Solar surface - Solyta

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- Large scale functional coatings of photocatalysts on polyester fabric
Abstract
This final thesis goal has been to try and develop a titanium dioxide coating for water purification. The coating should be used in a manufacturing industry on textiles. Water purification is achieved through photocatalysis with titanium dioxide and UV-radiation, where hydroxyl radicals form through oxidation. The kind of textile fiber chosen for the substrates is a polyester fiber. The substrates have been developed in several different shapes. Two different knitted patterns was developed, one flat patterned and one wavy patterned. Another kind of substrate was a spacer-type and there was also a substrate made from a PET-bottle. The different substrates were chosen to compare the different coatings effect on different surfaces and the different coatings were developed from two types of titanium dioxide. The coated substrates have been analyzed for hydroxyl radical generating properties. By testing a sample of water with titanium dioxide against a sample with water that had no additives, it was shown that titanium dioxide generates more hydroxyl radicals. The results of the work showed that the recipe containing 3 % titanium dioxide and 3 % acrylic binder showed good properties for water purification. In addition, the results showed a stronger effect for the wavy patterned substrate then the flat patterned, which is an interesting result that should be researched further in the future.

Key words
Titanium dioxide, water purification, coating, methylene blue dye, UV-radiation
Sammanfattning


Sökord

Titandioxid, vattenrening, beläggning, metylen blå färg, UV-strålning
Preface
This project is initiated by PhD Nils-Krister Persson at the Swedish School of Textiles, as a final thesis work for a bachelor degree in Textile engineering. He was also my supervisor in this project.

I would like to thank Nils-Krister Persson, Catrin Tammjärv and Maria Björklund at the Swedish school of Textiles, Claes Brodell at Diazo Kemi AB, Elisabeth Nordmark-Andersson, Niklas Nordmark and Ellinor Andersson for their editorial support.
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1. Introduction

1.1 Background
Clean water is essential for the survival of mankind. Nevertheless, 884 million people around the globe does not have access to clean water and 5000 children under the age of five dies every year from diseases caused by unclean water, lack of sanitary toilets and a bad knowledge of hygiene (Unicef). Therefore, the research on how to hygienise water is really important. A lot of studies has been done and are in progress on the subject (Daoud, 2004a) (Daoud, 2004b), still more research needs to be done. One of these studies has examined a sol-gel derived titanium dioxide finish for cotton fabric, the finish provides the fabric with a self cleaning surface (Gupta, 2008). There has also been another study (Qi, 2010) on cotton fabric and nanocrystalline titanium dioxide, in this study the coatings affect on coffee, red wine and curry stains were investigated. Also the coatings affect on the cottons fabrics loss of mechanical strength. There has been still another study (Tung, 2010) about self cleaning fabrics with titanium dioxide, but in this study the substrate was wool.

At the Swedish School of Textiles many different studies have been executed, some of them have been on the subject of water purification. In 2009 Sofia Morsten and Siiri Yngvesson did their thesis work for a bachelor degree and in 2010 they did another thesis about water purification for their masters. They tried to find a way to develop and produce a textile water purification prototype for use in developing countries. The results of their thesis were that a textile surface containing titanium dioxide in water gives a high decrease in bacterial content when exposed to solar radiation. The most favorable textile finish was a dense knitted material with a sol-gel coating (Yngvesson, 2010).

1.2 Purpose
The purpose is to develop a titanium dioxide (TiO$_2$) coating for a water cleansing textile for an industrial production. Through a photocatalysis the TiO$_2$ coating should kill bacteria such as E coli$^1$, when it is exposed to UV-light. Several different coating recipes will be evolved and tested. The substrates are made of polyester fibers in different forms, to see if it makes a difference how the substrate is shaped and how the coating is applied to the different shapes. The question this thesis aims to answer is;

*How can a titanium dioxide coating be developed for water purification for an industrial textile production?*

Further this thesis aims to be a continuance on Sofia Morsten and Siiri Yngvesson bachelor thesis from 2009, which is part of the Soltråd project, a research project at the Swedish School of Textiles (Yngvesson, 2009).

1.3 Delimitation
The substrates for this thesis are all from polyester fiber (PET). Both textile and plastic PET will be used to compare if there is a different coating textiles or hard plastic. The substrates are knitted and not woven, because it is easier to get many small substrates in different patterns when knitting than if they would be woven. The coatings are limited to two different solutions that will fit in a pad-dry-cure method$^2$. This method has been chosen because it is a method that is commonly used in the textile industry. All chemical substances are the same as used in the textile industry. Because of limited resources this thesis will only be able to

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$^1$See chapter 3.4 page 5.
$^2$See chapter 4.3 page 8.
investigate the generation of hydroxyl radicals with methylene blue powder. Unfortunately there will not be enough time to investigate the different coatings effect on bacteria such as E. coli.

