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Sterilization in Hydroponic Recycling System Using Visible Light–reactive Titanium Dioxide Photocatalysts

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This research was aimed at developing a hydroponic recycling system using a visible light– reactive titanium dioxide (TiO_2) photocatalyst. We made a sterilization system for the nutrient solution by utilizing the features of the filters coated with visible light–reactive titanium dioxide photocatalyst. The system is composed of a photocatalyst filter (300×300×20 mm), a 4–tier stair type processing channel (305 mm wide, 2,100 mm long, 30 mm high), a supply tank, a catchment tank, a water pump, a circulation control facility, an insolation sensor, and a data log. We also evaluated the sterilization performance using a number of filters, the initial density of spores in the nutrient solution, the species of spores, flow rates, and the amount of insolation. In the experiment with 20 filters, sterilization performance per 1 filter was 20.3% higher than one per 5 filters. Sterilization performance at a low initial density of spores (26 counts/0.9 mm^3) is 23.9% higher than at a high initial density of spores (440.3 counts/0.9 mm^3). *Colletotrichum* was more sensitive to the photocatalyst than *Fusarium*, and the slow flow rate (0.5 L/min) has more sterilization effects (9.0%) than the fast flow rate (1.0 L/min). The logarithmic relation of the average insolation and lethal intensity of unit spore density is correlated as 0.926 of the coefficient of determination (R^2). These results provide valuable information for TiO_2 photocatalytic treatment of waste nutrient solutions under solar light irradiation.

Key words: photocatalyst, titanium dioxide, visible light–reactive, nutrient solution, sterilization

INTRODUCTION

In recent years, researchers have been conducting experiments on a hydroponic system for sterilization and purification of nutrient solutions. The methods of supplying nutrient solutions in a hydroponic recycling system can be classified into two types: circulating and non–circulating systems. It has been discovered that in a non–circulating hydroponic system, 15 to 40 percent of the nutrient solution is drained out of the system, which may cause environmental pollution, such as eutrophication, and a waste of resources (Benoit, 1992; Sonneveld, 1993). Therefore, development of a hydroponic system able to recycle nutrient solutions is needed to address cost and environmental issues. Hydroponic systems produce pathogens, such as *Colletotrichum, Phytopthora*, and *Fusarium*. In the circulating hydroponic system, the nutrient solution is re–circulated throughout the whole cultivation bed, but once invaded by harmful germs such as Pythium or *Fusarium*; it can cause fatal damage to the crops. Therefore, it is crucial to improve the sterilization of nutrient solutions in a hydroponic system.

In general, killed bacteria can be done through a physical method (UV rays, filtering, or photocatalyst) or a chemical method (ozone or mineral metallic ions). Among these methods, sterilization by photocatalyst has the strongest oxidizing power and can even be activated with solar energy (Lee, 2007). Photocatalyst using titanium dioxide can cause photo–organic disintegration to destroy or cause the malfunction of cell membranes in bacterial cells (Miyama *et al*., 2002). Most photocatalyst treatments were conducted with UV irradiation (Lee 1999, Miyama *et al*., 2002, 2009), which has a shorter wavelength than visible light, and thus has higher quantum energy. In general, UV irradiation is more effective than visible light in the sterilization process.

Few studies are available on visible light–reactive photocatalysts using natural sunlight. Chung *et al*. (2010) investigated the visible light–reactive photocatalyst for recycling a nutrient solution in a hydroponic system, and found that the shape of the treatment channel was effective in reducing the waste of the nutrient solution and a filter material (ceramic) for better performance.

The objectives of this study were to develop a sterilization system using the visible light–reactive titanium dioxide photocatalyst and to evaluate the system through experiments.

MATERIALS & METHODS

System Design

The configuration of the sterilization system using visible light–reactive titanium dioxide is illustrated in Figure 1; drainage of the non–circulating solution is sterilized and reused in this system. Since a separate line is installed, the treated drainage is not mixed with the contents of the supply tank. Hashimoto *et al*. (2005) and Miyama (2009) used UV–light–reactive–photocatalyst ceramic filters, and set the entrance and exit to the sup-

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ply tank diagonally; however, nutrient solutions cannot flow equally throughout the filters. In this research, we installed a treatment channel/tank, resulting in the nutrient solution flowing freely.

