



## Preservative Equipment Based on Nitrogen Doping Titanium Dioxide Photocatalyst White Oyster Mushroom In Banyumeneng

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### Abstract

Oyster mushrooms easily wilt and turn brown because of bacteria and enzymatic reactions. Preservation of oyster mushrooms has been done in the freezer, which can only last 3-4 days. In addition, preservation using chemicals can cause side effects such as diarrhea to long-term cancer. The purpose of this study was to determine the shelf life of white oyster mushroom products using the innovation of making preservatives with titanium dioxide doped nitrogen (N-TiO<sub>2</sub>). The method used is the sol-gel method with TiCl<sub>4</sub> as the precursor of TiO<sub>2</sub> and diethylamine as the nitrogen-doped precursor. N-Doped TiO<sub>2</sub> was characterized using FTIR, XRD, and antibacterial activity tests. The results of the FTIR spectrometer showed peaks at 518 and 678 cm<sup>-1</sup>, which indicated the presence of vibrations from TiO<sub>2</sub>. Analysis of the crystalline phase of TiO<sub>2</sub> using XRD obtained a mixed phase in the form of anatase with peaks at 55.10 (2 $\theta$ ) and rutile at 27.40 (2 $\theta$ ). Anti-bacterial activity test showed that N-TiO<sub>2</sub> was able to inhibit the growth of *E. coli* bacteria by 6.48 $\pm$ 0.42 mm. The results of the organoleptic test showed that preservation using photocatalyst can extend the preservation time of oyster mushrooms, which is about 6-9 days, longer than the ideal preservation time of oyster mushrooms. This technological innovation can answer the problems of oyster mushroom farmers in Banyumeneng Village related to oyster mushroom harvests that are wasted because they are rotten and cannot be sold

### Introduction

The agricultural sector is a field that is one of the livelihoods of the Indonesian people, especially oyster mushroom farmers in Banyumeneng Village. White oyster mushroom (*Pleurotus ostreatus*) is a good source of nutrition, this is because white oyster mushroom (*Pleurotus ostreatus*) contains 35% protein, 9 types of amino acids, 2.20% fat consisting of 72% unsaturated fatty acids, and carbohydrates in every 100 g (Diviyasri *et al.*, 2021). White oyster mushroom (*Pleurotus ostreatus*) is not only known as a delicious food mushroom and has high nutrition, but is also known as a nutraceutical ingredient because

it demonstrates antimicrobial and antioxidant properties like palm sugar (Egra *et al.*, 2019; Winarni *et al.*, 2018). After surveying with Oyster Mushroom farmers in Banyumeneng Village, a preservation problem exists. White oyster mushrooms are a type of food that is easily damaged at room temperature, and if not packaged properly, they can only last 24 hours. Several studies have been carried out to maintain the freshness of mushrooms white oysters include storage at low temperatures (Li *et al.*, 2016). During the harvest season, many farmers suffer losses because they cannot store mushrooms for more than 3 days. Damage to the fruiting body of the fungus occurs due to

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the withering process, and the colour changes to brown due to an enzymatic reaction because it reacts with oxygen. Prevention can only be done by post-harvest handling, which is through preservation technology (Sugianto & Sholihah, 2021).

One of the existing preservation technologies uses titanium dioxide ( $\text{TiO}_2$ ). Titanium dioxide is an economically valuable photocatalyst substance (Daneshvar *et al.*, 2007). The working principle of the  $\text{TiO}_2$  photocatalyst is that when nano-sized zinc oxide is exposed to UV light, it will form superoxide compounds that can eliminate harmful microbes and bacteria (Georgekutty *et al.*, 2008).  $\text{TiO}_2$  nanoparticles have high antibacterial activity, so when  $\text{TiO}_2$  is coated on other substances such as glass, it can kill microbes and bacteria in the surrounding environment when exposed to UV light. Because zinc oxide is a catalyst, it will never deplete and will continue to participate in the reaction.  $\text{TiO}_2$  has a large surface area and charge transfer due to the easy enhancement of photon induction. The high surface area helps effective exposure to light and facilitates photochemical reactions on the surface, while the ease of charge transfer due to photon induction helps the photon-induced electron capture and donation process. These properties are also expected to provide benefits to nitrogen-doped  $\text{TiO}_2$  systems, so to take advantage of the superior properties of nano-sized materials, it is necessary to develop nitrogen-doped  $\text{TiO}_2$  mesoporous nanoparticles (Kay & Grätzel 1996). Titanium oxide ( $\text{TiO}_2$ ) has a bandgap of 3.2 eV. This is one of the problems in the application of the  $\text{TiO}_2$ -based solar spectrum because only 4 - 5% of sunlight is emitted in the UV region, so various efforts have been made to improve the response of  $\text{TiO}_2$  to visible light (Kaur & Gupta 2016). In the other situation, this is an advantage and a weakness, because titanium dioxide can act as a photocatalyst material, but is initiated by UV light (Georgekutty *et al.*, 2008). This weakness can be overcome by modifying the material using doping (Georgekutty *et al.*, 2008; Sankara Reddy *et al.*, 2013). Various studies have been conducted on the use of metal doping, ranging from alkali, alkaline earth, transition, and lanthanide elements, including metalloids