2. Method

2.1 Pre-studies
There are two different methods to gather information, a qualitative and a quantitative method. The difference between the two methods is that the quantitative method works with information that can be described with numbers, meanwhile the qualitative method works with information that can be described with words (Eliasson, 2006).

In this thesis different scientific methods have been analyzed and the work has in particular been based on previous results, reports, newspaper articles, field trips and books. The scientific method for this thesis is both the quantitative and qualitative research method. For the literature part of this thesis the qualitative research method was the one that was most frequently used. To get a deeper knowledge on the subject, books and articles were read and analyzed.

The gathering of information has been through different databases, magazines, articles, books, interviews and field trips. The key words that were most used in the research were; titan*, coating*, antimicrobial*, hygiene finish*, antimicrobial coating*, water purification and titanium dioxide. All the information that has been found on the subject have been studied, analyzed and processed. The field trip went to Gässlösa sewage plant in Borås, to get a fundamental knowledge on how sewage water is cleaned before they let it out into the nature again.

2.2 Execution
The greater part of this thesis is experimental. A lot of time was spent in the different laboratories at the Swedish School of textiles, depending on what part of the project that was being conducted. The substrates were knitted in the knitting laboratory and in the dye laboratory the recipes were mixed, then coated on to the substrates and finally tested for hydroxyl radicals.
3. Water purification
This section describes how water can be cleansed and how the photocatalysis works and how to detect photocatalysis.

3.1 Water purification in practice
There are many different ways to clean water and many different chemicals that can be used. It is also possible to clean water with only UV-radiation and a plastic container, two products of that kind are already on the market; SODIS (see 3.1.1) and Solvatten (see 3.1.2). Because many chemicals are harmful to human beings and animals it is important to consider what sort of chemical that is used in the process. TiO$_2$ is not harmful (Balfur, 1994), has great water cleansing properties and is easy to use in textile coatings and is therefore very suitable for water purification.

In many water filters there is a textile substrate of some sort. If this substrate has a pattern that generates a larger area it could contribute to more reaction sites and that would increase the water cleansing process. A substrate with high porosity could increase the reaction depending on how the water purification process is designed. It could also have the opposite effect if the water is cleansed with UV-radiation and the radiation is blocked from the organic contaminations by the cavities in the substrate. When comparing the SODIS method to a method with TiO$_2$, the TiO$_2$ method has an advantage that it can work faster and generate a stronger reaction if the textile has a functioning coating.

3.1.1 SODIS
SODIS is short for solar water disinfection – the SODIS method. It is a simple procedure to create disinfected drinking water with only UV-radiation and a bottle. The method is developed for underdeveloped countries in equatorial climates to prevent diseases like diarrhea. It is an initiative of Eawag (Eawag, 2011).

A transparent PET-bottle or glass bottle is filled with contaminated water and is then exposed to the sun for at least 6 hours. If it is very cloudy, more than half the sky, the water should be exposed to the sun for 2 consecutive days. If it is raining, for example during the rainy season in countries with equatorial climates the method is not recommended. The UV-radiation of the sun kills the diarrhea generating pathogens in the water. If the turbidity of the water is very high, the effect will be reduced. The water might need to be rinsed before it can be cleaned. The transparency of the PET-bottle is also important, if the bottle is heavily scratched, the bottle needs to be replaced. The bottle should not exceed the size of 3 liters.

The treated water should be used within 24 hours. It should be kept in the bottle and drunk directly from the bottle, or from a glass that have been poured immediately before use. (SODIS)

3.1.2 Solvatten
Solvatten is a Swedish invention for household water treatment (Solvatten). It is a portable 10 liters container that can provide drinkable water in 2-6 hours, producing 20-30 liters of water per day. With the help of UV-light, Solvatten can inactivate micro-organisms that causes diarrhea and diseases, it can also hygienise water that contains bacteria, viruses and parasites.

The product is based on three methods for treating contaminated water; filtration, heat pasteurization and UV-disinfection. The filter can be a piece of folded local fabric. The turbidity can affect the result if it is too high, therefore the water should be pre filtered. The filter in the container can reduce the turbidity with about 35 %. Solvatten is developed for countries with a lot of sun, but it does not necessarily mean it has to be a warm country.
Mountain areas as well as lowlands are suitable as well, as long as it is a sunny day. The container has an indicator that shows when the water treatment process is completed and the water is suitable for drinking.

The water treatment does not affect the taste of the water and it can also be used for personal hygiene, washing and rinsing of food. This could help preventing infections. Solvatten cannot make drinkable water out of saltwater or chemically polluted water. (Solvatten)

3.2 Photocatalysis
Photocatalysis is a chemical reaction where a catalyst is activated electromagnetically in the ultraviolet area. In a catalyzed photolysis the light is absorbed into the substrate. In a photogenerated catalysis the photocatalytic activity is dependent on the catalysts ability to create electron-hole pairs, which will then create free radicals which will undergo secondary reactions (Fujishima, 2000). The commercial application on this process is called an advanced oxidation process. It is common to use TiO$_2$ as a catalyst, but it is also possible to use other substances. Ultraviolet light does not always have to take part in the reaction. How the reaction is defined often depend on what sort of hydroxyl radical that is created in the reaction and how it is used (Hashimoto, 2005).

