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OPTIMIZING ANTI-MOSQUITO COATING WITH SOL-GEL PROCESS FOR COTTON TEXTILES

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ABSTRACT

Since insect-borne infectious diseases are responsible for a great number of deaths each year, it is essential to focus on developing methods to prevent them. Medicine has not provided a solution for this which is why manufacturing fabrics with anti-mosquito properties is essential.

The purpose of this thesis was to optimize anti-mosquito coating for cotton textiles. It was carried out using the sol-gel process. The work consisted of finding the best methods to fix sol-gel solutions and prepare films out of them. In addition, it was crucial to focus on retaining the physical properties of the textile substrate. This was examined with different characterization techniques.

The practical work of the project was performed in Universitat Politècnica de Catalunya in the Textile and Paper Department in Terrassa, Spain. This part of the thesis and all the results remain classified in order to respect the request from UPC.

One of the theoretical parts of the thesis focuses on understanding the coating and sol-gel process together with chemicals utilized in this work. The most important chemical was permethrin since it was the active principle. Various experimental procedures and techniques were used and it was necessary to study them first to comprehend them. The practical part describes all the processes and experiments which were performed during the project but they remain classified. The work itself consisted of mainly fixing sol-gel solutions and optimizing them based on the coatings results.

Key words: anti-mosquito, sol-gel process, coating, permethrin

Lahden ammattikorkeakoulu

Tekstiili- ja vaatetustekniikan koulutusohjelma

KOSKELA, SELINA:

Anti-hyönteiskouttauksen optimointi sol-gel-prosessilla puuvillatekstiileille

Tekstiili- ja vaatetustekniikan opinnäytetyö, 36 sivua

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TIIVISTELMÄ

Hyönteisten kuljettamat taudit ovat syy moniin kuolemiin joka vuosi, minkä takia on tärkeää kehittää tapoja, millä vältytään niiltä. Nykypäivän kehittynyt lääketiede ei ole löytänyt parannusta näihin tauteihin, minkä vuoksi olisi oleellista valmistaa kankaita, joissa olisi anti-hyönteisominaisuuksia, jotka estävät näiden tautien saamista.

Opinnäytetyön tarkoituksena oli optimoida sol-gel-prosessilla valmistettua anti-hyönteiskouttausta puuvillatekstiileille. Työssä oli tärkeää löytää mahdollisimman hyvät menetelmät valmistaa sol-gel-liuoksia sekä kouttaksia kankaalle ilman sen fyysisten ominaisuuksien menettämistä. Tätä tarkkailtiin erilaisilla kokeilla, jotka luonnehtivat kyseisiä ominaisuuksia.

Itse projektin käytännön osuus suoritettiin Katalonian ammattikorkeakoulussa Tekstiili- ja paperialalla Terrasassa, Espanjassa. Käytännönpuoli koostui lähinnä sol-gel-liuoksien sekä kouttaksien valmistamisesta ja näiden optimoimisesta. Tähän kuului myös erityyppisiä kokeita, joita suoritetaan koutatuille näytteille. Koululta tulleen toiveen mukaan tämä osa työstä ja kaikki tulokset pysyvät salaisena.

Opinnäytetyön teoriaosuus käsittelee muun muassa kouttaus- ja sol-gel-prosessia sekä kemikaaleja, joita on käytetty projektissa. Kemikaaleista tärkein on permetriini sen ollessa työn vaikuttava ainesosa. Teoriapuoli tarkastelee myös erilaisia projektissa käytettyjä kokeellisia menetelmiä ja tekniikoita, jotka on täytynyt hallita ennen niiden käyttämistä. Käytännön työssä käsiteltiin prosesseja ja tutkimuksia, joita suoritettiin opinnäytetyössä, mutta se osio pysyy salaisena. Työ koostui suurimmaksi osaksi liuosten valmistamisesta ja niiden optimoinnista koutatun kalvon tuloksien mukaan.

Asiasanat: anti-hyönteis, sol-gel-prosessi, kouttaus, permetriini

CONTENTS

TERMINOLGY

HCl: Hydrochloric acid is a colorless highly corrosive compound.

Permethrin: A synthetic chemical widely used in agriculture and as an insecticide.

TEOS: Tetraethoxysilane is a chemical compoud.

SEM: Scanning electron microscope is a characterization technique with magnification of 10 000 times.

Sol-gel process: A technique to apply thin film of a few nanometres, for example on fabrics.

Solubility: The ability of a substance to dissolve.

UPC: Universitat Politècnica de Catalunya

Wash fastness: The ability for textiles to retain a certain compound during washing.

1 INTRODUCTION

In the world today medicine is a highly developed area but it does not provide drugs to all diseases, for example the ones from insect-borne infectious diseases. Therefore other measures should be brought into use. One possibility is to treat fabrics with chemicals against insects and use them as a protection. In countries where this problem occurs for example nets are worn above beds preventing insects from reaching the skin but it is not completely safe since the insect might find gaps or holes in the net. When the net is treated with chemicals harmful for insects, they are mostly killed before managing to find a way through it. (Rodie 2011.)

Permethrin is a chemical used in agriculture and as an insecticide. It has low toxicity for mammals and therefore it can be used to impregnate fabrics. The World Health Organisation has published the maximum amount of permethrin which is recommended on a surface of a textile. This should be taken into account if wanting to treat fabrics with permethrin. Three different methods to impregnate fabrics with permethrin have been developed, from which the most widely used one include of dipping and spraying. (Faulde, Uedelhoven & Robbins 2003.)

The objective of this project was to optimize anti-mosquito coating with the sol-gel process for cotton fabrics. It was done in the Textile and Paper Department of UPC. All the needed chemical reagents and equipment was provided by UPC. Before the project could begin, it was required to master the process. Therefore general information about the sol-gel process and coating textiles was studied.

Fabrics are coated to give them new properties, change their appearance or improve their physical properties. It is a difficult process since the fabric loses its aesthetics and properties easily and therefore it is crucial to work with suitable methods and conditions. The sol-gel process is a wet-chemical technique which allows having a film only a few nanometers thin and transparent. Basically the process consists of hydrolysis, condensation, gelation, ageing and drying. Many factors can affect the preparation of sol-gel and the film prepared from it. It is essential to discover which conditions and methods are optimal to have the best possible film.

This project is focused on fixing sol-gel solutions and improving them based on the results. In the process itself attention is paid to quantities of chemicals, ways to fix solutions, and curing conditions. Padded samples are examined with different experiments. Magnification of the film, tests on crease recovery and stiffness of the fabric, extraction of permethrin and wash fastness are performed to discover the effects and stability of the coating.

The work is begun by preparing an experimental sol-gel solution to know how the coating behaves on a cotton fabric. Based on the results, different solutions with different curing conditions are fixed and optimized. When acceptable results are received, experiments should be performed to know whether the physical properties of the fabric have changed drastically. They should be taken into account in the sol-gel process since it is an important part of the optimizing.

2 GENERAL INFORMATION

To work in this field and understand the topic of the thesis, it is necessary to know the basics of coating textiles and the sol-gel process. Coated textiles can be considered as technical textiles, which are an important and quickly developing area and they are utilized in many fields. They are used in everyday life, extreme conditions, vehicles, buildings, protecting people et cetera. New ideas are constantly invented and, at the same time, existing ones are improved.

2.1 Coating textiles

Coating means a covering on a fabric which is applied on the surface with a certain process giving new properties, changing the appearance or improving the physical properties. This can be for example improving abrasion or giving resistance to dust, liquids and gases. Probably the most widely known coated fabric is a waterproof jacket which is availaible for everyone and it is worn as a protection from the rain. There are many different examples of protective clothing which are used in many areas: for example policemen, firemen and astronauts wear highly developed technical textiles. Nowadays, it is common to have different coated fabrics in homes and in daily usage. Most sportswear and clothes for different weather conditions are coated, and they are widely used every day. (Fung 2002, ix.)

In the making of technical textiles, production is mainly limited to processing with a viscous liquid. The basic method with liquid is to dip a substrate into it or spread it on top of the surface. After this, the substrate goes through a drying or curing process, which allows the coating to harden and finish the technical textile. (Horrocks & Anand 2000, 173.)

