

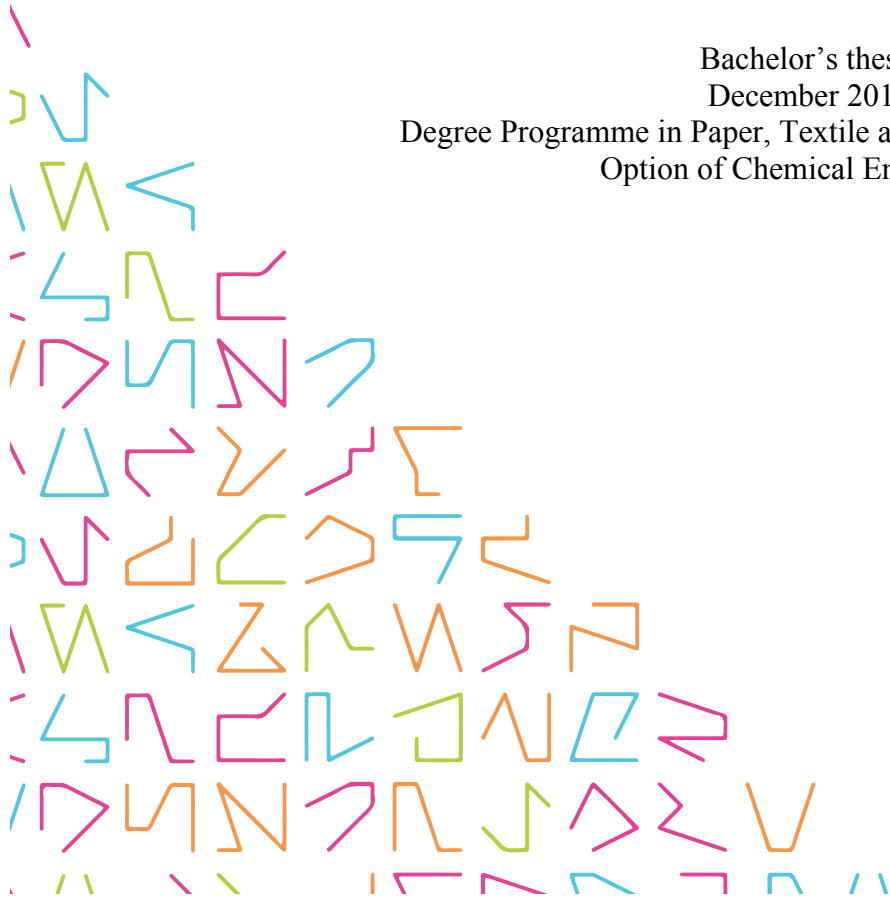


TAMPEREEN
AMMATTIKORKEAKOULU

SURFACE TREATMENTS OF NONWOVEN MATERIALS

Saana Saaristo

Bachelor's thesis
December 2016
Degree Programme in Paper, Textile and Chemical Engineering
Option of Chemical Engineering



ABSTRACT

Tampereen ammattikorkeakoulu
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SAANA SAARISTO
Surface Treatments of Nonwoven Materials

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The purpose of this thesis was to test an aerosol coating technique for nonwoven wet laid filter media. This thesis was done for Ahlstrom Tampere Corporation. Co-operating with Beneq Corporation the trials were set to test the nFOG™- equipment, the aerosol thin film coating equipment.

Ahlstrom Tampere Corporation wants to explore new techniques for chemical bonding and therefore in the theory part of this thesis the main focus is on alternative coating techniques. Special methods such as atomic layer deposition, liquid flame spray method and formation of self-assembled monolayers are introduced. These surface treatment methods are used for applications where uniform coating quality is requirement. From these techniques the liquid flame spray method could be suitable for nonwoven materials since it has been used for coating paper.

The empirical part consists of trials and testing trial materials. The trials were held in Ahlstrom's premises. The test results were positive. The equipment worked well even with high solid content binders and the coating quality was uniform.