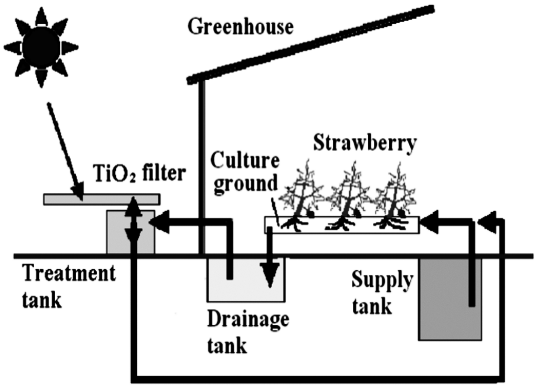

Fig. 1. Basic configuration of a hydroponic culture system for sterilization and purification by titanium dioxide photocatalyst.

System Organization

Figure 2 shows our sterilization system for nutrient solution using a visible light–reactive photocatalyst filter.

Four treatment channels (305 mm wide, 2,100 mm long, 30 mm high) were installed at different levels, and 5 photocatalyst filters were also installed on each channel. At the bottom of the treatment system, there were 200 L – capacity supply and collection tanks. The tank size, 200 liters, was chosen for an actual strawberry hydroponic cultivating system. Inside of each tank, a supply pump and water gauge are installed at the bottom of the tank so that residual suspension could be minimized. If a water gauge for the residual quantity indicated a level below 10 mm, the recycle treatment controller is activated, and tanks 1 and 2 reverse their roles of supply and drainage. In this way, nutrient solutions can be re–circulated and recycled.

A valve was installed at an underwater pump inside a supply tank to control the water flow, which was maintained by a 2 mm thick photocatalyst filter. Our model enables a 1000 m^2 non–circulating hydroponic system to treat 20–30% of the drainage and recycle it as well. Our preliminary experiment shows that 5 photocatalyst filters in 3 tiers (15 total filters) is less efficient than 15 filters in one tier. To compensate for this limitation, another treatment channel having5 photocatalyst filters was installed.

Control Algorithms for Circulation Treatment

In sterilizing the waste nutrient solution, treatment is not effective when the amount of insolation is low (cloudy or rainy days). If this is the case, the nutrient solution collected from primary treatment should be circulated and re–treated. Hashimoto *et al.* (2005) sent collected nutrient solutions from a supply tank to a treatment tank using an underwater pump, and then flowed the nutrient solution to the top of the treatment channel. However, in our system, the nutrient solution draining from a treatment tank was headed for a supply or drainage line controlled by two electronic valves. This circulating process is illustrated in Figure 3.

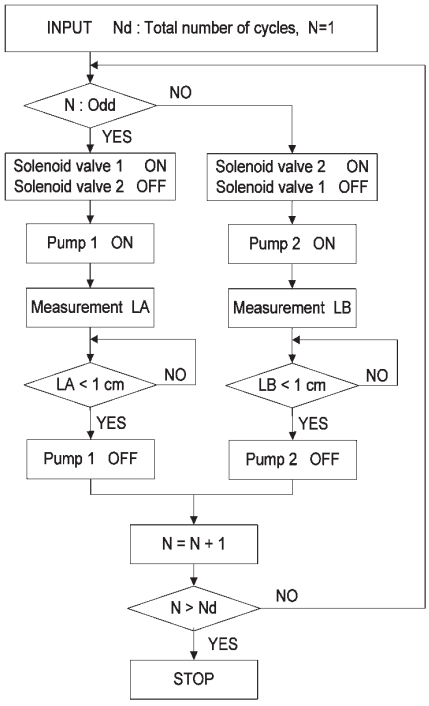

Fig. 3. Flowchart of the circulating process of nutrient solution.