According to Fung (2002, 4) the most basic method to coat a textile is the direct spreading of a polymer. Usually it is in the form of a thick liquid or paste and it is applied on the textile with a sharpened length of a metal called a doctor blade or knife. In a coating machine a fabric, to which a blade is used on, is held flat by the application of tension. The fluid is spread over the fabric by putting it in front of the doctor blade and moving the fabric constantly forward into a heated oven. The thickened liquid is also known as compound or resin. Fung explains (2002, 4) it is a mixture of the polymer and other ingredients which give certain properties or assist the spreading process. It can be either water based or solvent based. When water or solvent is evaporated, it leaves a polymer deposit on the fabric forming a compact layer. (Fung 2002,4.)

Coating textiles is complicated because when a film is applied, fabric loses its aesthetics and properties easily. Even with one layer it is difficult and problems might occur when the total weight of the fabric is higher and the nature of the surface has changed considerably. Therefore finding the right methods and conditions is essential for the coating process. (Fung 2002, 84.)

2.2 Sol-gel process

The sol-gel process is a wet-chemical technique discovered in the late 1800s but not until in the mid 1980s real interest in this process was developed. During this decade, it was discovered that under acid conditions the hydrolysis of tetraethyl orthosilicate (TEOS) led to the formation of SiO_2 particles. It is widely used in the fields of materials science and ceramic engineering. The sol is applied to different types of surfaces with industrial processes like dip coating. The film will be only a few nanometers thin and transparent, which is a huge improvement compared to other traditional coating methods. (Hench & West 1990, 33, Nanowerk 2011, Wikipedia 2011g.)

Basically, in the sol-gel process nanoparticles (colloids) are formed from the silanes with chemical reactions in a solution. Depending on the type of sol fixed, the solution evaporates in room temperature or when it is heated. On Nanowerks (2011) website, they explain that the sol becomes a viscous gel when the particles concatenate into a dense web due to their high reactivity. When it has dried out, it forms a compact layer. Mahltig and Textor (2008, 1) explains that a film of nanosol particles can be from a few nanometers up to 100 nm thick when a coating from nanosols can have a thickness of up to several hundred nanometers. Because the basic nanosols can be widely modified, it can lead to new functionalities which can be applied on various surfaces. Table 1 shows examples of what can be improved in textiles which have a sol-gel coating. (Mahltig & Textor 2008, 1, Nanowerk 2011, Wikepedia 2011g.)

TABLE 1. Properties improved with sol-gel coatings (Mahltig & Textor 2008, 2)

sol-gel coatings on textiles				
textile properties	**surface properties**	**optical properties**	**(bio-)active systems**	**further properties**
-stiffness/drape	-hydrophobilicity	-colour	-biocidal coatings	-heat resistance
-handle	-hydro-/ oleophobicity	-photochromic effects	-controlled release systems	-magnetic properties
-absorbency	-abrasion stability	-UV-absorption	-therapeutic systems	-conductivity
-permeability	-(photo-)catalytic activity		-immobilisation of biological materials (e.g. enzymes, cells)	-odour management
-felt-free	-barrier functions			

The process consists of five different phases: hydrolysis, condensation (Figure 1), gelation, ageing and drying. Many factors can influence the rate of hydrolysis and condensation reactions, such as pH, nature and concentration of catalyst, H_2O/Si molar ratio, temperature et cetera which is related to the characteristics and properties of a particular sol-gel network. (Brinker & Scherer 1990, 2-10, Mahltig & Textor 2008, 2, Sol-Gel Chemistry 2011.)

The process begins with the preparation of the sol. Sol, also known as solution, is a colloidal suspension in a liquid. Steiner and Walker (2011) explain that nanoparticles dispersed agglomerate together to form a continuous three-dimensional network. Sol is different from non-colloidal liquid in that it has the possibility to precipitate out the dispersed nanoparticles, which is not possible with non-colloidal liquid. In hydrolysis, inorganic or metal-organic precursors are used. Metal alkoxides are typically utilized as precursors because of their property to react readily with water. Hydrolysis can be done under acidic or alkaline conditions. Mahltig and Textor (2008, 2) are able to explain that nanosols often have weakly cross-linked condensation products if they are hydrolyzed under acidic conditions but catalyzing in alkaline conditions tends to make the sol's particles to aggregate with larger pores. In order to have the gel hydrolyzed, the precursors go through polycondensation in order to form inorganic polymers.

This network contains a liquid phase. (Mahltig & Textor 2008, 1-2, Sol-Gel Chemistry 2011, Steiner & Walker 2011, Wikipedia 2011g.)

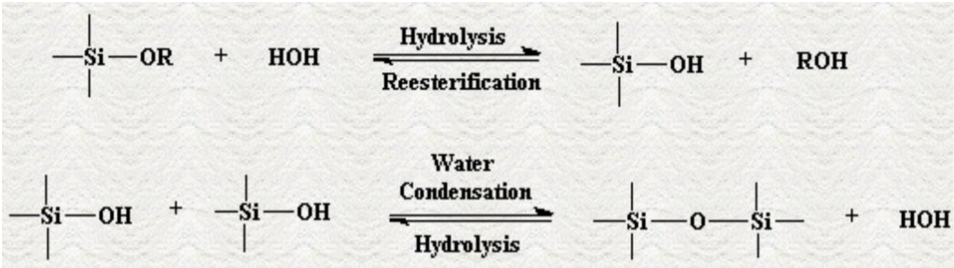

FIGURE 1. Hydrolysis and condensation in sol-gel process (Sol-Gel Chemistry 2011)

The next step is ageing in which polycondensation reactions continue. The ageing process can influence different properties of the resulting coatings, for example it can affect the coating's thickness, roughness and porosity. The gel is applied on the wanted surface, after which the curing begins. Depending on the material and the results wanted, temperature and time is chosen. Water and other liquids are evaporated during drying, and the gel forms a compact layer on the surface. The whole process is shown in Figure 2. (Mahltig & Textor, 2008, 4, Nanowerk 2011.)

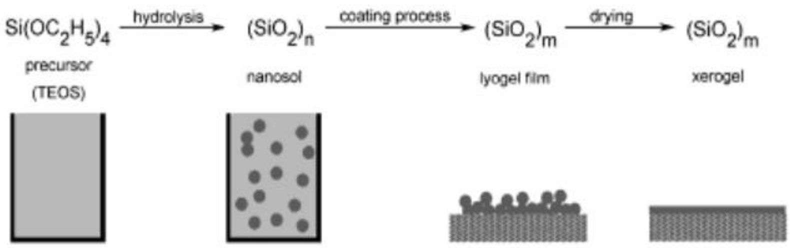

FIGURE 2. Sol-gel process with TEOS as precursor (Mahltig & Textor, 2008, 3)

2.3 Textiles with anti-mosquito properties

Textiles with anti-mosquito properties are considered to be an increasingly important component in the fight to reduce the incidence of insect-borne infectious diseases such as malaria, West Nile virus, encephalitis, dengue fever, Lyme disease et cetera. Rodie (2011) has come across with the information that these diseases afflict hundreds of millions of people each year and represent a significant portion of overall infectious diseases. This is ranked globally as second among all causes of death. Many of these diseases require vaccines and therapeutic drugs, which still need to be developed and therefore measures to protect people from these insects have to be taken in use. As one solution, permethrin-based insect repellent treatment is permanently bonded to fabrics for durable protection from mosquitoes and other disease-bearing, biting insects. (Rodie 2011.)

This chemical has been registered for textile as well as agricultural, pharmaceutical or other pest-control applications by the US Environmental Protection Agency (EPA). They have also considered permethrin-treated clothing to be safe for wearing by children and pregnant or lactating women. Clothes, nets and camping gear should be treated with permethrin to protect from mosquitoes and disease-bearing insects. (Rodie 2011.)

Three methods have been developed to impregnate fabrics with permethrin. One is to treat fabrics by dipping and spraying which allows permethrin to absorb onto the surface of the fibers. This is the most widely used method. The second one is to treat wool or silk fibers using solutions under heat and salt gradients to bind permethrin into the fibers. It prevents infestation and damage of woolen or silk fabrics from clothes moths and keratin-eating beetles. The last method is the specific polymerization of permethrin onto the fiber surface, which is called polymer-coating method. Fabrics are treated during production in a factory and are thereby ready to use. (Faulde, Uedelhoven & Robbins 2003.)