Key words: nonwoven, filter media, chemical bonding, atomic layer deposition, liquid flame spray

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ABBREVIATIONS AND TERMS

Adsorption	Binding of molecules, or particles to a surface
Agglomeration	Particles attaching to other particles forming cluster
Anisotropic	Material that has unequal physical properties along different axes
Copolymer	Two different types of monomers are joined in the same polymer chain.
Disulphide	Two sulphides bonded together with covalent bond
HVAC	An abbreviation of the words heating, ventilation and air conditioning
HEPA	High efficiency particulate air filter
ULPA	Ultra low penetration air
Thermoplastic polymer	Reversible polymer
Latex	Liquid emulsion of small particles

1 INTRODUCTION

This thesis was made for Ahlstrom Tampere. Ahlstrom Tampere plant is part of the Ahlstrom Corporation, which is a leading producer of high performance fibre-based products. The plant in Tampere works in filtration business area, producing mainly wet laid nonwoven filter media. (About Ahlstrom. 2016.)

The aim of this thesis was to test new coating technique for wet laid nonwoven materials and more specifically to test equipment for applying binder chemicals. Beneq Corporation is the developer and producer of the test equipment.

Ahlstrom Tampere Corporation uses chemical bonding in their process and wants to explore new techniques for chemical bonding. Because of a wide product range variety of the used binders and other additives is also wide. Some of the binders and flame retardant chemicals have higher viscosity and solid content and therefore their behaviour in the test equipment is of interest.

The theory part starts with company presentations in chapter 2. In chapter 3 the basic nonwoven process methods are introduced and also some of the commonly used raw material including fibres and bonding agents. Also information about nonwovens as filtration media, the filtration process and physical properties of the filter media are brought up briefly in chapter 3. In chapter 4 special surface treatment techniques as atomic layer deposition (ALD), liquid flame spray coating (LFS) and self-assembled monolayers (SAM) are introduced.

The empirical part (chapter 5. & 6.) of the thesis consists of pilot run and testing the formed material. The pilot run was done with the pilot scale wet laid machine in Ahlstrom's plant premises and the goal was to test the boundaries of the test equipment.

2 COMPANY PRESENTATIONS

2.1 Ahlstrom Corporation

Ahlstrom Corporation is a company that produces high performance fibre-based materials for different end use purposes. The company has partnerships with leading businesses around the world. (About Ahlstrom 2016.) The company has a wide range of product selection and the main market areas are: building and decoration, energy and environment, food, beverage and household, laboratory and life science, medical care and transportation. The materials produced are for example filters, filtration media, medical fabrics, wall coverings and food packaging. (Ahlstrom. Products 2016.)

President and the chief executive officer (CEO) of the company is Marco Levi. The company has 28 manufacturing plants and 3300 employees in 22 countries. Ahlstrom corporation was listed on the Nasdaq Helsinki since 2006 and the net sale in the year 2015 were 1,1 billion euros. (About Ahlstrom 2016.)

The company was founded in Finland in 1851 and the founder was Antti Alhström. In the beginning business was based on timber trading, sawmill operations and shipping the goods. Strong profits from the shipping business provided capital for expanding the business. The expansion included iron mills, paper mills and glass factories around Finland. (Ahlstrom. History 2016; Static flash history 2016).

During the first hundred years the company was steadily growing and expanding in Finland but major step forward was in 1963 when the company acquired the majority holding in the Italian paper company Cartiere Giacomo Bosso S.p.A. This made the company a pioneer in international operations. (Static flash history 2016.)

In the year 2001 company was split in to three separate companies: Ahlstrom (the manufacturing business), Ahlstrom Capital (private investment) and A. Ahlstrom corporation (took over the family's real estates and forest reserves). (Static flash history 20016.)

2.2 Ahlstrom Tampere Corporation

Ahlstrom Tampere Corporation produces advanced and high quality filtration materials. Multi layered nonwoven filtration materials are produced by using synthetic man made fibres and natural fibres as cellulose fibres. The plant employs more than 60 persons and it is part of Ahlstrom's filtration business area. The plant has a research and development department that co-operates with customers to improve quality. (Press releases 2006.)

2.2.1 Production

The new plant started operating in a year 2000, when the old factory was transferred from the city centre into the new location. All products are made with water laid process type (introduced in chapter 3.) and due to increased quality demands continuously quality controlling is needed. (Press releases 2006.)