TiO$_2$ is highly effective in oxidizing hydroxyl radicals, which eagerly attack and decompose organic contaminations in the water. The reaction mechanisms of the photocatalytic effect do not happen in the specific order, but the schematic view is 1-8, it is an overview of events that occurs parallel to each other. Step 1 shows the excitation of TiO$_2$ by UV-light. The oxidation reaction and reduction reaction is shown in step 2-5. Step 6-8 shows that oxygen is produced by water oxidation and consumed via reduction. (Hashimoto, 2005),(Fujishima, 2000).

\[
\begin{align*}
1) & \quad TiO_2 + 2hv \rightarrow 2e^- + 2H^+ \\
2) & \quad H_2O + 2h^+ \rightarrow (1/2)O_2 + 2H^+ \\
3) & \quad 2H^+ + 2e^- \rightarrow H_2 \\
4) & \quad H_2O + 2hv \rightarrow (1/2)O_2 + H_2 \\
5) & \quad 2H_2O + 4h^+ \rightarrow O_2 + 4H^+ \\
6) & \quad O_2 + 2H^+ + 2e^- \rightarrow H_2O_2 \\
7) & \quad O_2 + e^- \rightarrow O_2^- \\
8) & \quad O_2^- + 4e^- + 4H^+ \rightarrow H_2O
\end{align*}
\]

The difficulties with the TiO$_2$ photocatalysis are the controllability of the structural properties, the enhancement of catalytic activity, narrowing the band-gap energy and the immobilization to form films and membranes. If the TiO$_2$ is provided with sufficient energy by photons from UV-light, electrons and electron-holes are produced and a reduction reaction forms hydroxyl radicals, superoxide radical anions and hydroperoxyls. The hydroxyl radicals can oxidize most organic contaminations into their mineral product, such as H, O and CO. TiO$_2$ photocatalysis in general shows fast reaction kinetics for organic decomposition and for that reason requires short treatment times (Savage, 2009).
3.3 Titanium dioxide

Titanium dioxide is a naturally occurring oxide of titanium and it is also known as titanic oxide, titanium white, titanium anhydride or titania. It is a white and bright compound and its chemical formula is TiO₂ (Murphy, 2001). Titanium dioxide is extracted from four different minerals; rutile, anatase, brookite and ilmenite. Depending on which one of the four minerals the titanium dioxide is obtained from, it get different crystal structures (Wypych, 2010). From the first three minerals it gets an octahedral crystal structure. Both rutile and anatase has a tetragonal structure, the property that separates them is the arrangement and the stability of the structure, Image 1. Anatase is more stable then the rutile form. Brookite has an orthorhombic structure. Trigonal ilmenite is an earlier component in magma crystallizations (Patnaik, 2003).

The anatase form of titanium dioxide has the best absorption of light in the wavelengths between 380 nm and 420 nm and is therefore the only one that is suitable for photocatalysis. It is manufactured using a sulfate process. The process is a wet process with several different steps, either as a continues process or as a batch process (Wypych, 2010). Titanium dioxide is dissolved in concentrated sulfuric acid until it precipitates hydrous salts. This is then calcined. The product from the calcination process can take two forms, either anatase or rutile (Murphy, 2001).

3.3.1 Environmental effects and health aspects

Titanium dioxide is not a poisoning substance, its inertness results in lack of toxicity. Therefore, titanium dioxide is not classified as a health hazard (Balfur, 1994). The Swedish medical products agency’s provisional evaluation from 2007 states that titanium dioxide does not increase any cancerogenic hazard if it is digested or exposed to human skin (Läkemedelsverket, 2007). There have been no environmental effects reported for titanium dioxide.

3.4 Indication of photocatalytic effect

There are several different factors that can affect the photocatalytic affect of TiO₂; the impurity of the substance, its crystal form, the particals size, the turbidity of the water, the surface area of the substrate and the density of the surface. Another factor that can influence the result is the environmental factor. The intensity and wavelength of the light source and the time of which the TiO₂ is exposed to the lights source can also affect the result (Erdem, 2008).