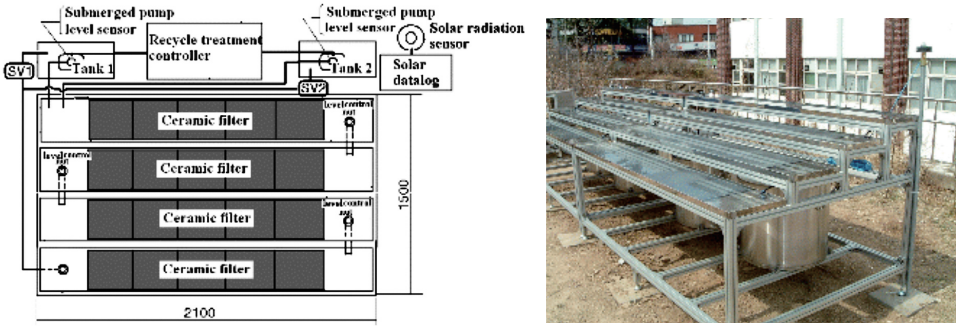

Fig. 2. The model of a sterilization system of nutrient solution using visible light–reactive type titanium dioxide photocatalyst.

Methods

A suspension of *Colletotrichum* spores was prepared with tap water and measured cell concentration, DOS (Density of Spores). It flowed to the top of the treatment channel through the underwater pump at 1 L/min, and maintained at a 2 mm water level for sterilization purposes. Suspension samples were collected from each treating channel's bottom valve, and spore density was measured. Then, the collected suspension was repeatedly circulated throughout the treatment channels to investigate the DOS changes. All measurements were replicated three times. In each experiment, samples were collected 10 times, and the spore population was counted using an optical microscope.

Our sterilization system was installed under sunlight to examine the performance with actual cultivation conditions. The average amount of insolation observed 410–920 W/m^2/day.

The variables that influence the sterilization performance are: the number of filters, the initial density of the spores in the nutrient solution, the species of spores, flow rates, and the amount of insolation. The visible light–reactive ceramic filters were equipped with 1, 2, and 5 sheets each; sterilization performance was measured depending on the number of filters. To trace the varied sterilization performances in indoor (with a vinyl cover) and outdoor environments (without a vinyl cover) 1 and 2 filters were applied. Also, sterilization of our treatment system (5 filters × 4 tiers = 20 total filters) was examined based on spore population and flow rate. The species of spores used in the experiment were *Colletotrichum* and *Fusarium*, and the flow rates were 0.5 L/min and 1 L/min.

DOS (Density of Spores), an index of sterilization performance, was statistically analyzed using SAS V9.1 (SAS Institute Inc., USA). Differences between variables were tested for significance with a one–way ANOVA. The dependent variable is the DOS rate, and the independent variables are the number of filters, initial density of the spores in the nutrient solution, the species of spores, flow rates, and the amount of insolation. SigmaPlot V8 (Systat Software, USA) was used to model sterilization performance by insolation, which will be useful for our system's practical applications.

RESULTS

Sterilization by the number of filters

Figure 4 shows DOS (Density of Spores) in cumulative number of treating filter sheets for filters: 1, 2, and 5. As shown in the figure, three different methods of filtering are examined. 1 filter × 20 treatments, 2 filters × 10 treatments, and 5 filters × 4 levels treatment in succession. A cumulative treating filter number is defined as the product of a filter number and treatments, e.g. 20 cumulative treating filter numbers = 1 filter × 20 treatments. As shown in figure, reduction of spore density at 1 filter is far better than the other two cases (2 and 5 filters); final spore densities are 6.2%, 11.8%, and 26.5% at each case.

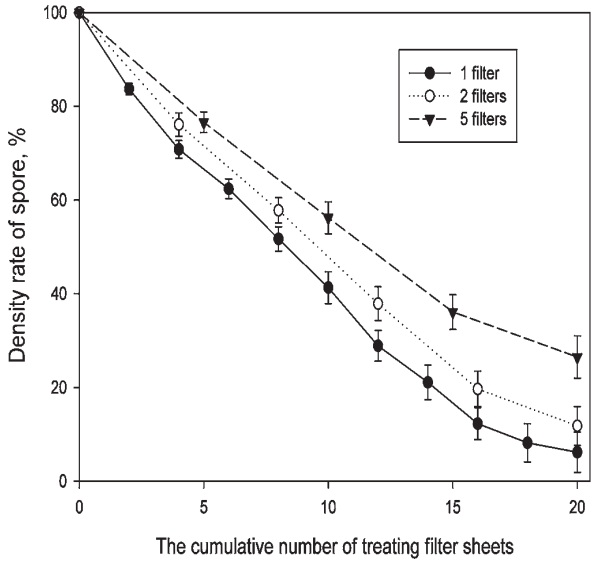

Fig. 4. Changes in the density of spores to cumulative number of treating filter sheets for treating filters ($P<0.05$).