3 MATERIALS

In this project it was required to use a textile substrate and different chemical reagents. From them, the most important of all is permethrin because it is the active principle. This chapter gives general information of these chemical reagents and the textile substrate.

3.1 Textile substrate

Cotton is a natural fiber which grows in a cotton plant. It is one of the oldest and most widely used natural fibers known in our time. The first traces of cotton is from 7 000 years ago found in archaeological sites. When cotton is harvested it needs to be combed in order to remove the seeds. Usually the fiber is spun into yarn or treated and used to make textiles. One single fiber is rather weak but a strong and smooth thread is formed when multiple curling fibers are straightened and twisted together. This can be knitted, woven and even dyed. Cotton can be also blended with other natural and synthetic fibers to give particular properties or to add texture and strength to the fiber. Cotton is soft, comfortable, strong and durable, it absorbs easily, and has good color retention and wrinkles readily. The fibers are composed of five concentric layers. It has a Z-spiral winding in each fiber. With a lower magnification it is possible to witness the spiral the fiber has as in Figure 3. The width of a fiber is approximately 12 µm (Figure 3). (Bryk 2011, Cotton Australia 2011, Wikepedia 2011a.)

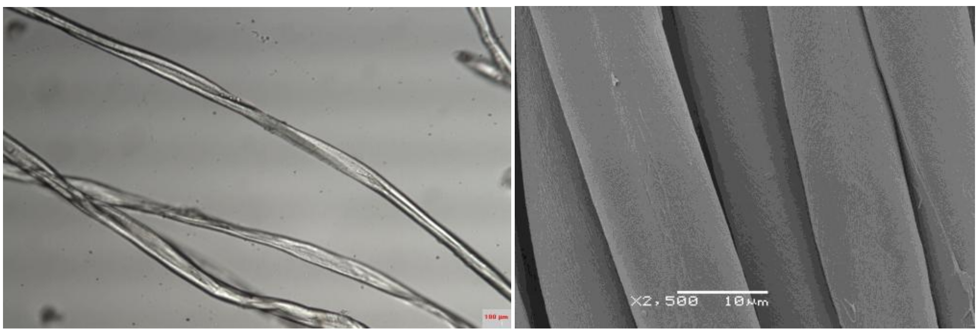

FIGURE 3. Cotton fibre

100 % cotton fabric was the textile substrate of this project. Characterization was performed to ensure the fabric was indeed pure cotton. This can be found in the chapter of characterization of cotton.

3.2 Chemicals

3.2.1 Permethrin

Permethrin is the active principle in the project and therefore the most important substance. When working, it was required to consider the limitations and chemical properties of permethrin. It is a man-made synthetic pyrethroid, which was originally derived from the crushed dried flower of the daisy Chrysanthemum Cinerariifolium. It belongs to synthetic pyrethroids, which are listed as one of the oldest classes of organic insecticides. Permethrin acts as a neurotoxin and when insects are exposed to it, it paralyzes the nervous system. It is extremely toxic to cold-blooded animals, for example insects and fish, but it is not found to have high toxicity for warm-blooded animals like mammals or birds. Permethrin functions in two different ways, either as a stomach poison when it is swallowed by insects or as a contact poison when insects are exposed to a direct contact. (Pesticide Management Education Program 1993, Shah 2011, Wikipedia 2011e.)

Permethrin is widely used in agriculture as an insecticide. Mainly it is utilized to protect crops, kill livestock parasites and pests on different fruits and vegetables. Permethrin is also used for industrial insect control. As an insect repellent or insect screen, permethrin is used in timber treatment, as a personal protective measure and in pet collars. It can also be found in healthcare, for example to eradicate parasites like head lice. In daily lives it is used among others in greenhouses, home gardens and for termite control. Permethrin is sold among others in the form of dusts, concentrates and smokes. (Pesticide Management Education Program 1993, U.S. Environmental Protection Agency 2006, Wikipedia 2011e.)

United States Environmental Protection Agency (EPA) has classified permethrin as a human carcinogen because of studies where mice fed with permethrin developed tumors. Permethrin has not been found to be highly toxic to humans but excessive exposure might cause nausea, headache, muscle weakness, excessive salivation, shortness of breath and seizures. (U.S. Environmental Protection Agency 2006, Wikipedia 2011e.)

Because permethrin has low toxicity for mammals, it absorbs poorly through the skin and it is quickly inactivated by the body. Direct contact with eyes and skin should be avoided especially with not diluted permethrin. However, it can be used for example in protective clothing when taking into account how much permethrin is safe to have in the fabric without any harm to humans. Mainly it is found in military clothes or mosquito nets in tropical areas. The latter ones which are used to cover the bed, are treated with a solution containing permethrin in order to decrease the chance for parasitic insects to spread diseases. When the insects reach the net, they are killed before they are able to find gaps or holes. In tropical areas where for example malaria is a huge problem, these types of clothing or nets are extremely important to prevent the spreading of diseases. (Pesticide Management Education Program 1993, U.S. Environmental Protection Agency 2006, Wikipedia 2011e.)

Permethrin is a synthetic chemical. It has many different trade names including Ambush, BW-21-Z, Cellutec, Ectiban, Eksmin, Exmin, FMC-33297, Indothrin, Kafil and Kestril. Permethrin's chemical formula is $C_{21}H_{20}Cl_2O_3$. It has overall four stereoisomers, of which two are enantiomeric pairs. Permethrin is a viscous-liquid which sometimes tends to crystallise at room temperature. This substance is stable and incompatible with strong oxidizing agents. All the properties of permethrin given to me by Ardanuy (2011) are available in Figure 4-5 and in Table 2. (Pesticide Management Education Program 1993.)

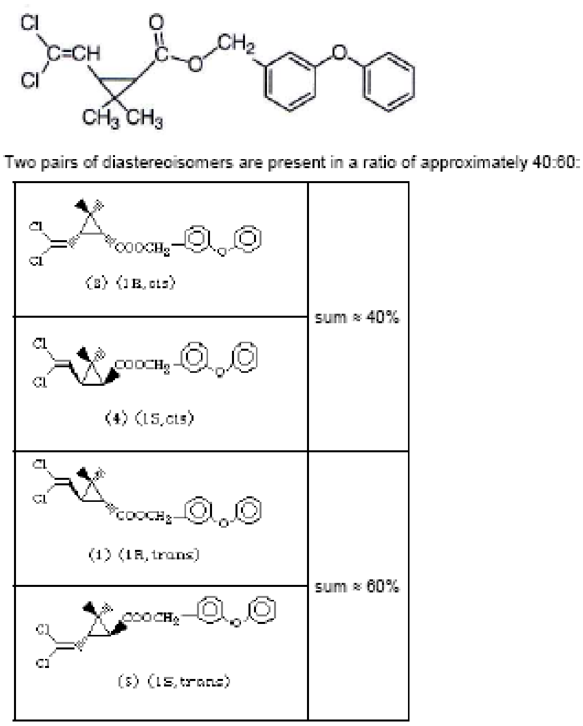

FIGURE 4. Permethrin (Ardanuy 2011)

TABLE 2. Properties of permethrin (Ardanuy 2011)

Recommended maximum on a surface of a textile [mg/m^2] (WHO)	500
Appearance	Liquid
Molecular formula	$C_{21}H_{20}Cl_2O_3$
Molecular mass [g/mol]	391.3
Melting point [ºc]	34-35, cis-isomers (63-65), trans-isomers (44-47)
Boiling point [ºc]	220-290
Temperature of decomposition [ºc]	252
Flash point [ºc]	100-131
Bulk density/specific gravity [g/cm^3]	1.29
LogP: Octanol-water partition coefficient	6.1
Solubility in water and in organic solvents [mg/L] at 20ºC	
Water	0.006
Acetone	>450000
Methanol	258000
p-xylene	1000000
Hexane	1000000
Vapour pressure at 25^{o}C [mPa]	0.002