2.2.2 Products

Ahlstrom Tampere plant has a large product selection. For the field of transportation the company manufactures products such as automotive and heavy-duty oil, air and fuel filtration media. Special Trinitex® technology allows production of a multi-layered filter media. It is possible to obtain a material that has different composition of the inner and outer layer. Both, natural and synthetic fibres can be used. (Ahlstrom Trinitex® for oil 2016.)

For the food processing technology Ahlstrom Tampere produces filter media for milk filtration. Product ranges from thin filter media (20 g/m²) to thicker filter media (150 g/m²). Products are certified for food contact safety and due to the durability even the strictest requirements are fulfilled. (Food processing filtration 2016.)

2.3 Beneq

Beneq is a fast growing company that was established in 2005. Beneq operates in a field of nanotechnology. The company first started with producing and selling ALD (atomic layer deposition) equipment for fine coating purposes. Now Beneq offers services from research and product development to customized coating solutions in industrial production scale. (Company milestones 2005-2016.)

Beneq has a wide range of applications for coating methods as for example the ALD-coating, aerosol thin film coatings and flame-assisted coating technology. Few applications to be mentioned are surface treatments and barrier coatings for electronics. Electronics are very sensitive to moisture and oxygen and by ALD-method it is possible to make protective barrier. As an Optical treatments coatings like anti-reflective and reflective coatings can be done. Strengthened glass- application uses an invisible coating on glass to make the material stronger and more resistant to damage. This service is used among glass and display industry. (Applications of atomic layer deposition 2005-2016.)

3 NONWOVENS

In this chapter production methods for nonwovens are presented and the main focus will be in wet laid process and products that are made by this method. Raw materials including fibres and bonding agents are also discussed. Some focus will be given to filtration process, important properties required for filter media and filtration mechanisms.

The knowledge in the field of nonwovens has increased over past decades and the nonwovens have partly replaced traditional textile industry. The diagram below (in the figure 1.) presents the development of nonwoven production in Europe between the years 2000 and 2014 (weight in thousand tonnes). The production has been steadily growing.

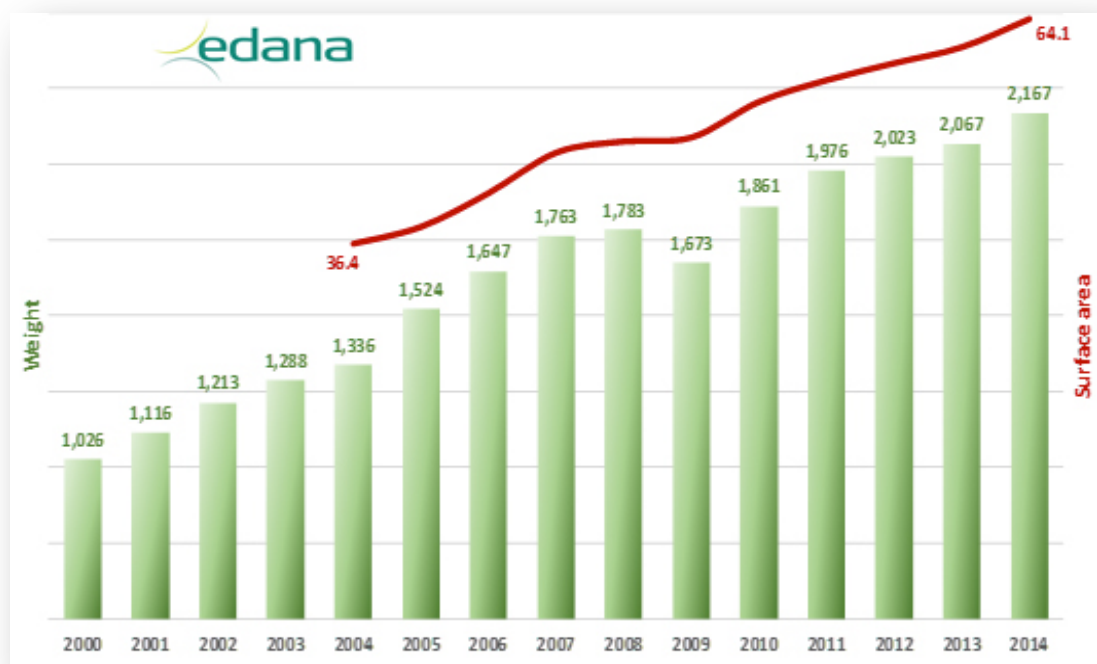


Figure 1. Development of nonwoven production in Europe (Facts and figures 2014)

Nonwovens are defined by ISO standard 9092 as “ sheet of fibres, continuous filaments, or chopped yarns of any nature or origin, that have been formed into a web by any means, and bonded together by any means, with the exception of weaving or knitting.” (Discover nonwovens. What are nonwovens.)