An indicator to see if there is any photocatalytic effect is to do a methylene blue dye test. The methylene blue turns colorless when reacting with oxygen and that indicates that there are hydroxyl radicals. It is also possible to detect the unpaired electronholes in the photocatalysis with an Electrone Spin Resonance (ESR) (Grisco, 2001). The electrons and electron-holes that are produced in the photocatalysis goes through a reduction reaction that forms hydroxyl radicals that can oxidize most organic contaminations, such as Escherichia coli, which is a member of the Enterobacteriaceae family (Ohgaki, 2001). It is a species that is well known for being a food borne pathogens. It is classified as a coliform, which is a general term that describes Gram-negative asporogenous rods that ferment lactose within 48 hours. They
are also known for their colonies that are dark and radiate a green sheen when exposed to methylene blue. It normally lives in the gut of animals and human beings and is therefore an indicator of fecal contamination (Robinson, 2000). Not all E. coli strains causes disease (Blackburn, 2009).

Turbidity is the measurement of material in the water and is measured in nephelometric turbidity units (NTU). Nephelometry is a method that measures light that pass through a sample at a certain angle. The material in the water can be; clay, silt, organic and inorganic matters as well as micro-organisms. It is measured by detecting how much light is scattered by particles in a sample compared to a reference (Crittenden, 2005). For decades turbidity has been used as an indicator of whether the water is of drinking quality or not and how efficient water filtrations are. To establish a good filtration result, the turbidity removal should be efficient. Pathogens which aggregate with particles should be removed. Turbidity is not an effective source of measurement when it comes to the quality of drinking water, because the method does not say what the nature of the particles contaminating the water is, just that the water is unclean (Letterman, 1999). The particles in the water can also serve as a protection for the pathogens when the water is cleaned with UV-light, blocking the light from the pathogens. The particles may also absorb toxic substances and encase them (AWWA, 2011).

3.5 Methylene blue

Methylene blue (MB) is a chemical compound that can be used in a variety of different fields, such as biology and chemistry. It is commonly used in biology to stain bacteria in order to easier see the shapes of the bacteria under a microscope. It appears as a solid green odorless powder in room temperature and when it is dissolved in water it turns blue. The blue color is a reaction between the MB and oxygen, if the oxygen would be removed, the blue color would disappear (American Chemistry Council, 2005-2010). Its chemical name is tetramethylthionine chloride (Cragan, 1999). It should not be confused with methyl blue or methyl violet.

MB can be used to detect hydroxyl radicals in a photocatalytic reaction with TiO₂. Because the MB turns colorless when reacting with oxygen it works as a decolorization. According to studies the MB has a higher photocatalytic decoloring efficiency with solar light irradiation then with an artificial UV light source (Kuo, 2000).

\[ \begin{align*}
9) \quad MB + TiO_2 & \rightarrow MB + h^+ + e^- \\
10) e^- + O_2 & \rightarrow O_2^- \\
11) h^+ + OH^- & \rightarrow OH \\
12) MB + O_2 & \rightarrow MB + 2(O(\text{^1D}))
\end{align*} \]

The methylene blue molecules absorbed on to TiO₂ can either undergo a photosensitization mechanism (9-11) or form electronically excited oxygen atoms, the singlet oxygen atom (12) that will promote the decoloring efficiency (Kuo, 2000).

3.6 Chroma

A spectrophotometer is used to get information on materials spectrally selective characters. Valuable information can be obtained from spectral curves to identify colors (Koleske, 1995). The spectrophotometer shows lightness (L*), chroma (C*) and hue (h) of the water sample. In order to compare the different water samples with one variable that is comparable to all the samples, it is most suitable to compare DC*. DC* is delta-chroma which indicates the “purity” of the color (Tomsic, 2000). When a color get washed out, the DC* number
decreases. It is therefore a good indicator for the MB which turns transparent when generating hydroxyl radicals.
4. Experimental studies
This section describes the experimental work that has been done for this thesis. What different materials have been used and how the recipes have been put together. The section also describes how the substrates have been coated and what sort of test they have undergone.

4.1 Introduction
The coating that was developed is only meant to work for water purification. If the coating should be used for air purification a lot more research would be needed on the subject and for this project there is not enough time. Two kinds of coatings have been developed into several different recipes, one based on water and TiO$_2$ and one based on ethanol and TiO$_2$. It is also possible to make coatings with plastisol or silicon, but both of these substances are very difficult to work with, therefore they were not a part of this project. In order to compare the results from the different recipes, all the substrates that were used are polyester fiber (PET). The yarn is made from filament polyester fibers. The substrates were knitted into different shapes in the hand knit laboratory. It is easier to get many small substrates in different patterns when knitting than if they would be woven. The coatings were also applied on a PET-bottle and a spacer-material. There are many different ways to apply a coating to a substrate, in this thesis work the pad-dry-cure method$^3$ were used. This method has been chosen because it is a simple but efficient method to get an even coating on the substrate. Other methods that were discussed but eliminated were to apply the coating with a squeegee or to spray the coating on to the substrate.