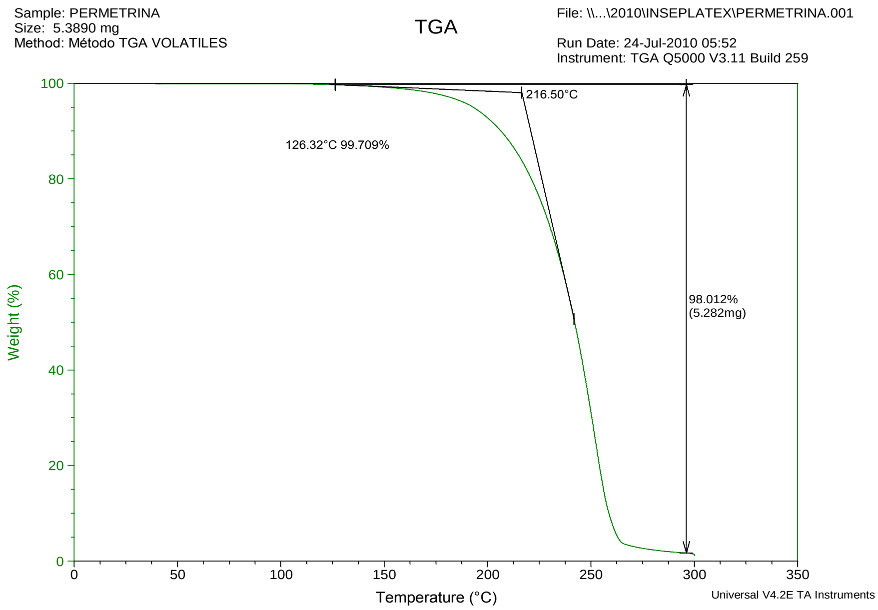

FIGURE 5. Thermogravimetry of permethrin (Ardanuy 2011)

3.2.2 Tetraethoxysilane

TEOS, tetraethoxysilane, also know as tetraethyl orthosilicate, is a chemical compound, whose formula is $C_8H_{20}O_4Si$. Information and properties of TEOS can be found in Table 3. This compound is generally used as a precursor to silicon dioxide and as a cross linking agent in silicone polymers. The odor of TEOS is irritating to the eyes and nose. When it is in contact with skin or eyes, it can cause severe irritation or burns. (National Center for Biotechnology Information 2004d, Wikipedia 2011h.)

TABLE 3. Properties of TEOS (National Center for Biotechnology Information 2004d)

Molecular Formula	$C_8H_{20}O_4Si$
Molecular Weight [g/mol]	208.3
Boiling Point [ºC]	168.8
Flash Point [ºC]	45
Density [g/cm^2]	0.94
Appearance	Transparent liquid
H-Bond Donor	0
H-Bond Acceptor	4
Rotatable Bond Count	8
Exact Mass	208.11
MonoIsotopic Mass	208.11
Topological Polar Surface Area	36.9
Heave Atom Count	13
Complexity	91.2

3.2.3 Ethanol and methanol

Ethanol and methanol are very similar to each other by their appearance and chemical properties. Both are alcohols and belong to the same chemical group. Ethanol has one more carbon atom and two hydrogens compared to methanol. (Wikipedia 2011b, Wikipedia 2011d.)

Methanol, also known as methyl alcohol, is the simplest aliphatic alcohol and it is also the first member of the homologous series. It has a hydroxyl group -OH bonded to a carbon atom and therefore belongs to the group of chemical compounds with the same property. Methanol’s composition is liquid and it is transparent. Its molecular formula is CH_3OH (Table 4). It is extremely hydroscopic and miscible in water and organic solvents. It is very volatile and when burned its flame color is bright white. (National Center for Biotechnology Information 2004c, O’Leary 2000b.)

TABLE 4. Chemical properties of methanol (National Center for Biotechnology Information 2004c)

Molecular Formula	CH_3OH
Molecular Weight [g/mol]	32.04
Melting Point [^oC]	-97.8
Boiling Point [^oC]	64.7
Density in 25 ^oC [g/ml]	0.7866
Appearance	Transparent liquid

Methanol can be a dangerous chemical but it has many uses. It has a distinctive smell and a burning taste. It is a potent nerve poison and therefore toxic. It should never be ingested, inhaled or come in contact with skin. Methanol is an important industrial material and it is used among other things in the manufacture of formaldehyde, as an anti-freeze agent in car radiators, and as a solvent in paint and varnish industry. (O'Leary 2000b, Wikipedia 2011d.)

Ethanol, also known as ethyl alcohol, is also a colorless liquid and it is the second member of the aliphatic alcohol series. It is an alcohol and belongs to a group of chemical compounds whose molecules contain a hydroxyl group, -OH, which is bonded to a carbon atom. Ethanol's molecular formula is CH_3CH_2OH (Table 5). Under normal conditions ethanol is an extremely volatile and flammable liquid. When it is burned the color of the flame is bright blue. Its odor and taste is strong and characteristic. Ethanol is soluble in water. (National Center for Biotechnology Information 2004a, O'Leary 2000a, Shakhashiri 2009.)

TABLE 5. Chemical properties of ethanol (National Center for Biotechnology Information 2004a)

Molecular Formula	CH_3CH_2OH
Molecular Weight [g/mol]	46.07
Melting Point [^oC]	-114.1
Boiling Point [^oC]	78.5
Density in 20 ^oC [g/ml]	0.789
Appearance	Transparent liquid

Ethanol is used widely in many different areas. For example alcohol drinks and ethanoic acid are manufactured from it. Ethanol is also utilized to preserve biological specimens and used as a fuel, fluid in thermometers, solvent for paint, varnish and drugs. Ethanol is mainly used industrially as a mixture of 95 % ethanol and 5 % distilled water. There is also available pure ethanol but it is more expensive and therefore used only if absolutely required. Ethanol is toxic and when it has been drunk, the body begins to dispose of it immediately mainly by liver. (O'Leary 2000a, Shakhashiri 2009, Wikipedia 2011b.)

3.2.4 Hydrocholoric acid

Hydrochloric acid, HCl, is a colorless and odorless liquid which is an extremely corrosive compound. It is the aqueous solution of hydrogen chloride gas and its properties can be found in Table 6. HCl is a monoprotic acid and therefore can dissociate only once. There are seven common strong acids and from them HCl is the most unlikely to undergo an interfering oxidation-reduction reaction. HCl is not flammable and it is miscible in water. This acid is very corrosive to eyes, skin and mucous membranes. Also short- and long-term inhaling or acute oral exposure can give many different dangerous symptoms. HCl is among other things used in the production of chlorides, fertilizers, dyes and in textile and rubber industries. (National Center for Biotechnology Information 2004b, Princeton University 2006, Wikipedia 2011c.)

TABLE 6. Properties of hydrochloric acid (National Center for Biotechnology Information 2004b)

Molecular Formula	HCl
Molecular Weight [g/mol]	36.47
Melting Point [°C]	-114.22
Boiling Point [°C] at 760 mm Hg	-85.05
Density in [g/l]	1.639
Appearance	Transparent liquid

4 EXPERIMENTAL PROCEDURES AND TECHNIQUES

Different kinds of equipment were used in this project and therefore it was necessary to study them first. It was important to master them because these machines are very sensitive and can be damaged easily with wrong usage.

4.1 Experimental procedures

4.1.1 Padding machine

Padding machines are widely used in paper and textile industry and in order to coat textiles, this machine is required. It allows getting the excess solution out of samples and making it spread evenly on the fabric. Samples, which have a chemical solution in them, are inserted between the rolls in the machine. The speed (m/min) of the lower roll is adjustable and the upper roll is lowered on top of it so that both move. The pressure (kg/cm^2) in the padding comes from the upper roll which can be adjusted. The one available for usage in UPC was an industrial machine (Figure 6).