Thus, when the cumulative number of filters is identical, 1 filter with repetitive treatments is more efficient than successive treatments with filters. However, in a practical application, one should consider other system performances as well.

Sterilization by initial Density of Spores (DOS)

In Figure 5, sterilization performance depending on initial DOS at 5 filter sheets is shown. The density rate of spores was defined as the survival rate to the initial population of spores. Density rates of spores after 20 treatments were 7.7% at low initial DOS (26/0.9 mm^3), 13.6% at middle initial DOS (91.2/0.9 mm^3), and 31.6% at high initial DOS (139.1/0.9 mm^3).

Thus, the lower the initial DOS is, the better the sterilization performance is. Sterilization performance is decreased when the initial DOS is higher because it takes

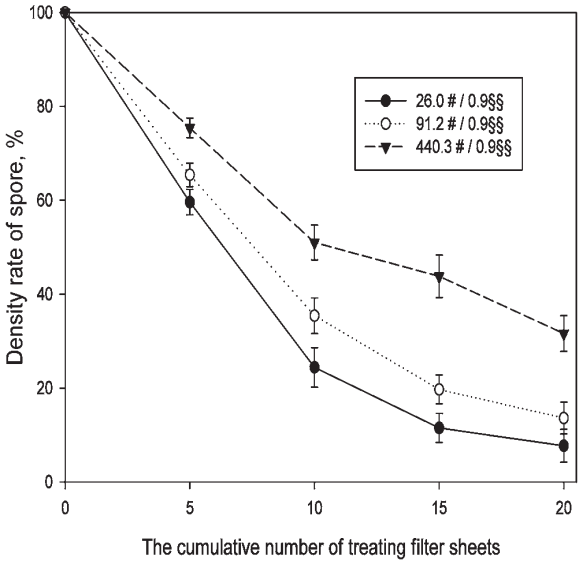

Fig. 5. Changes in the density of spores to cumulative number of treating filter sheets for the concentration level of spores ($P<0.05$).

more time for the decomposition of organic compounds by photocatalyst.

Sterilization by species of spores

Figure 6 shows the changes in DOS for the two different spore species, *Fusarium* and *Colletotrichum*. Density rates of spores after 20 treatments were 22.8% for *Fusarium* and 18.6% for *Colletotrichum* respectively, which means *Colletotrichum* is more sensitive to photocatalyst filters than *Fusarium*. Since *Fusarium*'s spore is bigger than *Colletotrichum*, breakdown of the former could require more treatments than the latter. It turns out that sterilization performance by photocatalyst depends on the size and weight of the spores.

Sterilization by flow rate

Figure 7 presents DOS to the cumulative number of treating filter sheets for the different flow rates of nutrient solutions. As the flow rates increased twice from 0.5 L/min to 1.0 L/min, DOS rates after 20 treatments increased from 11.6% to 20.6% accordingly, which means a low flow rate is better for sterilization. However, sterilization by flow rate is influenced by other factors such as the initial DOS, the size of the spores, etc.

Sterilization by vinyl covering

Figures 8 and 9 illustrates DOS with and without a vinyl cover at one and two filters respectively. In Figure 8, with one filter, the final DOS with and without a vinyl

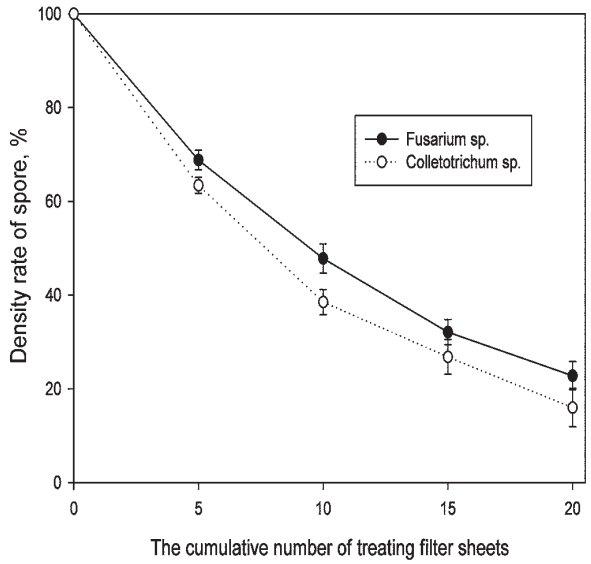

Fig. 6. Changes in the density of spores to cumulative number of treating filter sheets for the different spore species (P<0.05).