To see if the coating had the correct properties for water purification water samples were taken during a methylene blue test and analyzed in a spectrophotometer. The chroma value indicated if the coating were functioning or not. The water sample was also inspected by the naked eye for a change in the blue tone of the water. If a difference could be noticed by eye, the spectrophotometer would verify the result. The light source in this project for the solar radiation was a xenon-lamp from Suntech Group AB in Vänersborg, model name HI-1200. It is a lamp that simulates sunlight. Therefore the UV-radiation from the lamp is not that high. Different distances between the lamp and the sample had to be examined to see if it had any effect on the results. Because of the limited time resources for this project no test for bacteria could be done, only a MB test to detect hydroxyl radicals.

4.2 Substrates
Four different types of substrates have been developed.

- A wavy pattern, knitting kind 1:1 elastic and stocking stitch, knitted on a hand knit machine with a filament polyester yarn, PES Dtex 167/48/1.
- A flat pattern, knitting kind 1:1 elastic, knitted on a hand knit machine with a filament polyester yarn, PES Dtex 167/48/1.
- A spacer material.
- A piece of an ordinary PET-bottle.

The knitted patterns were developed on a hand knit machine, image 2. Several patterns were reviewed before the two that were selected. A few test samples were knitted to get the

$^3$ See chapter 4.3 page 8
right size and shape, image 3. Then 18 substrates were knitted in each pattern, image 4. The spacer material was already developed by the technicians at the knitting laboratory. The PET-bottle was put in a hot water bath for a couple of hours to dissolve the glue from the label on the bottle. The label could then easily be removed without any residue. Round substrates in the same size as the test bowl were then cut out with a knife, image 5.

4.3 Recipes
Six different recipes with TiO$_2$ have been used.

- The first recipe contains water, 1% TiO$_2$ powder and 1% acrylic binder and has a yellow and white marking.
- The second recipe contains water, 2% TiO$_2$ powder and 2% acrylic binder and has a green marking.
- The third recipe contains water, 3% TiO$_2$ powder and 3% acrylic binder and has a lilac marking.
- The fourth recipe contains water and 2% acrylic binder and has a pink marking.
- The fifth recipe contains the same amount of chemicals as recipe 3 but is prepared in a different way, see chapter 4.4. The recipe has a blue marking.
- The sixth recipe is a sol-gel mix, containing titanium-IV isopropoxide (Ti(O-iPr)$_4$), ethanol (70%) and acetic acid and has a red marking.

The recipes and coating methods followed Gupta (Gupta, 2008), but have been slightly altered in this experiment due to limited resources and to fit an industrial textile process. The TiO$_2$ powder, Aerosilde TiO$_2$ P25, is a hydrophilic fumed and highly dispersed titanium dioxide manufactured by Aerosil. The titanium (IV) isopropoxide, 97% is manufactured by Sigma-Aldrich. The acrylic binder Jaypol B2 is a binder for the textile industry provided from Diazo Kemi AB.

Only the knitted substrates have been used to all different recipes. The spacer material was only coated with recipe 6 and the PET-bottle with recipe 1 and 6. For a complete overview of all different samples see table 6 page 16. The wavy patterned and flat pattern knit should have been applied with recipe 1-3 in two different ways. One way was to dip yarn in TiO$_2$ solution before knitting and the other way was to knit the substrate first and then dip it in the coating. Because the TiO$_2$ particles did not stick to the yarn as much as needed, it was not possible to knit the yarn without ruining the machine. Therefore, the coating could only be applied after knitting, see chapter 4.4.1 page 12.
Recipe 1 Yellow and white
The first recipe contains 98 % water, 1 % TiO₂ powder and 1 % acrylic binder. 196 g water was mixed with 2 g of TiO₂ powder and 2 g acrylic binder. It was then stirred with an even pace until the solution was smooth, image 6. 3 flat substrates (Yellow 1.X) were dipped in the solution one by one and the solution was stirred for 2 minutes with each substrate, image 7. Another coating was made in the same way and 3 wavy substrates (Yellow 2.X) were dipped in that solution. A third solution was made were one of each knitted substrate and one piece of a PET-bottle were dipped.

The first 6 substrates were washed together and marked with yellow thread, image 8. The two substrates that were not washed after curing was marked with white thread, see table 1.

<table>
<thead>
<tr>
<th></th>
<th>Weight (g)</th>
<th>Washing</th>
<th>Adhesion 1-5</th>
</tr>
</thead>
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<tr>
<td>Yellow 1.1</td>
<td>5.00</td>
<td>60°C</td>
<td>1</td>
</tr>
<tr>
<td>Yellow 1.2</td>
<td>4.59</td>
<td>60°C</td>
<td>1</td>
</tr>
<tr>
<td>Yellow 1.3</td>
<td>3.78</td>
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<td>1</td>
</tr>
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<td>4.36</td>
<td>60°C</td>
<td>1</td>
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<td>4.36</td>
<td>60°C</td>
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<td>4.26</td>
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</tr>
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<td></td>
</tr>
<tr>
<td>White 2.1</td>
<td>No</td>
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<td></td>
</tr>
<tr>
<td>PET-bottle</td>
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<td>5</td>
<td></td>
</tr>
</tbody>
</table>

Table 1. Weight, washing and adhesion result for recipe 1.