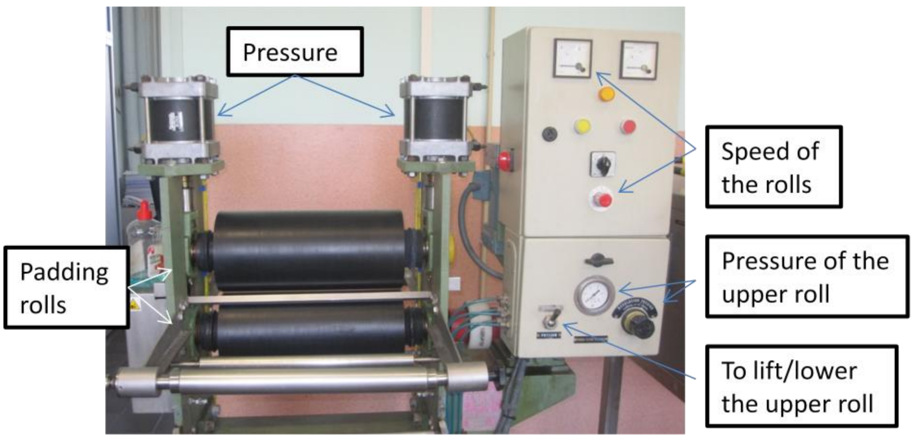

FIGURE 6. Padding machine

A padding machine has strict instructions on what must be done before and after using this equipment in order not to damage it. When a sample is inserted between the rolls, you must be careful not to put fingers near them and you must insert the sample under a safety metal bar. The sample can be taken off immediately after use from the other side. The rolls should be cleaned immediately with extreme caution. When washing, they should be rolling towards the cleaner preventing the fingers getting in between the rolls. In order to handle the rolls gently, a soft sponge should be used for washing and a towel for drying.

4.1.2 Drying machine

After textiles have gone through a padding machine, they have to be dried. One machine used to achieve this is a drying machine (Figure 7). These machines are widely used in textile industry and due to this, it was better to work with an industrial machine to have similar conditions as in a real textile company. This equipment allows adjusting the temperature (°C) and time (s/min). Both of these can be done next to their meter. The machine uses air in drying instead of the normal oven. When the machine is adjusted to the wanted temperature and certain timing, the sample is placed in a metal frame which allows it to stay straight and be stretched. When the sample is in the metal frame and about to be inserted into the machine, the safety bar has to be put down in order to prevent any injuries since the frame is pushed out with high speed from the machine when the drying is finished.

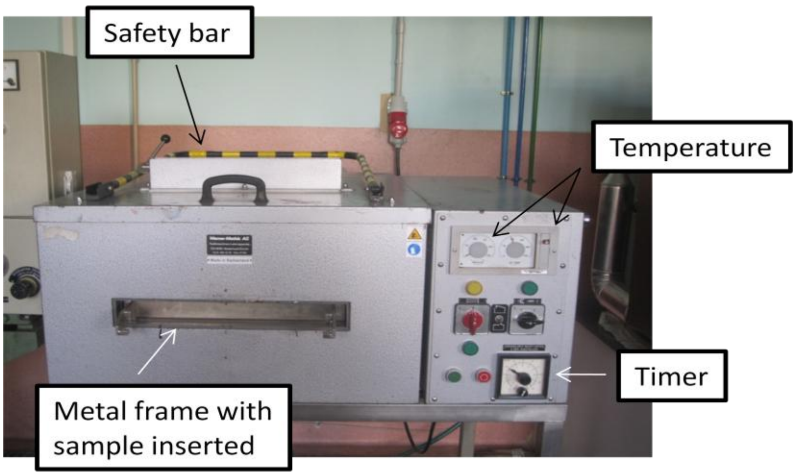

FIGURE 7. Drying machine

4.1.3 Oven for curing

The drying machine alone is not enough for the process and therefore an oven is required for drying and curing samples for longer times. The oven (Figure 8) has the possibility to adjust the temperature (ºC) and timer (min/h) for required conditions. The temperature is set by pressing the button for setting.

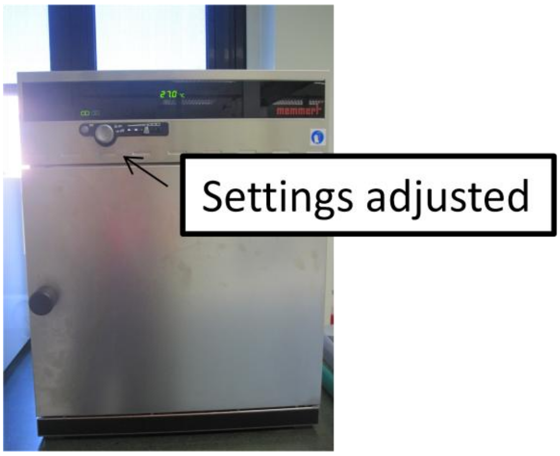

FIGURE 8. Oven for curing

4.1.4 Vibromatic

A vibromatic (Figure 9) is used to stir solutions with certain speed (U/min) and time (min/∞). The maximum number of samples is eight at a time. The samples are placed in a metal bar which goes through the machine. It functions by turning slightly the metal bar to which the solutions are attached. It is possible to adjust the speed how fast this metal bars keeps turning and for how long. When solutions are stirred in this machine, it is necessary that the bottles have their corks on them; otherwise some of the solution might get spilled. Also, it is advisable to check the speed so that it is not too high because otherwise some of the solution might get spilled even though it has a cork on it. A vibromatic has eight places to stir solutions and therefore it is the most useful for mixing if many solutions require stirring at the same time.

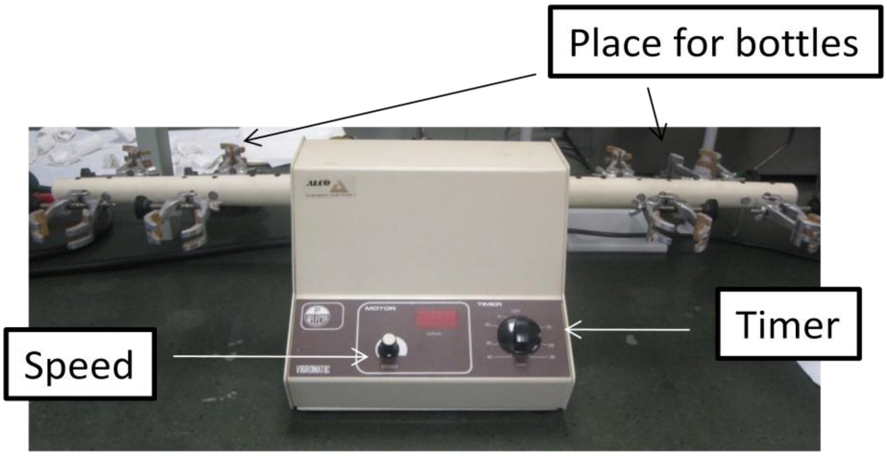

FIGURE 9. Vibromatic

4.1.5 Ultrasonic bath

An ultasonic bath (Figure 10) is mainly utilized in cleaning of delicate items but can be used to stir solutions. The latter function is the reason why it was needed in this project. It is a bath usually filled with water. Bottles are placed on top of a metal net which is inside the bath. It is possible to adjust time (min/∞) and temperature (ºC) for stirring. An ultrasonic bath functions by using high frequency sound waves (20-400 kHz). A glass is placed inside the bath, after which the conditions are chosen. The machine begins working when the time for stirring is chosen. In an ultra bath it is possible to have many solutions in it at the same time, depending on their size.

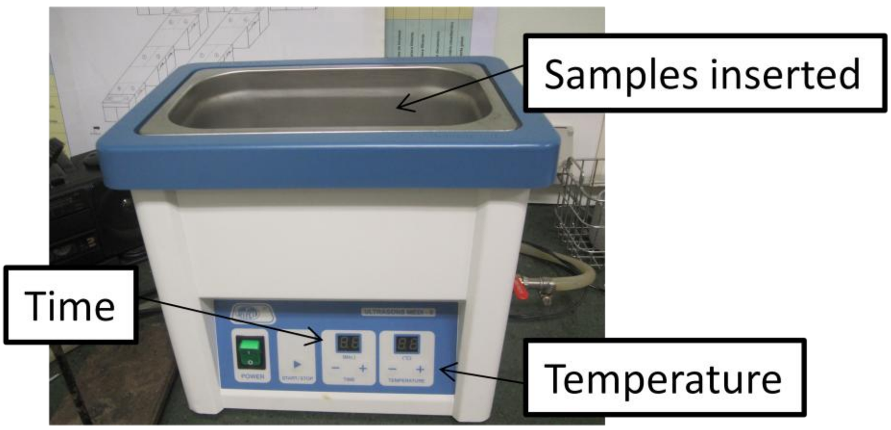

FIGURE 10. Ultrasonic bath

4.1.6 Magnetic stirrer

A vibromatic and ultrasonic bath are not the only machines to stir solutions, there are also magnetic stirrers. The one operated in this project was MicroMagMix (Figure 11). The speed (Rpm) and temperature (°C) are adjustable. When this machine is used, a magnet is put into a glass containing the solution and the glass placed on top of the machine. The machine has a magnet in the middle of it which forces the magnet inside the solution to move with a certain speed making it stirr the solution. The glass with the solution has to be positioned in the middle in order to have the magnet in the right place. Speed and temperature is chosen as wanted and the stirring can begin. With magnetic stirrer it is possible to work with only one solution at a time, which makes it the most unpractical choice.

