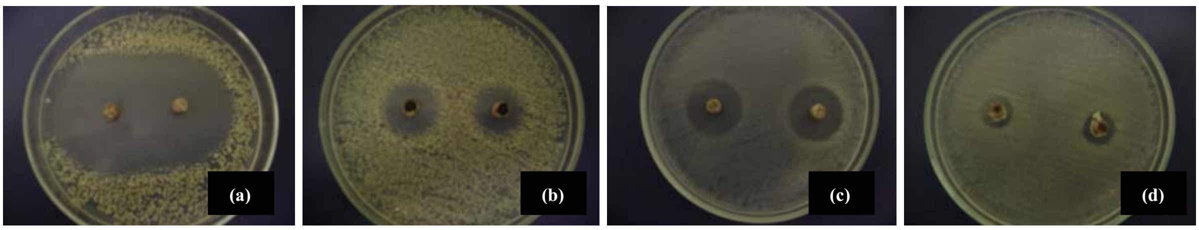

Fig.2 – Agar Diffusion Tests to BWPC. (a) no exposed to high temperature – *SA* bacteria, (b) after exposition at 100 ^{o}C during 720 hours – *SA* bacteria, (c) no exposed to high temperature – *EC* bacteria e (d) after exposition at 100 ^{o}C during 720 hours – *EC* bacteria.

The observed microbiological behavior indicates the possibility of the presence of reactions of the thermal degradation involving the wood elements, the matrix of polymer and the Triclosan additive due to its exposition in the high temperature. The reactions of thermal degradation can affect the bactericide active principle of the Triclosan and decrease the bactericide action of the BWPC.

The Fig. 3 presents the dependence of the bactericide action for the types of bacteria *SA* and *EC* in function of the different times of exposition in temperature of 110 ^{o}C. The results show that the bactericide action decreased about 70% during 100 hours of exposition and after of this time the action is constant. The same effects were observed for the bactericide action of the BWPC for the type of bacteria *EC*. However, the bactericide action to the *EC* was more significant because these microorganisms are less resistant (gram positive) than the bacteria *SA* (gram negative).

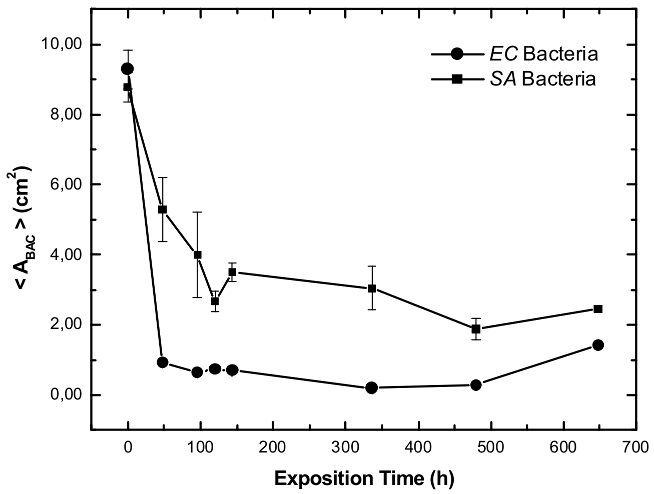

Fig.3 – Effect of the exposition time at 110 °C at the bactericide action of the BWPC to bacteria type SA and EC.

Thermo-analyses results (TGA)

The TGA results indicated that temperatures between 100 °C and 150 °C promote the thermal degradation of the pure Triclosan with small rates, while larger temperatures promote the degradation with great rate. For confirm this result, the pure Triclosan was exposited at 110 °C during 24 hours and was observed its complete degradation.

FT-IR results

The FT-IR technical was applied to evaluate the effect of the temperature in the structure of the BWPC. The FT-IR spectrum for BWPC is very complicated because the presence of many substances in the mixture. The spectrum presents many bands by cellulose, lignin, hemicelulose, polypropylene matrix and Triclosan additive.