Recipe 2 Green
The second recipe contains of 96 % water, 2 % TiO₂ powder and 2 % acrylic binder. 192 g water was mixed with 3 g of TiO₂ powder and 3 g acrylic binder. It was then stirred with an even pace until the solution was smooth. 3 flat substrates (Green 1.X) were dipped in the solution one by one and the solution was stirred for 2 minutes with each substrate. Another coating was made in the same way and 3 wavy substrates (Green 2.X) were dipped in that solution.

The 6 substrates were washed together and marked with green thread, see table 2.

<table>
<thead>
<tr>
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<th>Weight (g)</th>
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<tr>
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<tr>
<td>Green 2.3</td>
<td>4.50</td>
<td>60°C</td>
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</tbody>
</table>

Table 2. Weight, washing and adhesion result for recipe 2.
Recipe 3 Lilac
The third recipe contains of 94 % water, 3 % TiO\textsubscript{2} powder and 3 % acrylic binder. 188 g water was mixed with 6 g of TiO\textsubscript{2} powder and 6 g acrylic binder. It was then stirred with an even pace until the solution was smooth. 3 flat substrates (Lilac 1.X) were dipped in the solution one by one and the solution was stirred for 2 minutes with each substrate. Another coating was made in the same way and 3 wavy substrates (Lilac 2.X) were dipped in that solution.

The 6 substrates were washed together and marked with lilac thread, see table 3.

<table>
<thead>
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<th>Weight (g)</th>
<th>Washing</th>
<th>Adhesion 1-5</th>
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</tr>
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Table 3. Weight, washing and adhesion result for recipe 3.

Recipe 4 Pink
The fourth recipe contains of 99 % water and 1 % acrylic binder. 198 g water was mixed with 2 g acrylic binder. It was then stirred with an even pace until solution was smooth. One flat substrate (Pink 1.1) and one wavy substrate (Pink 2.1) was dipped in the solution for 2 minutes one by one and stirred with an even pace.

The substrates were not washed, only marked with pink thread.

Recipe 5 Blue
The fifth recipe contains of 94 % water, 3 % TiO\textsubscript{2} powder and 3 % acrylic binder. 188 g water was mixed with 6 g of TiO\textsubscript{2} powder and 6 g acrylic binder. It was then stirred with an even pace until the solution was smooth. 3 flat substrates (Blue 1.X) were dipped in the solution one by one and the solution was stirred for 2 minutes with each substrate. Another coating was made in the same way and 3 wavy substrates (Blue 2.X) were dipped in that solution.

The 6 substrates were washed together and marked with blue thread, see table 4.

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<th>Weight (g)</th>
<th>Washing</th>
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Table 4. Weight, washing and adhesion result for recipe 5.

Recipe 6 Red
The sixth recipe was a sol-gel mix, containing 11.36 grams titanium- IV tetraisopropoxide (Ti(O-iPr)\textsubscript{4}), 184.4 grams ethanol and 0.2 grams acetic acid. The Ti(O-iPr)\textsubscript{4} and acetic acid were mixed together. Then the ethanol was dropped into the mix, one drop at the time for approximately two hours during constant stirring. The mix was then supposed to be stirred until it was...
transparent, but after 76 hours most of the solution had evaporated, so another mix was
prepared and stirred for 12 hours. It was not transparent when the substrates were dipped. The
substrates were dipped for 1 minute under stirring. 3 flat patterned substrates (Red 1.X) and 3
wavy patterned substrates (Red 2.X), one spacer-material and one piece of a PET-bottle were
dipped.

The 6 knitted substrates were not washed, only marked with red thread, see table 5.

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<th>Weight before dipping</th>
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<th>Adhesion</th>
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</table>

Table 5. Weight and adhesion result for recipe 6.

4.4 Method of coating
The substrates have all been coated with a pad-dry-cure method, were the substrate first is
dipped into a pad-batch, then dried in an oven and then cured at a higher temperature in an
oven. Depending on the recipe the pad time and dry- and cure temperature can differ.

- Recipe 1-3 were all dipped in coating for 2 minutes, whilst stirring the coating. The substrates were then
calendered, image 10, dried in a pre-heated oven for 4
minutes in 80°C and then cured for 3 minutes in
140°C.
- Recipe 4 was dipped in coating for 2 minutes, whilst
stirring the coating. The substrates were then
calendered, dried in a pre-heated oven for 4 minutes
in 80°C and then cured for 3 minutes in 140°C.
- Recipe 5 was dipped in coating for 2 minutes, whilst
stirring the coating. The substrates were then
calendered, dried in a pre-heated oven for 4 minutes
in 80°C and then cured for 3 minutes in 140°C.
- Recipe 6 was dipped in coating for 1 minute. The substrates were then dried in a pre-
heated oven for 10 minutes in 80°C and then cured for 30 minutes in 100°C.