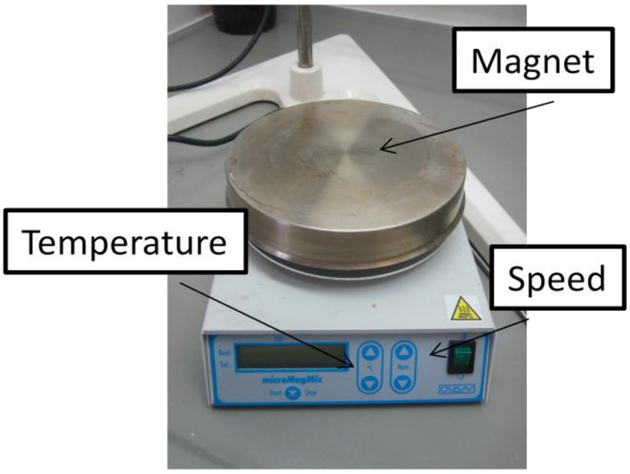

FIGURE 11. Magnetic stirrer

4.1.7 System for continuous extraction

In the project, permethrin had to be extracted continuously from samples and therefore a system for continuous extraction was required. Basically a soxhlet extraction system is for extracting compounds with low solubility from solid mixtures. In this machine (Figure 12) the maximum of soxhlets is four at a time. The temperature is adjusted from the bottom in front of the machine.

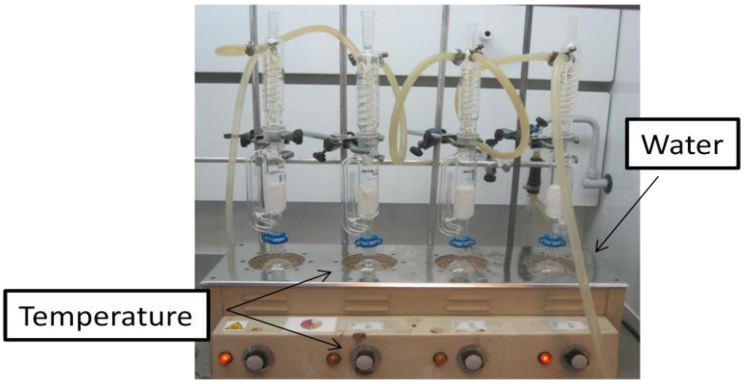

FIGURE 12. Soxhlet extraction

The soxhlet (Figure 13) has three main parts: condenser, “soxhlet” extractor and a round bottomed flask. The upper part is called the condenser, to which the water pipe goes where cold water is circulating. Below the condenser is the soxhlet extractor where a sample is placed. The final part of the soxhlet is the round bottomed flask. Inside the flask the extracting solvent is inserted, which is heated throughout the process. The solvent evaporates and in condenser it comes in contact with cold water which makes it condensate back to liquid and fall into the extractor. The sample, from which a component is extracted, is held in the extractor. The condensated solvent drops continuously into the extractor washing the sample and extracting the desired compound. When the surface is high enough, the liquid with the extracted compound falls inside the flask. This system is continuous and has to be run many times in order to extract the whole component.

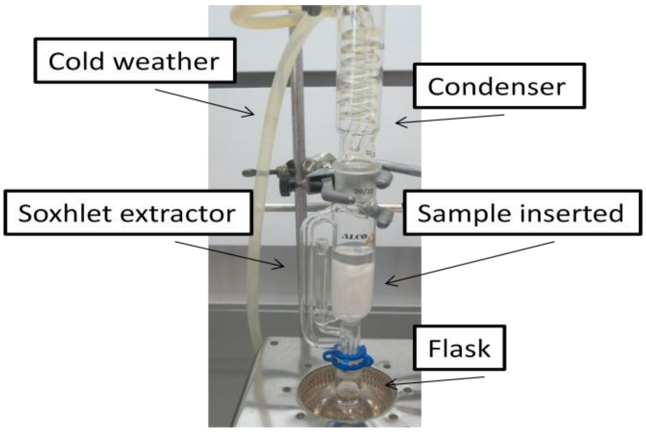

FIGURE 13. Soxhlet

4.1.8 PH-meter

A pH-meter (Figure 14) is used to measure the pH of a solution. The machine itself has an electronic meter in it and a special measuring probe, a glass electrode, which is connected to it. When beginning to operate the pH-meter, the machine should be calibrated. This is done by using standard solutions with pH 4 and 7. First the electrode needs to be cleaned with distilled water and dried gently after which it is inserted in the solution with pH 7. It is advisable to shake it while the meter is measuring the pH. When this is finished, the electrode is washed again with distilled water and dried. The next step is to insert it inside the solution with pH 4 and wait until the display shows the number 000 which means the calibration is done.

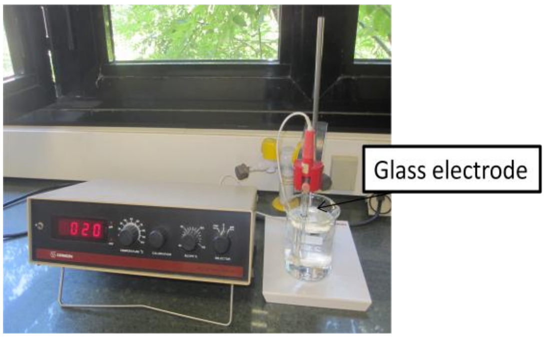

FIGURE 14. PH meter

4.1.9 Dessicator

Desiccators (Figure 15) are sealable enclosures made of heavy glass which prevents humidity getting into samples. It has desiccants which absorb all excess moisture. The color of desiccants is blue but when they have absorbed humidity they begin to turn white. When they appear not to have a blue color anymore, they should be changed or dried in order for the desiccator to function. Opening a desiccator exposes samples to the humidity in the room which has to be taken into account when working with desiccators.

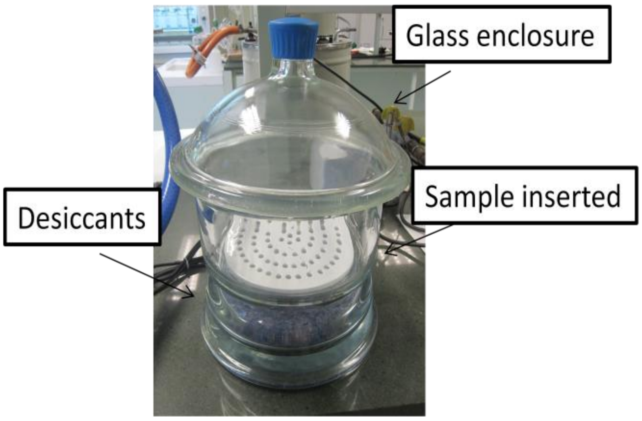

FIGURE 15. Desiccator

4.1.10 Linitest

A linitest (Figure 16) in this kind of work is utilized to know the wash fastness of coated fabrics. There are capsules which are inserted inside the device. The washing temperature (°C) is adjustable. The cork has a seal on it to make sure the capsule is completely sealed. A sample is placed inside the capsule with water and reference detergent. It is essential to seal and place it properly in the washing machine. Linitest allows working with many capsules at the same time.

