

Effects of the Temperature and UV Radiation on the Antimicrobial Action of Bactericidal Wood Polymer Composite (BWPC)

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Summary: This paper presents an investigation of the influences of temperature and UV radiation on the antimicrobial properties of Bactericide Wood Polymer Composite (BWPC). The composite was formulated with polypropylene (PP), wood flour from *Pinus elliotis* and a triclosan compound. The BWPC was exposed to different time periods and to UV radiation at 100 °C. Microbiological tests were conducted with *Escherichia coli* and *Staphylococcus aureus* bacteria. These results showed that the bactericide action is strongly affected by the time of exposure of the composite to high temperature and UV radiation.

Keywords: composite degradation and polymer degradation; polymer-matrix composites; recycling; wood; wood fibers

Introduction

WPC (Wood Polymer Composite) material has been the subject of frequent studies over the last two decades with regard to processing,^[1–2] properties, and composition,^[3–6] as well as biodegradability and photo-degradability.^[7–9] This polymeric composite is made of a wood mass in a polymeric resin. The wood residue can be obtained at low cost. This composite is of great interest to the wood transformation industry. WPC is an important material for sectors of the plastic industry and has displayed an average annual growth rate of approximately 18% in North America and 14% in Europe,^[10] with widespread

applications within various sectors of the construction and automobile industries.

An advantage of composites is that they can combine various properties of polymeric additives used during processing. These characteristics permit a large range of applications within the area of engineering composites. By using biocide additives in the polymer, it is possible to create bactericide properties in the WPC. Due to facility of processing, materials with bactericide properties can potentially be applied in the manufacture of products for home use, public restrooms, and laboratory and hospital settings, among others. To incorporate the bactericidal property, polymeric additives containing a triclosan compound have been used at the University of the Extremo Sul Catarinense (UNESC-Brazil) recently. One of the projects evaluated the possibility of incorporating a bactericidal property into WPC by investigating triclosan additives with different magnitudes of action and different temperature effects.^[11] The results showed an excellent microbiological response from the BWPC (Bactericide Wood Polymer

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Composite) produced by an extrusion process.

WPC with bactericide properties (BWPC) could provide affordable applications for wide use in society. In powder form, this bactericide composite can be applied in aviary buildings or in other similar environments as a cheap alternative for the prevention of bacterial diseases in domestic animal care. However, this material might be used in various farm environments operating under different climatic conditions, such as radiation, temperature, or atmospheric conditions. These factors could be responsible for a decrease in composite performance due to reactions between the additive the elements present in the composite or in the atmosphere. When exposed to natural environments, the material surface can reach high temperatures due to light reflection resulting in thermo-degradation of the additives or other composite elements. In this context, exposure time, temperature, and the presence of ultraviolet radiation (UV radiation) can be that will promote to the decreasing of the microbiological action of the BWPC.

This work presents an investigation of the influence of temperature on the antimicrobial properties of Bactericide Wood Polymer Composites (BWPC) after different exposure time periods at 100 °C and under various UV radiation conditions.

Experimental Part

The homopolymer polypropylene (PP – Ipiranga Chemical S.A.) was used as the composite matrix and wood flour was used as the filler element. *Pinus elliotis* wood specimens with a particle size in the range of 28 to 100 mesh were dried for 100 minutes at 110 °C. The polypropylene, dried reinforcement (wood flour) and bactericidal additive were mixed at room temperature in a homogenous system. The added bactericidal agent was a commercial compound containing Triclosan (CIBA Chemical). The Bactericide Wood Polymer Com-

posite (BWPC) was prepared via an extrusion process with a mixture of polypropylene and the reinforcement in proportion of 1:1 % w/w; there was 2.0 wt% of bactericidal additive. The BWPC was processed in the Oryzon-OZ-E-EX-L22 extrusion system, with a screw velocity of 90 rpm, and four thermal regions controlled with temperatures of 160 °C, 160 °C, 175 °C, and 175 °C. For microbiological testing, six extruded cylindrical samples of BWPC were taken with diameters of 0.50 cm and height of 0.50 cm. The samples were submitted to microbiological tests via agar diffusion using the bacteria *Escherichia coli* (EC- ATCC 25922) and *Staphylococcus aureus* (SA- ATCC 25923).