3.1 Production methods for nonwoven products

In a production stage of nonwoven materials fibres are arranged in a web form. Mostly used fibres are staple fibres packed in bales or fibre filaments extruded from polymer granules. Nonwoven materials can be divided in three main groups based on how the material is made. The main process methods are: drylaid, wetlaid and spunlaid processes. Drylaid process reminds textile process, wetlaid paper process and spunlaid has its origins from polymer extrusion. (Chapman 2010, 3.) The chart in figure 2. shows the quantities of nonwoven production in Europe by manufacturing process. In general the production of nonwovens has been increasing since the year 2000. Mostly used methods have been spinnelt or drylaid processes.

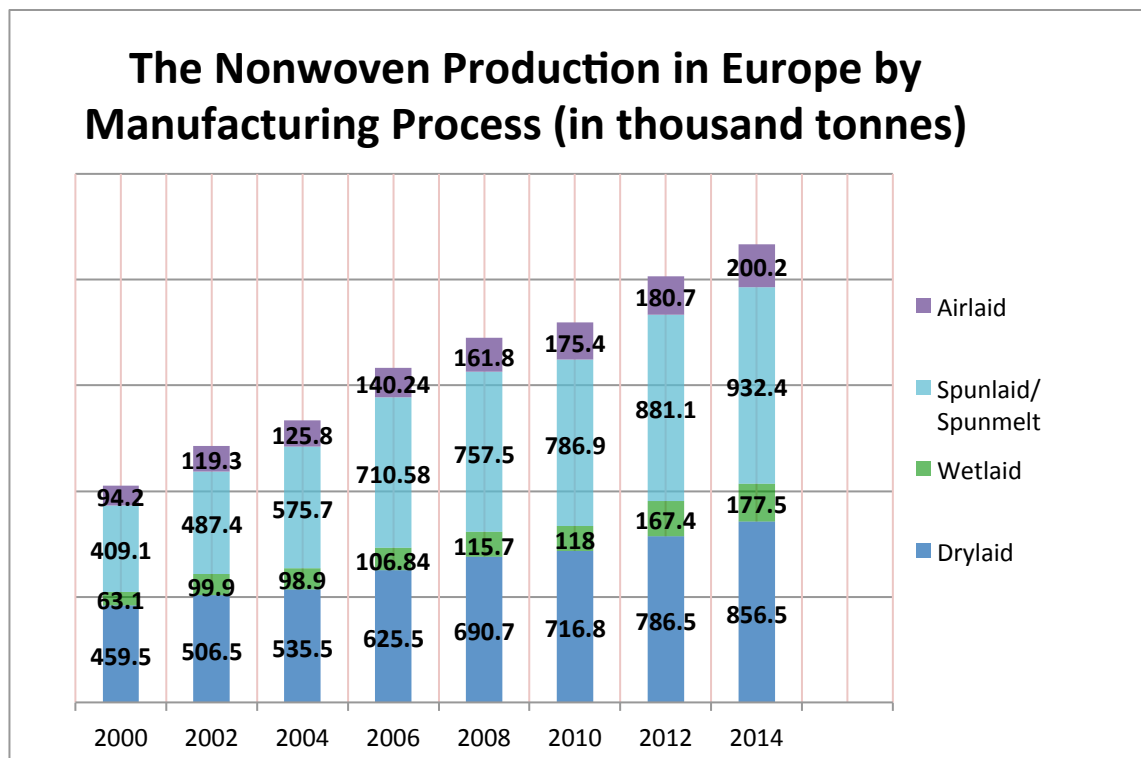


Figure 2. Nonwoven production in Europe by manufacturing process (Facts and figures 2014. Reformed)

3.1.1 Drylaid methods; carding and airlaying

Carding

Carding process (Figure 3.) is a dry laid method. The process starts with manipulating entangled fibre tufts packed in bales, resulting in individual fibres. The fibre bales are opened and blended in a special machine and the fibre tufts are conveyed by pneumatic transport in to the next stage. (Formation. Discover nonwovens.)