such as Sb. Most of the doping does not show an increase in photo activity compared to pure  $\text{TiO}_2$ , which can be characterized that doping innovation is needed in the development of  $\text{TiO}_2$  by using nitrogen. Nitrogen was found to be the most effective dopant because its size is not much different from that of oxygen, and its small ionization energy, which accelerates the reaction between  $\text{TiO}_2$  and nitrogen (Sankara Reddy *et al.*, 2013; Yang *et al.*, 2021). With the above problems, the purpose of this study was to determine the shelf life of white oyster mushroom products using the innovation of making preservatives with  $\text{TiO}_2$  and nitrogen doping as a preservative and antibacterial solution in white oyster mushrooms.

## Method

The time it takes for the creation of this tool and research is five months. Activities are carried out in three places, that is, the Chemical Engineering Laboratory of Universitas Diponegoro and the Terpadu Laboratory of Universitas Diponegoro. After the mushrooms are finished harvesting, fresh mushrooms are selected and separated from mushrooms that are rotten or damp. Then, clean the roots of the mushrooms. After that, the mushrooms can be stored in our innovative storage device with a longer shelf life. Preparation of titanium dioxide ( $\text{TiO}_2$ ) anatase phase was carried out by the sol-gel method with  $\text{TiCl}_4$  as a precursor. The beaker containing titanium chloride ( $\text{TiCl}_4$ ) was added  $(\text{NH}_4)_2\text{SO}_4$  0.5 M. Then reacted with  $\text{NH}_4\text{OH}$  at 70 °C and stirred at 300 rpm. The reaction product is in the form of a gel, and then washed with water to remove chlorine ions. Furthermore, the gel was dispersed into an ethanol solution to remove water and dried at 60 °C for 48 hrs to become powder.

N- $\text{TiO}_2$  was made based on modified Saragih's (2011) research. Next, mixing 5 g of  $\text{TiO}_2$  with 270 mL of absolute ethanol occurred, and researchers proceeded with the process of sonication for 10 minutes. Then, researchers mixed 10 mL of absolute ethanol, 10 mL of distilled water, and 2 M HCl to achieve pH 2. Next, researchers stirred the mixture with a magnetic stirrer for 30 minutes to form a solution. Further, stirring the mixture of  $\text{TiO}_2$  and ethanol with a magnetic stirrer for 30

minutes occurred. Researchers incorporated urea into the  $\text{TiO}_2$  sol mixture, which was followed by stirring with a magnetic stirrer for 60 minutes. Evaporating the sol at a temperature of 70 °C under the hood for 1 hour occurred. Furthermore, heating the sol in the furnace at a temperature of 500 °C for 1 hour occurred.

Following the acquisition of N- $\text{TiO}_2$ , the application is carried out using the system spray coating method. The spray method is designed to evenly coat the glass with N- $\text{TiO}_2$ . A thin layer of N- $\text{TiO}_2$  can be formed on the glass plate using the spray method. This method can also save the use of N- $\text{TiO}_2$ , so that this method can produce N- $\text{TiO}_2$ -coated glass plates at a lower cost. After obtaining the  $\text{TiO}_2$ -coated N glass plate, the tool was made preservative and antibacterial in white oyster mushrooms with tools that have been made. To increase the lifetime of the oyster, after the photocatalyst process, put the oyster mushrooms in the vacuum container. Therefore, the use of nitrogen gas increases the time of food preservation. SEM-EDS is used to analyze the size of  $\text{TiO}_2$  nanoparticles, with a standard size range of 400-900  $\text{cm}^{-1}$ .