The BWPC samples were exposed to atmospheric conditions with a controlled temperature (100 °C) over different time periods for a total of 650 hours. After the exposition to high temperature under different periods of time, four samples were submitted to microbiological tests using agar diffusion with the bacteria *Escherichia coli* (EC- ATCC 25922) and *Staphylococcus aureus* (SA- ATCC 25923).

The effect of UV radiation exposition on the bactericidal action of the BWPC was studied. The samples were exposed using a UV camera with a 30 Watt power for 15 days. After this, four samples were exposed for different time period and submitted to microbiological tests using agar diffusion with the bacteria *Escherichia coli* (EC- ATCC 25922) and *Staphylococcus aureus* (SA- ATCC 25923).

To evaluate the bactericide action, a method that measures the size of the bactericide area of action was used. An illustration of this method is presented in Figure 1. The bactericide action promotes the death of the bacteria, and as a result, a free region of bacteria colonies surrounds the sample. This region is defined by a *halo* witch is proportional to the number of dead bacteria. In this way, the evaluating of the halo size can be calculated associating the numeric values of this area with the bactericide action and with the factors of interest. This study considers A_{bac} (Bacter-



Figure 1.

Illustration of the bactericide and sample areas.

icidal Area) as a measure of the bactericide action of the BWPC composite sample.^[12]

Results and Discussion

Figure 2 presents results of the agar diffusion tests for the Bactericide Wood Polymer Composites (BWPC) at room temperature. The microbiological tests indicate an excellent bactericide effect of the BWPC produced with the Triclosan compound additive. The bactericide area had a value of 12.57 cm^2 for the bacteria *Escherichia coli* and 23.76 cm^2 for the *Staphylococcus aureus* bacteria. However, with the *Escherichia coli* bacteria, the microbiological halo is smaller because this microorganism has a higher resistance to the bactericide compound than the *Staphylococcus aureus* bacteria. Microbiological studies indicate that *Escherichia coli* is a gram-negative type of bacteria while *Staphylococcus aureus* is gram-positive. In general, gram-negative bacteria contain a cellular wall with higher resistance to

bactericide action than the gram positive bacteria.^[13]

Figure 3 shows microbiological test results for the BWPC samples produced with 2.00 wt% triclosan compound and exposed to high temperature (100°C) over different time periods. The results indicate a strong influence of temperature on the bactericide action of the BWPC. An increase in the exposure time promotes a decrease of the bactericide action of the BWPC for both types of bacteria. These results can be seen as indication of the presence of thermal degradation reactions involving the composite elements and the triclosan molecules in the bactericide additive.

The microbiological tests show bactericide halos with areas of 1.80 cm^2 (Figure 3a) and 12.00 cm^2 (Figure 3b) for the bacteria *Escherichia coli* and bacteria *Staphylococcus aureus* respectively, for BWPC samples exposed previously to a temperature of 100°C over 150 hours.

Figures 3c and 3d show microbiological test results with *Escherichia coli* bacteria

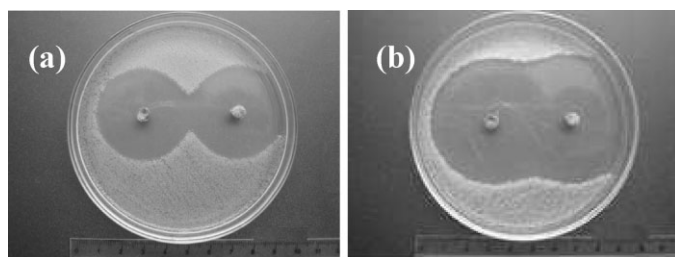


Figure 2.