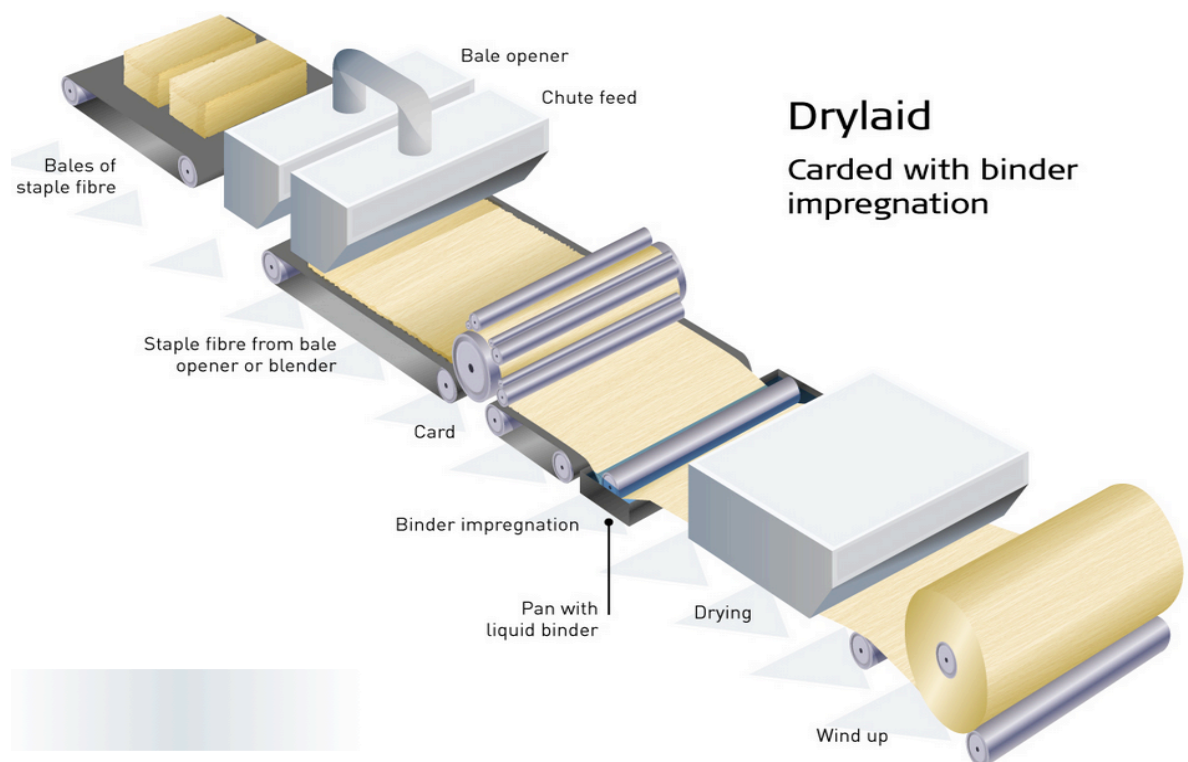


Figure 3. The carding process machinery (Formation. Discover nonwovens.)

The second process stage is the actual carding. The carding is a mechanical process in which fibres are combed by a cylinder surface covered with needles. Unpacked fibres are transported on a conveyor belt towards the carding machine. The carding machine is a large metallic cylinder covered with needles or metallic wire. Above of the carding cylinder are placed several rolling drums also covered with needles. The mat of fibres is combed while moving between carding cylinder and the drums. The cylinder and the drums are inclined in opposite directions and because of that and the different rolling speeds of cylinders the fibres are pulled apart. Due to this action fibres are aligned in a machine direction. (Chapman 2010, 5; Formation. Discover nonwovens.)

After carding the coherent fibre web is transported through a pan or a pool filled with liquid binder. Finally after binder stage the web goes to heating unit where the binder is cured. (Formation. Discover nonwovens.)