This method is used to identify the crystalline phase in the material by determining the parameters of the lattice structure and to find the particle size. An activity test of antibacterial activity was carried out on the type of E coli bacteria. Antibacterial activity test was carried whole-plate diffusion method using Nutrient Agar (NA) solid media with an incubation time of 1x24 hrs at 37 °C. This method is used to determine the effectiveness of the equipment in preserving and reducing the bacteria that are causing problems in white

oyster mushrooms. The effectiveness of the tool is demonstrated by comparing preservation using the refrigerator, and our tool innovations include identifying the organoleptic analysis, including shapes, colours, and odours in white oyster mushrooms.

## Result and Discussion

The initial process of making preservatives and antibacterial compounds in white oyster mushrooms begins with the synthesis process. Synthesis is carried out by the gel-sol method with the base material of *titanium tetrachloride*. The process of making nanoparticles is formed by a nanosol solution prepared with controlled hydrolyse of titanium tetrachloride in water. 5% in isopropyl alcohol (5:95) is dropped 1ml/min into 900 ml of distilled water at PH 2 (with). The mixture is stirred for 12 hrs. This solution is stable for some time. The doping with nitrogen is formed from the same sol stirred using a magnetic stirrer, followed by the addition of a diethylamine solution (to produce a nitrogen-sol 1:1 (Volume: Volume diethylamine)). The solution is calcined at a temperature of 400 °C for 2 hours to produce the  $\text{TiO}_2$  and N- $\text{TiO}_2$ . Nano-N-doped  $\text{TiO}_2$  obtained from this method is the basic material that will be used to coat glass plates with preservatives and antibacterial devices. After getting N-doped  $\text{TiO}_2$  then done the manufacture of glass plates N-doped  $\text{TiO}_2$  with a coating system using the spray method was done. This spray method is intended to make the glass covered with N-doped evenly. Here is the process of coating N-doped  $\text{TiO}_2$  on glass. Subsequently, making a glass plate coated with N-doped  $\text{TiO}_2$ , then the manufacture of

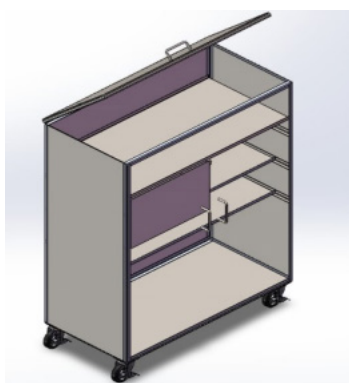


Figure 1. Equipment Design

preservatives on oyster mushrooms, designing outer equipment using metal and polymer plastic. Design of equipment made of metal and plastic so that the tool can be used for a long time. Then,  $\text{TiO}_2$  is made on a glass plate so that it can absorb sunlight and act as a photocatalyst.

The design of this equipment is made from several parts of the process, which includes the photocatalyst process. In this case, the photocatalyst process uses nitrogen-doped  $\text{TiO}_2$  and is placed in a vacuum chamber. In addition, the plan for this equipment is to apply white oyster nanotechnology, which is growing rapidly in Indonesia. Here are the specifications of the design of the tool that will be applied in the middle and top part (Figure 1). The preservative tool consists of 3 parts, at the top, only in use a lamp. Its main function is as a source of photons in the photocatalyst process. The middle part serves as a storage area as well as a place for the photocatalyst to take place. Then, at the bottom serves to store fresh mushrooms.

The process in the middle part of this equipment is the process of preserving oyster

mushrooms, as well as the process of drying oyster mushrooms. In this equipment, there are methods of photocatalysis. The use of photocatalyst processes serves as an antibacterial in a vacuum condition. Combining the two processes is due to the lack of antibacterial ability of the photocatalyst process. In addition to improve the equipment, it can be added the hygrometer in order to check the humidity. At the top of this equipment, a lamp is used as the source of photons in the photocatalyst process (Figure 2). This preservative tool uses 2 methods, namely, photocatalyst and ozonation. Equipped with a fan and a climate control system. The Ozonation process functions as a preservative due to the antibacterial ability of the ozonation process. Then the photocatalyst process serves as an antibacterial supporting ozonation. The combination of the two processes is due to the lack of antibacterial ability of the photocatalyst process and the wasteful use of ozone.