Agar diffusion tests with BWPC at room temperature: (a) Microbiological results with *Escherichia coli* bacteria and (b) Microbiological results with *Staphylococcus aureus* bacteria.

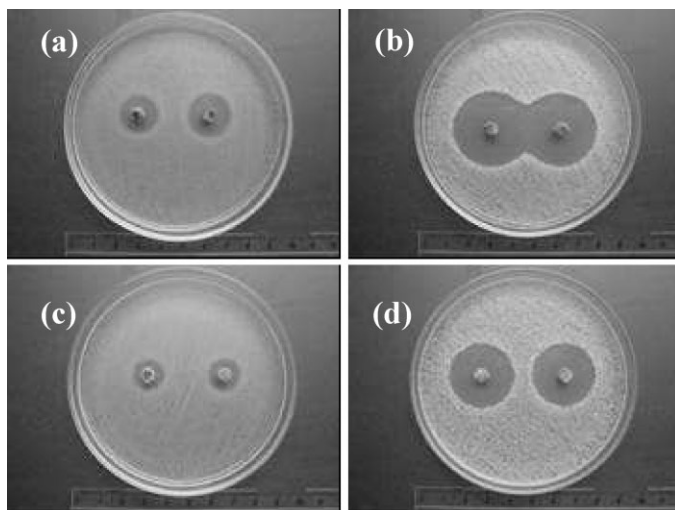


Figure 3.

Agar diffusion tests for BWPC produced with 2.00 wt% triclosan compound exposed to 100 °C. After exposure time of 150 hours: (a) Microbiological test with *Escherichia coli* bacteria and (b) Microbiological test with *Staphylococcus aureus* bacteria. After exposure time of 650 hours: (c) Microbiological test with *Staphylococcus aureus* bacteria and (d) Microbiological test with *Escherichia coli* *Staphylococcus aureus* bacteria.

and *Staphylococcus aureus* bacteria, respectively, for BWPC after exposure at 100 °C over a period of 650 hours. The results show that for an exposure over 200 hours, the magnitude of the bactericide effect of the BWPC is relatively unchanged. Therefore, beyond this time, there are bactericide effects that remain practically constant. For *Escherichia coli* bacteria, the bactericide area of effect after 200 hours of exposure is around 3.50 cm² while for bacteria *Staphylococcus aureus* the bactericide effect is around 1.50 cm².

The comparison of the bactericide action between unexposed BWPC samples and BWPC samples exposed for over 200 hours demonstrates a significant decrease in the bactericide effect. After the critical exposition time (200 hours), the magnitude of bactericide action decreases about 85% for *Staphylococcus aureus* bacteria and 89% for *Escherichia coli* bacteria. These results indicate a significant effect of temperature on the bactericide action of the BWPC.

The presence of lower microbiological effects after an exposure time of 200 hours

indicates the possibility that the polymeric matrix initially protects the triclosan molecules from thermal-degradation reactions. However, the significant decrease in the magnitude of the bactericide action for samples exposed to high temperature indicates the probable presence of thermal reactions in the composite. These thermal reactions can alter the bactericide effect of the triclosan additive by degrading the triclosan molecules that are unprotected by the polymeric matrix.

Figure 4 presents thermo-gravimetric analysis results (TGA) for the pure Triclosan molecule. The TGA results indicate that temperatures between 100 °C and 150 °C promote a low rate of thermal degradation of pure Triclosan, while high temperatures promote a high degradation rate. The TGA result indicates if pure triclosan compound was submitted to temperatures over 100 °C during its processing the bactericidal additive will be degraded and its bactericidal properties probability prejudiced.