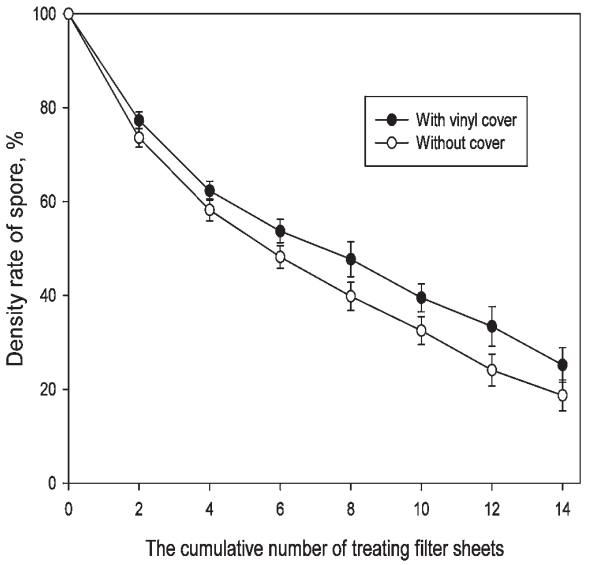

Fig. 8. Changes in the density of spores with and without a vinyl cover using one filter (P<0.05).

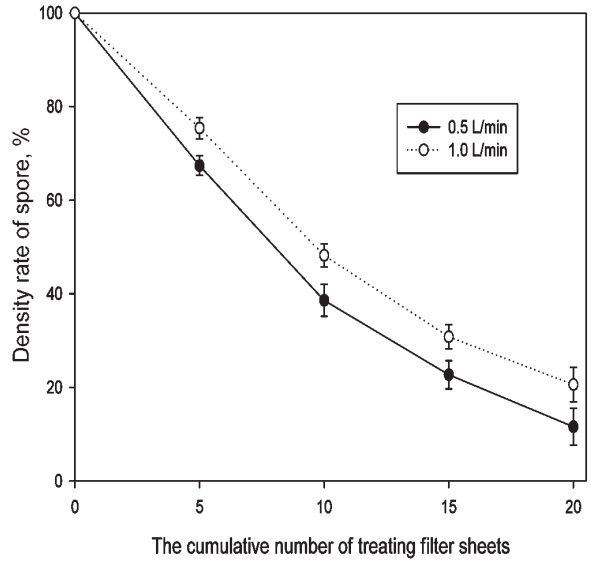

Fig. 7. Changes in the density of spores to cumulative number of treating filter sheets for the different flow rates of nutrient solutions (P<0.05).

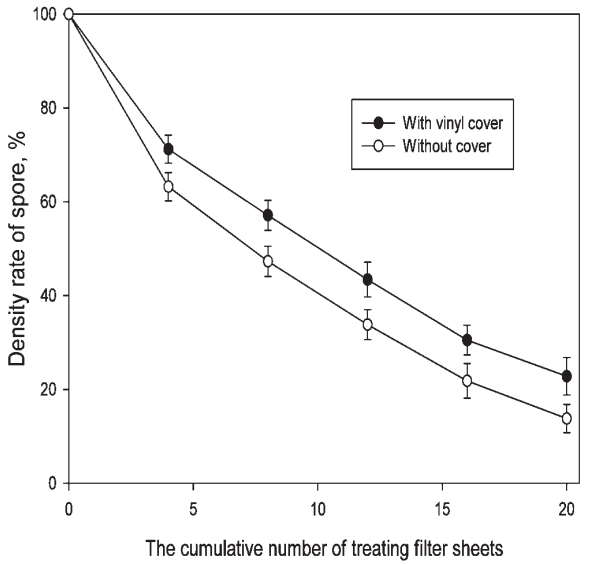

Fig. 9. Changes in the density of spores with and without a vinyl cover using two filters (P<0.05).

cover were 25.2% and 18.7%, respectively. That is, sterilization performance with a vinyl cover declined 6.5% compared to one without a vinyl cover. When installing a system in a greenhouse, therefore, we should consider the decrease in the sterilization performance.