Nano particles of  $\text{TiO}_2$  that have been made using the sol-gel method were then performed characterization in the form of FTIR analysis to determine out oxides and



Figure 2. Equipment for the Preservation and Antibacterial Treatment of the White Oyster Mushroom



Figure 3. Result of Mushroom Preservation Using the Tools



XRD to find out the crystal form produced from the synthesis. Here are the results of the characterization of  $\text{TiO}_2$  nanoparticles. Based on the results of the FTIR spectrometer obtained peaks were obtained at 518 and 678  $\text{cm}^{-1}$ , which indicate the vibration of  $\text{TiO}_2$  (Figure 3). This is following research by Silva (2013), which pinpointed peak  $\text{TiO}_2$  in the range of 400-900  $\text{cm}^{-1}$  (Silva *et al.*, 2013). Based on Figure 4, analysis of the crystal phase  $\text{TiO}_2$  using XRD obtained a mixed phase in the form of anatase with a peak at 55.10 ( $2\theta$ ) and rutile at 27.40 ( $2\theta$ ). Oyster mushroom preservatives have a power of 50 Watts from a total of 5 lamps for preservation at night. This tool also has a load of 100 kg and can store a lot quantity of oyster mushrooms. The low energy of electricity for this equipment makes the more valuable than other food preservation equipment. In addition to mold preservatives, an Ultra High Purity nitrogen tube is devoted to food packaging. Photocatalysis is a method used to inhibit the growth of bacteria, which will cause fungi to rot quickly. This process can be called a continuous oxidation process, which also functions to oxidize dyes. The continuous oxidation process occurs due to the formation of hydroxide radicals (OH), which are strong oxidizing agents that can completely mineralize organic pollutants (Faisal *et al.*, 2007; Khan *et al.*, 2023).  $\text{TiO}_2$  is a type of semiconductor material that is non-toxic, widely available, has good chemical stability under visible light, and is relatively cheap to manufacture.  $\text{TiO}_2$  can absorb photon energy in most of the sunlight spectrum because it has a large band gap ( $>3.00$  eV), so  $\text{TiO}_2$  is often used in chemical reactions (Yuwono *et al.*, 2011; Etacheri *et al.*, 2015). The conduction band of titanium dioxide is particularly suitable for anthocyanin dyes, as it affects the injection of electrons from the dye molecules into the oxide semiconductor.  $\text{TiO}_2$  has a large surface area and charge transfer due to the easy enhancement of photon induction. The high surface area helps effective exposure to light and facilitates photochemical reactions on the surface, while the easy charge transfer due to photon induction helps the photon-induced electron capture and donation process (Narayan 2012).

Further, the photocatalyst reactions

produce  $\text{O}_2$  and OH radicals that can oxidise various bacteria with an efficiency of up to 82.47%. Technology based on this photocatalytic concept can also kill bacteria and microorganisms that cause meat decay. As a result, the use of this tool has the potential to become a white oyster mushroom. Oyster mushrooms are more durable and do not rot as easily as other mushrooms. Because ozone is a gas molecule that can kill bacteria in a vacuum system, oyster mushrooms are free of decay. An N- $\text{TiO}_2$  antibacterial activity test is done to find out the effectiveness in preserving oyster mushrooms. *E. coli* is one of the bacteria that cause white oyster mushrooms to rot easily. Therefore, antibacterial activity tests are carried out on types of *E. coli* bacteria. Antibacterial activity test is carried out by the well diffusion method using nutrient agar solid media (NA) with an incubation time of 1x24 hrs at a temperature of 37°C. The results obtained by N- $\text{TiO}_2$  can inhibit the growth of *E. coli* bacteria by  $6.48 \pm 0.42$  mm. Based on Wang's research (2014), N- $\text{TiO}_2$  can reduce bacteria by 98.70% using visible light. The mechanism of preserving oyster mushrooms with this tool is when the glass plate that has been coated with N-doped  $\text{TiO}_2$  is illuminated by sunlight/lamp (Figure 3) (Wang & Swerdloff 2014).

Based on organoleptic observations and water content measurements in this study, it can be concluded that the effective preservation time is 6-9 days with a soft texture, water content still around 80-82%, and a slightly brown colour. The rapid decline in the quality of oyster mushrooms is caused by several factors, one of which is that the mushrooms do not have a protective cuticle, high levels of respiration, and high water content (Brennan *et al.*, 2000). This damage is characterized by a change in the colour of the mushroom body to brownish due to enzyme damage, physical damage, microbiology, and shrinking of the white oyster mushroom mass (Castellanos-Reyes *et al.*, 2021). The quality of mushrooms continues to decline due to the quality of temperature in this tool, which follows the high temperature conditions around the room in that area. If oyster mushrooms are stored at high temperatures, it can cause a decrease in the weight loss value of oyster mushrooms.