4.4.1 Coating of yarn
When washing the yarn all the threads were tangled together and it was not possible to
separate them and make a skein, image 11. Therefore a skein that had not been washed was
coated instead, but most of the coating stuck to the yarn as lumps, image 12, instead of
attaching to the fiber forming a smooth coating. Also the TiO₂ powder from the coating came
of the yarn which made it impossible to knit in the hand knitting machines. Another problem
was that the fibers of the yarn swelled from the heat of the oven, image 13.
4.5 Methylene blue dye-test

A methylene blue dye-test was developed to detect hydroxyl radicals. A bowl with a mix of 500 ml of water and 1.6 milligram of MB powder was placed under a Zenon-lamp for 60 minutes. The distance between the lamp and the table that the bowl was placed on was 77 cm. The MB powder was from GFS Chemicals. A magnetic flea was placed in the water to stir at an even pace, *image 14*. 100 ml of water was removed from the bowl after ten and thirty minutes and was used to measure the color deviation in a spectrophotometer, Datacolor check pro model 200-1522. Two additional samples were measured, one before the water mix was put under the lamp and one after 60 minutes. The four different sample results were then compared to detect any hydroxyl radicals.

*Image 14. Coated sample under UV-light before and after a reaction has occurred.*

Four reference samples were also measured. One with only water and MB dye, two with the water + MB and a substrate without coating, one flat knitted and one wavy knitted and one with water, MB dye and 1 gram of TiO$_2$ powder.

The color deviation of the water samples were measured in the spectrophotometer and a chroma value for each sample was taken. The chroma value of the first sample was then compared to the chroma value of the following samples to see if there had been any reaction in the water. If the chroma value had decreased hydroxyl radicals had been detected. The chroma value in the first sample for all the knitted substrates were between 15-20 and the last sample in the tests were hydroxyl radicals were detected the chroma value was between 0-5, *figure 1 and 2 page 17.*
4.6 Washing
All knitted substrates were washed at 60°C in a household washing machine before they were coated. After the substrates dipped in recipe 1-3 were cured they were washed in an ordinary household washing machine at 60°C and left to dry at room temperature. Recipe 4 and 6 was not washed at all after curing. The substrates dipped in recipe 5 were washed in an ordinary household washing machine at 40°C and left to dry at room temperature. The substrates dipped in recipe 6 were tested once before washing and then hand washed and dried at room temperature.

4.7 Adhesion test
An adhesion test was made on all the coated substrates to investigate the adhesion of the coatings after laundry. A piece of Scotch was taped on to the substrate and rubbed two times, it was then removed. If there were a low degree of adhesion between the substrate and the coating, the coating would come off and transfer on to the Scotch. The result of the test was the valued in the interval 1-5, the value was estimated from sample to sample and 5 was the better value.
5. Results
The results from the experiments with the different recipes and substrates are described in this section.

5.1 Substrates and coatings
The adhesive test showed that the 60°C washing on the substrates coated with recipe 1-3 was very tough on the coating and a lot of the coating came off in the wash. Observed through a microscope there was a great difference in the texture of the substrates when comparing the substrates that had not been washed at all. The unwashed sample, image 16, had a lusterless finish compared to the washed samples, image 15, that had a more gleaming finish.


5.2 Photocatalytic effect

5.2.1 Methylene blue dye-test
In the reference tests with no substrates and with substrates that did not have any coating, there was no significant reaction. With the reference containing water, MB dye and TiO$_2$ powder there was a significant reaction after less than ten minutes. The substrates coated with recipe 1 that had been washed gave no significant effect. However, the substrate that had not been washed had a clear reaction after 30 minutes. It had the same effect again after a gentle wash by hand. Only the first wavy patterned substrate coated with recipe 3 had a significant reaction. All the substrates that had been coated with recipe 5 gave significant reactions, but the substrates that had been dipped first in the coating gave an even stronger reaction. The PET-bottle coated with recipe 1 gave a minor reaction and a lot of the coating came off the bottle in to the water mix. The substrates coated with recipe 4 gave a visible reaction in the test, but not nearly as strong as the ones coated with the recipes that contained TiO$_2$. Recipe 6 did not give any visible results before or after washing. A full overview of all substrates is shown in Table 6 page 16.

5.2.2 Efficiency
It is possible to see a significant reaction with the naked eye, but in order to see small reactions as well the readings from the spectrophotometer will help. Figure 1-3 shows the delta chroma value for different exposure times for the different substrates. The initial measured value for the first water sample could differ a little between the samples because of the human factor when mixing the water with the MB, but as seen in figure 1, figure 2 and figure 3 it does not affect the final reading of the result. If the test for example had a value of 15 at the first sample, it would still have the value of 15 at the last sample if there had been no reaction. As shown in figure 1-3 the delta chroma value for the samples have large decrease if
their delta chromas value indicates great properties for water purification. The figures also show that the TiO\textsubscript{2} needs at least 30 minutes to react properly.