The microbiological tests presented in the Figure 3 shows the presence of the

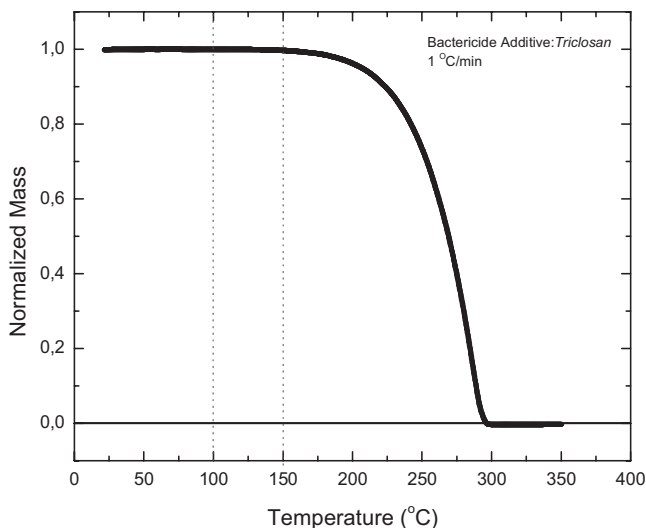


Figure 4.

Thermal Gravimetric Analyses (TGA) for the bactericide additive Triclosan.

bactericidal action in the BWPC after its processing at temperatures between 160 °C and 175 °C. These results suggest that the Triclosan molecules protected by the polymeric matrix are not subject to significant thermal degradation, while the unprotected molecules are degraded by high temperature conditions. Therefore, due to degradation of those unprotected Triclosan molecules, the bactericide action of the BWPC is

altered after significant exposure time at high temperature.

Figure 5 shows the dependence of the bactericide effect of the BWPC on the exposure time at 100 °C. The microbiological results indicate an exponential dependence. An increase in the exposure time promotes an exponential decrease of the magnitude of the bactericide effect, as shown in the figure. This behavior can be

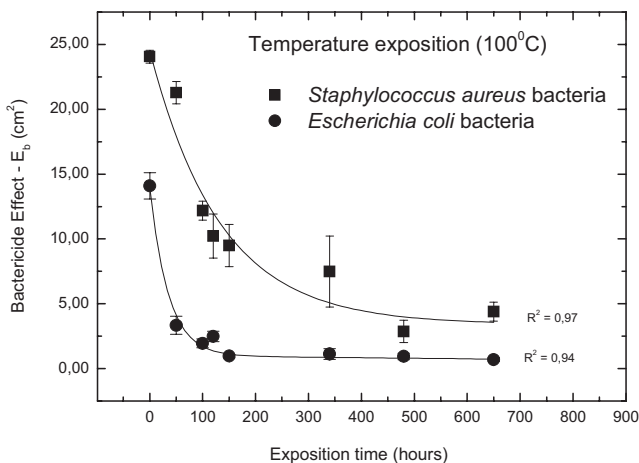


Figure 5.

Dependence of the bactericide effect of the BWPC with 2.0 wt% of triclosan compound on the exposure time at 100 °C.

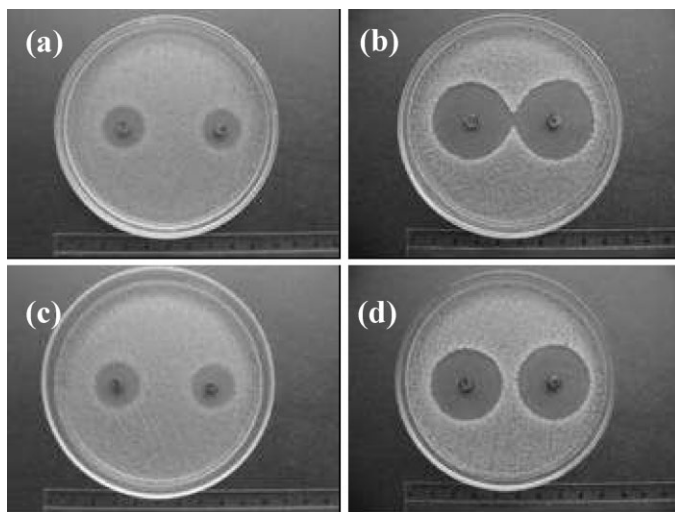


Figure 6.