The Figures 4a and 4b presents the comparison between BWPC no exposed and BWPC exposed at temperature 100 °C. The results show significant modifications in the BWPC. The increase of the signal around 1738 cm^{-1} (carbonyls) and 1200, 1250 and 1350 cm^{-1} indict the formation of the bonds between C (carbon) and O (oxygen) due to presence of reactions of thermal oxidation in the composites [5, 6]. The spectrums show that the signals in the regions 450 - 580 cm^{-1}, around 1100 cm^{-1} and 3000 - 3500 cm^{-1} decreased after the exposition at 100 °C. These results can be indicting the decrease of the bonds of the type C-Cl and $-CH_2-OH$ presents in the Triclosan structure. The decrease of the signal by vibration states of the bonds C-Cl can be a indicative of the break of this bonds, affecting the bactericide action of the BWPC. The Cl atoms are the bactericide action principle of the Triclosan and after its exposition at high temperature the C-Cl bonds are broken and the atoms of Cl are liberated in the volatile form. The decrease too of the signal correspondent to $-CH_2-OH$ indicts the possible modification of the Triclosan structure with the liberation of the groups of OH. The increase of the signal in the region 800 – 1000 cm^{-1} indict the increase of the bonds of kind ϕ-CO-Cl and Cl-CO-O. These results can be associated with the thermo-oxidation of the Triclosan molecules.

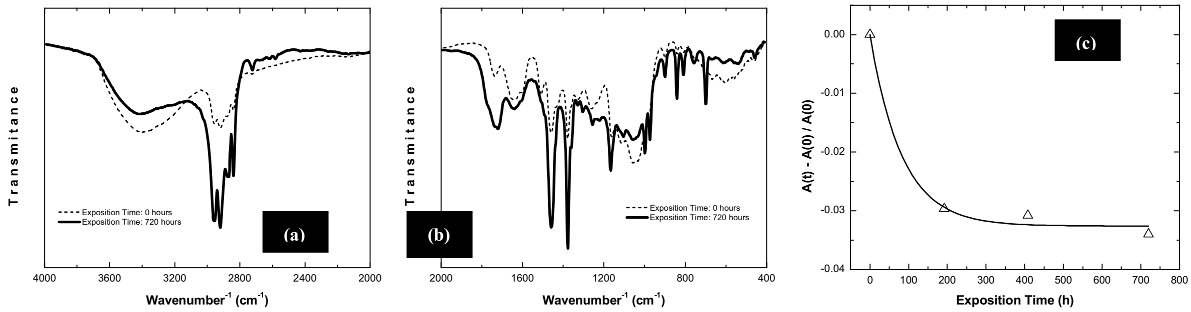

Fig.4 – (a) and (b) FT-IR Spectrum comparison to BWPC exposed at 100 °C during 720 hours and no exposed, respectively, and (c) Comparison between FT-IR spectrums areas (450 – 600 cm^{-1}) to BWPC exposed at 100 °C during different times.

A comparison between FT-IR spectrums (450-600 cm^{-1}) to Bactericide Wood Polymer Composites exposed at 100 °C during different times is showed in the Figure 4c. The results show the dependence of the area of the signal of the FT-IR with the exposition time. The increasing of the exposition time promotes an exponential decrease of the signal until times of 100 h, being practically constant after this time. These results present same behavior of the FT-IR in the region of the Triclosan than the microbiological behavior that presents for the microbiological tests of the BWPC, Figure 3. After 100 hours of the exposition the bactericide action is constant and small because the fraction of the additive that it didn't suffer thermal degradation, protected by the matrix of the polypropylene, it still presents bactericide action.

Conclusions

The results indicated a strong dependence of the bactericide action of the BWPC with the time of the exposition in ambient with relative high temperature. Microbiological results showed that the exposition time in high temperature promote the decreasing of the bactericide action to *EC* (gram positive) and *SA* (gram negative) with an exponential dependence with the time until 100 hours, being constant after this time.

The TGA and FT-IR experiments presented the effect of the high temperature in the chemical structure of the BWPC, Additive and *Pinus*. These results indicted the probable presence of thermal reactions of oxidation of the Triclosan during its exposition.

The results indicated too that the decrease of the bactericide action happens in a significant way to approximately 100 hours of the exposition. The bactericide action of the BWPC after this time is practically constant because probably the Triclosan that the matrix of the polymer protected it wasn't degraded.

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