In Figure 9, two filters were used in the system with and without a vinyl cover. The final DOS rate was 22.8% with a vinyl cover and 13.8% without a vinyl cover. This result shows that sterilization decreases 9.0% when the filer is covered by vinyl. These results indicate that sterilization is better when a sterilization system is installed without a vinyl cover (directly under the solar light).

Modeling Sterilization by the amount of insolation

Figure 10 shows the relationship between average insolation and the lethal intensity of unit spore density on a one–day basis with *Colletotrichum*. Lethal intensity of unit spore densityis obtained by dividing the value of the initial DOS by the decreased percentage of spore density per one cumulative treating filter sheet, then multiplying the result by 1000. The limits of insolation (320 W/m^2) in the figure indicates the limits of utilization if a system is installed in a greenhouse.

Average insolation and the lethal intensity of unit spore density is linear at semi–log gradations; the amount of insolation is proportional to lethal intensity. Regression analysis (eq (1)) indicated that the fitted model accounted for more than 90% of the variations in the experiment; R^2 is 0.926. In actual hydroponics, spore density in the nutrient solution is much less than the ones used in the current experiment, which should be considered for practical use.

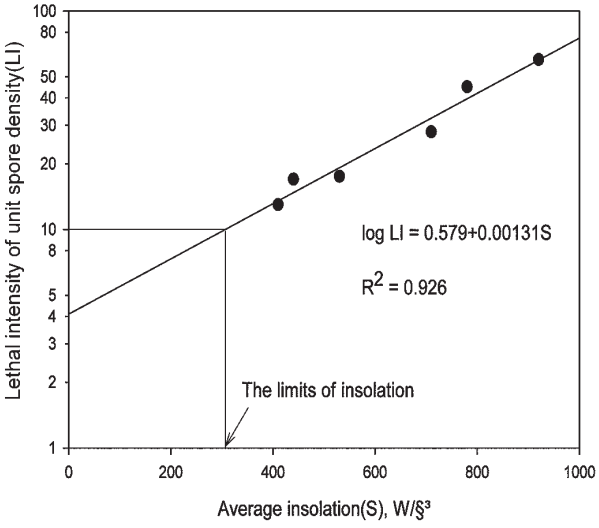

Fig. 10. Logarithmic relation of insolation and lethal intensity of unit spore density.
$\text{LogLI} = 0.579+1.31\times10^{-3}\text{S}$ (1)
Where, LI : Lethal intensity of unit spore density
S : Average insolution (W/m^2)

CONCLUSION

This study aimed to develop a hydroponic recycling system using a visible light–reactive titanium dioxide photocatalyst. We made a sterilization system for a nutrient solution by utilizing filters coated with a visible light–reactive titanium dioxide photocatalyst, and then measured the sterilization performance depending on variables such as the number of filters, the initial density of the spores in the nutrient solution, the species of the spores, flow rates, and the amount of insolation. The results are:

(1) A developed model consists of a visible light–reactive photocatalyst filter (300×300×20 mm), four–tiers of processing channels (305 mm wide, 2,100 mm long, 30 mm high), a supply tank, a catch tank, an under water pump, a control device for the circulating process, an insolation sensor, and a data logger. In addition, a surface adjusting nut was installed to regulate the water level at 2 mm.

(2) With 20 filters, the sterilization performance per 1 filter shows 5.6 counts/0.9 mm^3 of the final spore density. The result indicates a 20.3% higher sterilization performance than one per 5 filters.

(3) The higher the initial DOS, the less the sterilization performance; after 20 treatments, the final DOS rates are 7.7% at low initial DOS (26.0 counts/0.9 mm^3) and 31.6% at high initial DOS (139.1 counts/0.9 mm^3).

(4) *Colletotrichum* was more sensitive to photocatalyst than *Fusarium*, approximately 4.2% lower at final DOS.

(5) The slow flow rate (0.5 L/min) had more sterilization effects (9.0%) than the fast flow rate (1.0 L/min).

(6) In sterilization, depending on an indoor–outdoor operation (with or without a vinyl covering), outdoor treatment had a 6.5% higher sterilization than inside treatment.

(7) The logarithmic relation of the average insolation and the lethal intensity of unit spore density is correlated as 0.926 of the coefficient of determination (R^2).

POSTSCRIPT

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