Agar diffusion tests for BWPC produced with 2.00 wt% the triclosan compound, exposed to UV radiation. Exposure time of 120 hours: (a) Microbiological test with *Escherichia coli* bacteria and (b) with *Staphylococcus aureus* bacteria. Exposure time of 350 hours: (c) Microbiological test with *Escherichia coli* bacteria and (d) with *Staphylococcus aureus* bacteria.

associated with the degradation mechanisms activated in the composite via the effect of temperature on the triclosan molecules, thus affecting the bactericide action associated with these molecules.

Figure 6 shows microbiological test results for BWPC samples produced with 2.00 wt% triclosan compound and exposed to ultraviolet radiation over different time periods. The results indicate an influence of UV on the bactericide action of the BWPC. An increase in the time exposure promotes a decrease in the bactericide effect for both types of bacteria. These results can be an indication of the presence of UV degradation reactions involving the triclosan molecules in the composite.

The microbiological results show bactericide halos with areas of 3.40 cm² and 10.20 cm² for BWPC samples exposed to UV radiation over five days (120 hours) with the *Escherichia coli* bacteria and *Staphylococcus aureus* bacteria, respectively, as shown in Figures 6a and 6b. Comparison of the microbiological halos for the exposed and unexposed samples indicates significant differences in the

bactericide action of the BWPC. Exposure to UV during this period caused a decrease in bactericide magnitude of 73% and 57% for *Escherichia coli* bacteria and *Staphylococcus aureus* bacteria, respectively. These results indicate the possibility of occurring UV-induced degradation reactions involving the triclosan additive and affecting the bactericide action of the BWPC.

Figures 6c and 6d show microbiological results for BWPC exposed to UV over a period of fifteen days (360 hours) with *Escherichia coli* bacteria and *Staphylococcus aureus* bacteria, respectively. The results show that for an exposure time above 120 hours, the magnitude of the BWPC bactericide effect does not show significant changes, but presents a low bactericide action. The presence of microbiological effects after long UV exposure time indicates the possibility that the polymeric matrix may protect the triclosan molecules from degradation. This condition may permit UV degradation reactions with a lower degradation rate.

The microbiological results indicate an exponential dependence of the bactericide

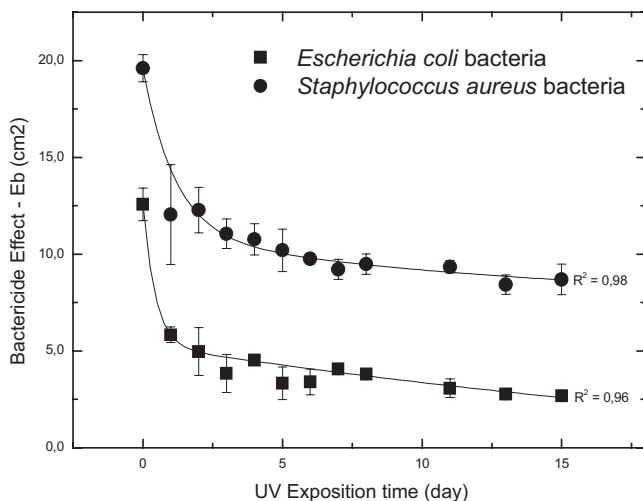


Figure 7.

Dependence of the BWPC bactericide effect on the UV exposure time periods. BWPC produced with 2.00 wt% triclosan compound.

effect on the composite relative to UV exposure time. An increase in exposure time promotes an exponential decrease in the magnitude of the bactericide effect, as seen in Figure 7. This behavior can be associated with the degradation mechanisms in the composite caused by the interaction between the triclosan molecules and the UV-radiation, affecting the bactericide action associated with these molecules.