Fungi are protected by a thin and porous epidermis, so they are unable to prevent rapid surface dehydration. Dehydration is related to temperature, humidity, osmotic pressure, and differences in solution concentration (Murcia *et al.*, 2009).

This is due to the presence of superoxide compounds (Table 1). The superoxide compound will oxidize the bacteria that are in the storage room of oyster mushrooms, so the bacteria will die.  $\text{TiO}_2$  N-doped material is a catalyst, so the photocatalyst reaction can continue continuously (Divyasri *et al.*, 2021). The slightly brownish colour obtained in the preservation data is due to the lack of temperature control in oyster mushrooms, so a temperature control tool is needed that can maintain the temperature of oyster mushroom preservation that ranges from 25-30°C. It can be concluded that preservation using photocatalysts can extend the preservation time of oyster mushrooms, which is about 6-9 days, longer than the ideal preservation of oyster mushrooms (6<sup>th</sup> day).

Photocatalysts are a method of AOPs (Advanced Oxidation Processes). Characteristic

of AOPs is the formation of highly active free radicals, especially hydroxyl radicals (OH) (Litter 1999). Here are the toughness of oyster mushroom preservatives: Can be used in the long term, Able to kill bacteria in oyster mushrooms, Able to preserve oyster mushrooms in a vacuum system, Effectively used by oyster mushroom farmers, Low cost, and The drying process does not depend on the weather. SWOT analysis is needed in the development of tools in the future. This is the SWOT analysis of this equipment (Table 2) (Silva *et al.*, 2013). To achieve food self-sufficiency in the 3T region, the role of technology is needed. In this case, the focus of the problem to be discussed is increasing the potential of rural areas in the oyster mushroom agricultural sector in Banyumeneng Village. Oyster mushroom is a food commodity that is one of the main needs of people in daily life. The problem is in rural areas where there is no electricity and a refrigerator in the preservation process, which can cause big losses for farmers, that is fungi can easily rot. Therefore, the authors offer a solution in the form of photocatalyst-based oyster mushroom processing technology and

Table 1. Organoleptic Analysis

Time	Texture	Water Content	Color
0 day	Soft	90%	White
3 day	Soft	88%	Rare brown
6 day	Soft	82%	Rare brown
9 day	Soft	80%	Rare brown
12 day	Hard	60%	Brown
15 day	Very Hard	30%	Very Brown

Table 2. SWOT of the equipment

<b>Strengths</b>	<b>Weakness</b>
1. Environmentally friendly technology	The synthesis process must be done in the laboratory for safety
2. Able to speed up the drying process of salty white oyster mushrooms	2. Still use electricity.
3. Low cost	3. There has been no development with the relevant parties.
4. Easy to use	
<b>Opportunities</b>	<b>Threats</b>
1. The need for renewable technology in agriculture	1. Scratched glass-coated photocatalyst Ag Doped $\text{TiO}_2$
2. Low implementation costs.	2. The public does not understand very well about photocatalysts and ozone technology
3. Can be used long term	

the presence of ozone technology as an agent in the oyster mushroom preservation process. It is hoped that the role of this technology can improve the welfare of people in coastal areas. On the other hand, this technology can be used with electricity or without electricity, that is, utilizing sunlight. The results of the FTIR spectrometer showed peaks at 518 and 678  $\text{cm}^{-1}$ , which indicated the presence of vibrations from  $\text{TiO}_2$ . Analysis of the crystalline phase of  $\text{TiO}_2$  using XRD obtained a mixed phase in the form of anatase with peaks at 55.10 ( $2\theta$ ) and rutile at 27.40 ( $2\theta$ ). Anti-bacterial activity test showed that N- $\text{TiO}_2$  was able to inhibit the growth of *E. coli* bacteria by  $6.48 \pm 0.42$  mm. The results of the organoleptic test showed that preservation using photocatalyst can extend the preservation time of oyster mushrooms, which is about 6-9 days, longer than the ideal preservation time of oyster mushrooms.

## Conclusion

The use of tool design is quite effective, where there are 3 parts of the preservative that have a function in the process of preserving white oyster mushrooms. The results of the organoleptic test showed that preservation using photocatalyst could extend the preservation time of oyster mushrooms, which was about 6-9 days, 3 days longer than the ideal preservation time of oyster mushrooms. The use of ozone and photocatalyst technology is not dangerous, because both are environmentally friendly technologies and easy to apply to white oyster mushrooms. This technological innovation can answer the problem of oyster mushroom farmers in Banyumeneng Village related to the oyster mushroom harvest, which is wasted because it is rotten and cannot be sold.

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