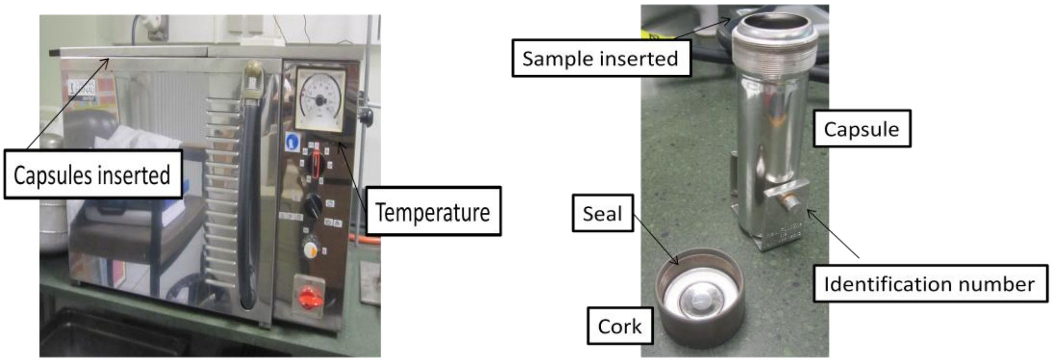

FIGURE 16. Linitest and a capsule

4.2 Characterization techniques

4.2.1 Scanning electron microscope

In the project it was necessary to know how the film is on the fibers and therefore a scanning electron microscope (Figure 17) was required. It is also known as SEM and it is a characterization technique with very high resolution. It has a possibility of high magnification of x 10 000. An SEM is attached to a computer and on the screen is shown the part of a sample where the microscope is aimed. The magnification is adjusted manually in front of the device. The samples are placed inside the device on top of a small metal weight.

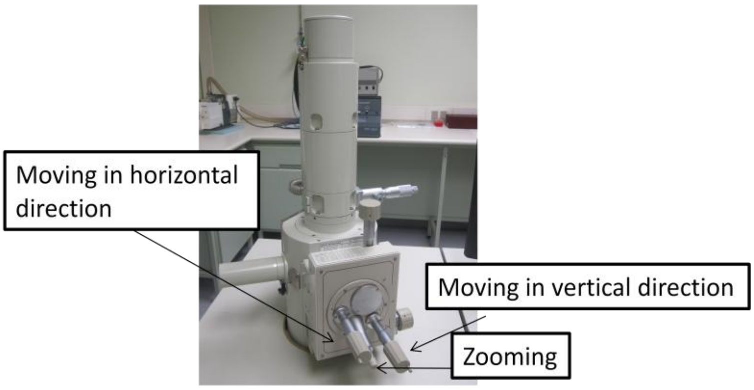

FIGURE 17. Scanning electron microscope

This machine functions by scanning a sample with a high-energy beam of electrons in a raster scan pattern. The sample's atoms interact with the SEM's electrons giving signals with information of the sample's surface topography, composition and other properties. There are different signals with different electrons: secondary electrons, back-scattered electrons, characteristic X-rays, light specimen current and transmitted electrons. Usually a single machine does not possess all possible signals but secondary electron detectors are typical in all SEMs. Before inserting samples inside the machine they should be cut to the size of a metal weight. They are gently glued on the metal weigh in order to prevent them from moving. Each metal weight is identified by numbers. (Wikipedia, 2011f.)

4.2.2 Ultraviolet and Visible spectrophotometer

To determine the amount of extracted permethrin in an extraction solution, it was required to have an Ultraviolet and Visible (UV Vis) spectrophotometer (Figure 18). The name refers to its two light sources, ultraviolet and visible light. It functions by measuring the intensity of light before passing through a sample and comparing it to the intensity of light after passing through the same sample. This ratio is called transmittance. Typically this machine is utilized to determine the quantity of a colorless substance in a solution. Ultraviolet is in the range from 200 to 400 nm and visible light from 400 to 800 nm. (Dios 2011, Intertek Plastics Technology Laboratories 2011, Wikipedia 2011i.)

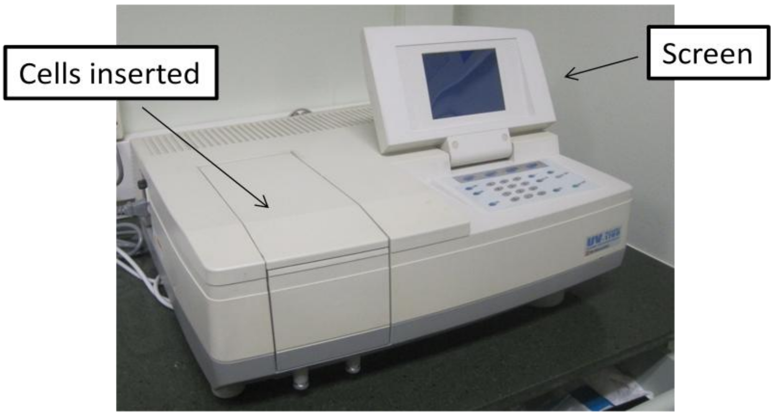

FIGURE 18. Ultraviolet and Visible spectrophotometer

The law on which this system is based on is Beer-Lambert Law. A prism or diffraction grading separates a beam of visible or ultraviolet light into its component wavelengths. Using a half-mirrored device each wavelength is split into two equal intensity beams. One beam, reference beam, is guided to go through the cell with a reference sample while the other beam, sample beam, goes through the cell with a solution being measured. The intensities of lights are measured by electronic detectors and compared. Figure 19 demonstrates how a UV Vis spectrophotometer functions. (Dios 2011, Wikipedia 2011i.)

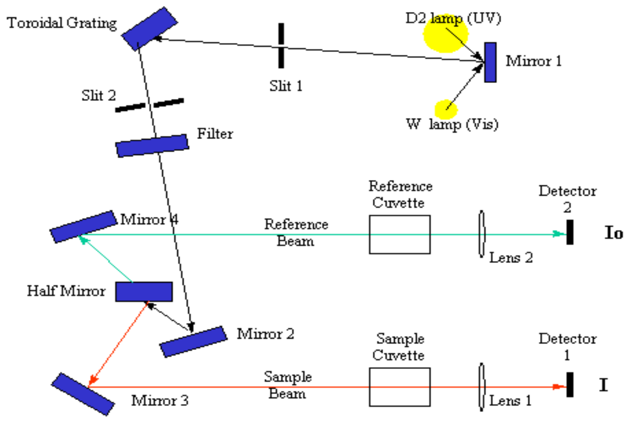

FIGURE 19. Function of UV-Vis Spectrophotometer (Dios 2011)

When this machine is operated, first a solution with known concentration is prepared. The needed amount is inserted inside the sample cell and in the other identical cell, which is used as a reference, the same solution is inserted but without the substance of which information is wanted. The cells are placed into the machine. The UV-Vis spectrophotometer makes a spectrum from these results. It is possible to make a calibration curve with many samples when their concentrations are known. The curve is based on their absorbance in a certain wavelength. Using this, an unknown concentration can be determined based on its absorbance.

Cells which are used in the measurement depend on the solution. A typical size is 10 mm but it can be even 1 mm. Cells have to be transparent in order for the lights to pass through it. Because permethrin absorbs light in the range of 200 – 400 nm it requires cells made of quarz.

4.2.3 Shirley stiffness tester

With a Shirley stiffness tester (Figure 20) it is possible to characterize a fabric sample’s bending height, flexural rigidity and bending modulus. The tester has a mirror where a line is reflected which is used to know how far the specimen has been extended. From a fabric sample, a 25 mm x 200 mm rectangular strip is cut out. This strip is placed on a horizontal platform of the tester and it is extended in the direction of its length by a ruler on top of it. The increasing part begins to overhang and bend under its own mass. It is extended until the tip reaches a plane passing through the edge of the platform and inclined at an angle of 41.5 ° below the horizontal. At this point the bending length is read from the scale of the tester. From this value it is possible to calculate the mass and the unit area of the fabric, the flexural rigidity and the bending modulus. When making the measures it is advisable to make at least three specimens on both lengthways and width ways direction of the fabric to get as accurate results as possible. With one strip, the measures are made on both sides and on both ends, getting overall four results. (Ardanuy 2011, Park 2006.)