The dependence of the BWPC bactericide effect on UV radiation and temperature is similar in both cases. An increase in exposure time promotes a decrease in the magnitude of the bactericide effect. However, for the same exposure time, the temperature causes a greater decrease in the bactericide effect.

Research by Yu et al. and Milagros et al. shows the presence of triclosan photo-degradation reactions induced by UV radiation. Products of these reactions with triclosan are quinone and triclosan hydroquinone^[14] and dibenzodichloro-*p*-dioxin.^[15] These products exhibit a lower bactericide action than the original triclosan molecules and thus affect the bactericide properties of the BWPC. Exposure to high temperature and UV radiation can

induce these reactions, accelerating the degradation process of the triclosan molecules and decreasing the bactericide action of the BWPC. However, the triclosan molecules protected from the degradation reactions by the polymeric matrix, resulting in the retention of a low but significant bactericide effect.

Conclusion

The results indicate a strong dependence of the BWPC bactericide action on exposure time to an environment with relatively high temperature and the presence of UV radiation. The microbiological results showed that exposure time at high temperature and UV radiation both promote a decrease in the bactericide action with an exponential relationship that approaches a constant after 150 hours of exposure.

These observations can be associated with thermal reactions and photo reactions involving the triclosan molecules that are induced by high temperature and UV radiation. However, the triclosan molecules protected by the polymeric matrix retain a low but significant bactericide effect on the BWPC. This is a strong indication that the

environmental conditions are an important factor for analyses prior to the application of BWPC's.

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- [1] P. W. Balasurya, L. Ye, Y. W. Mai, *Composites Part A*. **2001**, 32, 619–629.
- [2] D. Bhattacharyya, M. Bowis, K. Jayaraman, *Composites Science and Technology*. **2003**, 63, 353–365.
- [3] E. Byskov, J. Christoffersen, C. D. Christensen, J. S. Poulsen, *International Journal of Solids and Structures*. **2002**, 39, 3649–3673.
- [4] F. M. B. Coutinho, T. H. S. Costa, *Polymer Testing*. **1999**, 18, 581–587.
- [5] S. B. Elvy, G. R. Dennis, L. T. Ng, *Journal of Materials Processing Technology*. **1995**, 48, 365–372.
- [6] M. N. Ichazo, C. Albano, J. Gonzáles, R. Perera, M. V. Candal, *Composites Structures*. **2001**, 54, 207–214.
- [7] C. Tascioglu, B. Goodell, R. Lopez-Anido, M. Peterson, W. Halteman, J. Jellison, *International Biodeterioration & Biodegradation*. **2003**, 51, 157–165.
- [8] B. Geoge, E. Suttie, A. Merlin, X. Deglise, *Polymer Degradation and Stability*. **2005**, 88, 268–274.
- [9] M. M. Laurent, P. K. Donatien, J. Zhang, *Journal of Applied Polymer Science*. **2001**, 80, 1943–1950.
- [10] B. S. Gupta, I. Reiniati, M. P. G. Laborie, *Colloids and Surfaces A: Physicochemical Engineering Aspects*. **2007**, 302(1–3), 388–395.
- [11] M. A. Fiori, M. M. S. Paula, E. Angioletto, M. F. Santos, H. G. Riella, M. G. N. Quadri, *Materials Science Forum*. **2008**, vols. 591–593, 362–367.
- [12] M. A. Fiori, M. M. S. Paula, A. M. Bernardin, H. G. Riella, E. Angioletto, *Materials Science and Engineering C*. **2009**, 29, 1569–1573.
- [13] S. P. Denyer, J. Y. Maillard, *Journal of Applied Microbiology*. **2002**, 92, 35–45.
- [14] M. Milagros, M. J. Gómez, I. Ferrer, A. Agüera, M. D. Hernando, A. R. F. Alba, *Analytica Chimica Acta*. **2004**, 524, 241–247.
- [15] J. C. Yu, T. Y. Kwong, Q. Luo, Cai. Zongwei, *Chemosphere*. **2006**, 65, 390–399.