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<th>TiO\textsubscript{2} 2%</th>
<th>TiO\textsubscript{2} 3%</th>
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<th>Sol-gel</th>
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Table 6. An overview of all substrates, recipe, washing and the final results.
Figure 1. Photocatalytic decolorization of MB, flat patterns.

Figure 2. Photocatalytic decolorization of MB, wavy patterns.

Figure 3. Photocatalytic decolorization of MB with coatings of recipe 6.
6. Conclusion

The purpose of this thesis was to develop a titanium dioxide coating for water purification for an industrial production in textiles. The question this thesis aimed to answer was:

*How can a titanium dioxide coating be developed for water purification for an industrial textile production?*

The experimental part of this thesis shows that the coating for recipe 5 generates hydroxyl radicals. Hydroxyl radicals and antimicrobial properties are related, for that reason it is possible to assume that the coating that generates hydroxyl radicals also should have properties suitable for water purification. It is therefore possible to make a full industrial production of textiles coated with TiO₂ for water purification purposes. But in order to do so there are some factors that needs to be considered from the conclusions in this section.

The first conclusion is that the results of the coatings depend on how they are washed after they have been cured, if they are washed at all. The substrates that were not washed gave a good result, but they also shed a lot of the coating in to the water which is not a positive result. The substrate coated with TiO₂ powder, that were hand washed, showed better hydroxyl generating properties. The substrates that were washed in a machine at 40°C had to be coated with 3 % TiO₂ powder to show any good properties.

Another conclusion is that the pattern of the substrate affects the hydroxyl generating properties. The wavy pattern which generates a larger area then the flat pattern shows faster and stronger properties for hydroxyl generation. It is therefore possible to conclude that larger areas generate more hydroxyl radicals and hence better water purification properties. The time the substrate is exposed to solar light is also an important factor to consider. The test result shows that it takes at least 30 minutes before a proper reaction occurs.

It has also been shown in this report that the structure of the substrate affects the coatings ability to attach to the substrate. A hard material like a PET-bottle is not a suitable substrate for this kind of coatings. The coating comes off in the water and the water purification properties were very poor. Also the sol-gel coating in this report did not show any properties for water purification.
7. Discussion
The reason recipe 1-3 failed was because they could not handle the roughness of being laundered in 60°C. When the substrate coated with recipe 1 was tested without any washing, it gave a really good result generating hydroxyl radicals. Therefore, a try with recipe 3 but with a kinder washing was made, this resulted in recipe 5, which gave very good results.

An interesting observation was that the wavy patterned substrates generated more hydroxyl radicals. This was discovered when the water with MB turned clear faster with a wavy patterned substrate in it, than with a flat patterned substrate. The reason why the wavy patterned substrate is better than the flat patterned one could be that it generates a bigger reaction surface and can therefore contain more TiO₂. This is only a theory and more research needs to be made.

Recipe 4 which only contains water and acrylic binder did also get a small result of generating hydroxyl radicals. This could be because of the acrylic binder that might contain small doses of TiO₂. The content is not declared on the material safety data sheet.

When preparing recipe 6 the sol-gel solution should have turned transparent. But after 76 hours of constant stirring, the solution still had a shade of white. After careful consideration I decided to try the coating anyway. As seen in the results the coating did not work and the reason for that might be because it never turned transparent. The ethanol percent content were only 70 and in other articles the percent content have been as high as 99.7 (Daoud, 2004a), this could also be a factor why the coating did not work.

The PET-bottle coated with recipe 1 gave a minor reaction but most of the coating came off during the test, therefore it would not manage a second test. The PET-bottle coated with sol-gel did not show any hydroxyl radicals and after the test all the coating had come off the bottle. Conclusively the PET-bottle did not work as a substrate for this purpose and a soft textile with a wavy patterned is more suitable for coatings intended for water purification. The spacer material that was treated by the sol-gel method showed no reactions.
8. Future investigations
There are two things in this thesis that I think would be very interesting to do a deeper analysis about.

The first one is the possibility to coat the yarn before it has been knitted. If it is possible to make a coating that really sticks to the fiber I think it would be more effective to coat the yarn before it is knitted. The coating would be more evenly placed over the entire sample and it would also cover more of the fiber and would therefore have a larger area to react on.

The second thing is the wavy pattern. It would be really interesting to knit several different patterns and compare them to each other. To see if large areas generates more hydroxyl radicals or if it was just a coincident in my case. It is the same thought as with coating before knitting; does a larger area generate a greater reaction?

I think there are a lot of different angles to this subject that can be researched even further. It is an important subject now and even more so in the future.
References


**Picture:**
Image 1, (Koleske, 1995).
Image 2-16, own photos.
Figure 1-3, illustration.