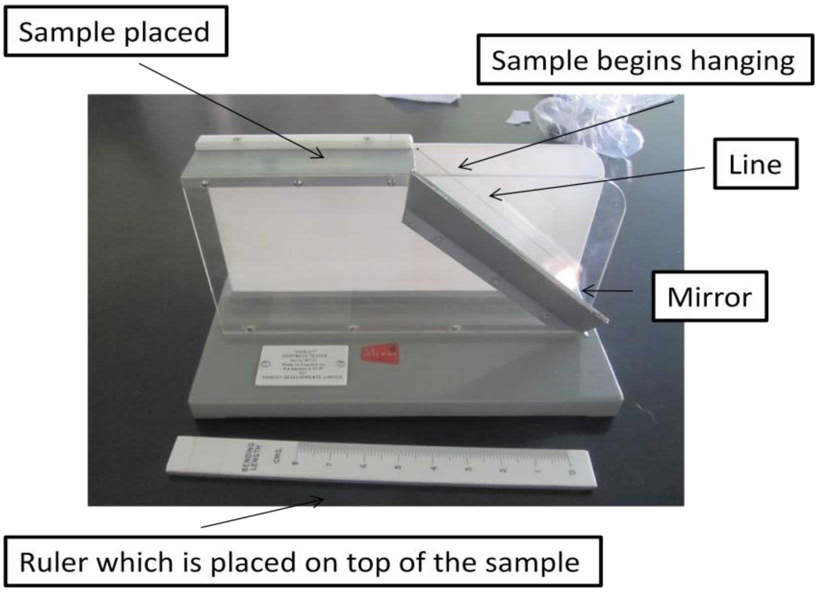

FIGURE 20. Shirley Stiffness Tester

In order to calculate the wanted information, it is necessary to use different formulas which are given in the instructions of the machine. Stiffness is calculated from the results of measuring the bending length. Before beginning the measurements, the weight of a sample has to be known in g/m². Stiffness has to be calculated separately for warp and weft direction. Calculations for this are found in Figure 21. To calculate the total stiffness of a sample, a formula (Figure 22) is required. In order to have the bending modulus (kg/cm²) of a sample, the thickness of a sample has to be known. It should be measured at least from four different parts of the sample and the average thickness t can be counted. With this information, the bending modulus can be calculated (Figure 23). (Ardanuy 2011.)

$$S = 0.10 * \ p * c^3$$

FIGURE 21. S is stiffness (mg/cm), p is g/m^2 and c is the measurement for bending length (cm) (Ardanuy 2011)

$$S_{total} = \ \sqrt{S_{warp} \ + \ S_{weft}}$$

FIGURE 22. Sample’s total stiffness (Ardanuy 2011)

$$B = \ \frac{12}{10^6} * \ \frac{S_{total}}{t^3}$$

FIGURE 23. Formula to calculate B, bending modulus of a sample (kg/cm^2), t is thickness (cm) (Ardanuy 2011)

4.2.4 Shirley crimp tester

After yarn is knitted or weaved, it gets a crimp and it is not possible to know the real length without stretching it into its original size. A Shirley crimp tester (Figure 24) allows calculating the actual length of a yarn by stretching it. The device has two clamps which are used to hold and stretch a yarn. Beneath one clamp there is a ruler to measure the length of the yarn. On the other side of the device there is a movable clamp with a weight attected to it. (Ardanuy 2011.)

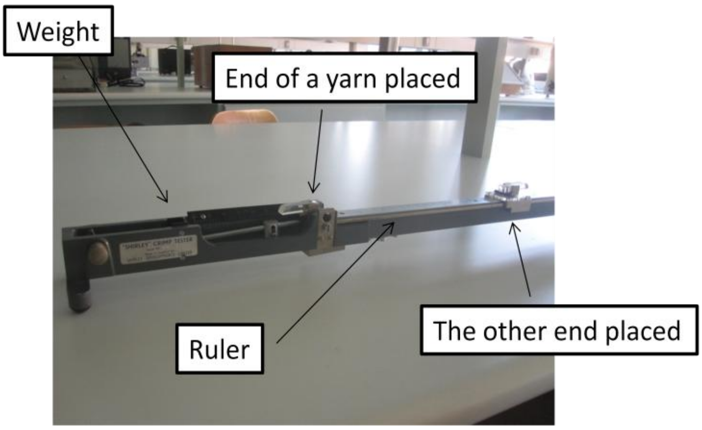

FIGURE 24. Shirley Crimp Tester

There are certain instructions how to place a yarn in this device. A yarn's end is placed in the clamp which does not move and the other end in the movable clamp. This device has a small weight, which can be adjusted, to function as resistance. The movable clamp is placed so that all required parts are aligned to have the measure of a yarn without its crimp. The result is read from the ruler which is under the small device. It is advisable to perform the measurement at least four times and calculate the average. (Ardanuy 2011.)

4.2.5 Shirley crease recovery tester

The crease recovery of a sample is measured with a Shirley crease recovery tester (Figure 25). This is performed based on ISO 2313. The machine has a rounded meter which has degrees to measure the crease recovery from 0 to 180^{o}. At least three specimens are cut from a sample in both warp and weft direction. The size of a specimen is cut based on a metal measurement with size 25 mm x 40 mm. This specimen is carefully folded into half and placed between two smooth glass plates. A weight of 1 kg is put on top of the plates and held there for five minutes after which the specimen is taken out with tweezers. It is directly moved to the clamp and allowed to recover from the crease for five minutes. The angle during this time is 90^{0}. When the time has passed, the part of the specimen which is hanging is moved to the same line as the metal piece on the bottom of the instrument. When they are aligned, the degree is read, which tells how much the specimen has recovered from the crease. (Ardanuy 2011.)

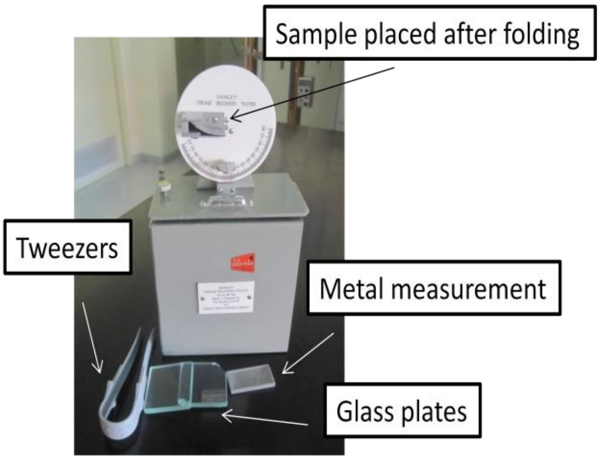

FIGURE 25. Shirley Crease Recovery Tester

5 CONCLUSIONS

This kind of research work is crucial in order to develop methods for preventing insect-borne diseases. There are ways to impregnate fabrics with anti-mosquito properties but they affect the physical properties of a textile. The sol-gel process, on the other hand, can produce film which is only a few nanometers thick film and retains these properties. Therefore the utilization of the sol-gel process enables the manufacturing of comfortable and safe products for protection from insect-borne diseases.

It was hard to begin the project without knowing how the sol-gel will behave and function, how much chemicals should be used and if they would even be soluble in these solutions, which conditions are the best for padding and curing, et cetera. There was much to be considered and during this project there was not enough time to improve every aspect and see how it affects the results. However, a satisfactory sol-gel which had a good film was found. Based on these experiments, it is easy to continue the work.

In this work different aspects were examined. In the beginning, the chemicals solubility of the chemicals was tested and during the project it was found out how much it is necessary to use chemicals in order to have a satisfactory film with permethrin in it. It was important to take magnifications from the films, in order to see whether they had spread evenly on the fibers, forming a compact film. It is essential to retain the textile properties of cotton with the coating. The most important aspect is to have permethrin in the fabric but stay within the limit which is given by the World Health Organisation. All these were examined in this research work but the results stay classified.

5.1 Reaching the objective

Overall, the work itself was a success and the objective of this thesis was reached. Many problems were encountered when it came to fixing solutions, performing experiments or having satisfactory results. By solving these problems, it was possible to have better results from the coatings and the process itself. All this produced a lot of knowledge about the sol-gel process and how a sol-gel film affects a fabric’s physical properties.

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