Airlaid

As well as the carding process an airlaid process (Figure 4.) also manipulates fibres in their dry state. Very short fibres can be used but staple fibres are also used. The first step is defibration of fibres. A fluff pulp of fibres is delivered in form of highly compressed rolls. Fibre rolls are fed to hammer mills that are separating the pulp into individual fibres. A one kind of a hammer mill can be seen in a figure 2. Inside of the mill small hammers are rotating at high speed. (Chapman 2010, 5-6; Formation. Discover nonwovens.)

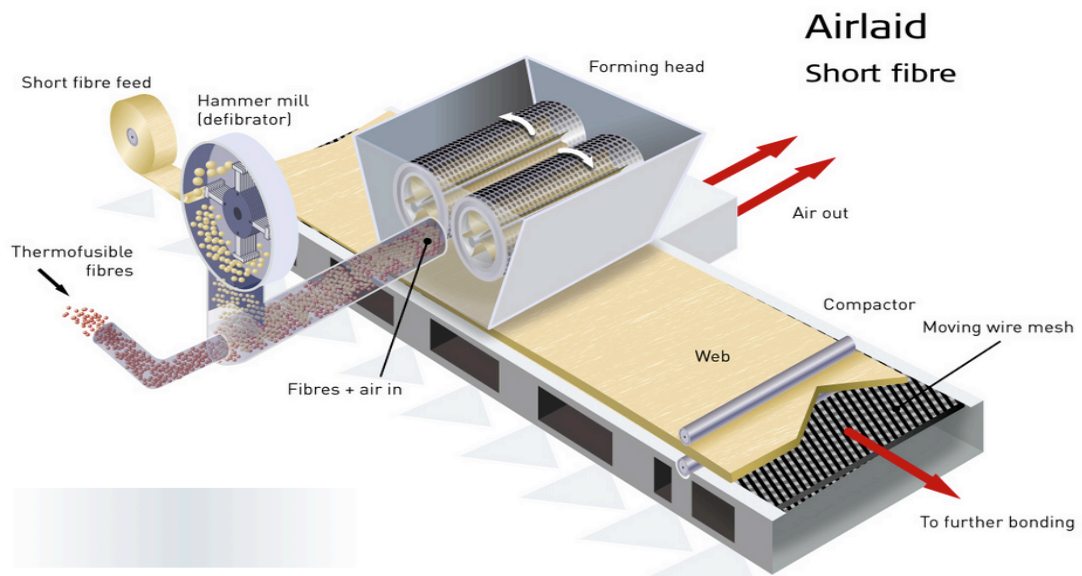


Figure 4. The carding process machine (Formation. Discover nonwovens.)

Web forming unit has large perforated cylinders rotating in opposite directions above a moving conveyer belt. Fibres are fed in to the cylinders using airstream. Through the cylinder holes fibres are set on the belt and a randomly oriented web is formed. Vacuum

system below the forming wire keeps the web in place. The formed web is compressed between rollers so that it would be coherent for binding process. (Chapman 2010, 5-6; Formation. Discover nonwovens.)

Several binding methods can be used depending on the fibre material. Commonly used methods are thermal, chemical and hydrogen bonding. Thermal bonding requires synthetic fibres (polymers) that are melt due to heat. Hydrogen bonding occurs between natural cellulose fibres when fibres are in close contact and moisture is removed. (Chapman 2010, 6.)

3.1.2 Wetlaid

Wet laid nonwoven process is similar to paper making process. In wet laid process fibres are blended in water and a product is formed on a forming wire by sieving the water. Organisation called EDENA (The European Nonwovens and Disposables Association) has distinguish a definition for wetlaid nonwoven fabrics and according to that definition: “material is regarded as a nonwoven if more than 50 % by mass of its fibrous content is made up of fibres with a length to diameter ratio greater than 300, or more than 30 % fibre content for materials with density less than $0,40 \text{ g/cm}^3$ ”. (Chapman 2010, 6-7.)

In wet laid process fibres are blended in water to form a suspension. The fibre suspension is formed in a separate unit called “mass unit”. The prepared fibre suspension is pumped from tanks to the wet laid machine. The machine (Figure 5.) consists of headbox, continuously moving forming wire, dewatering boxes, binding units and dryer units.

The fibre fluid is pumped to the headbox and from the headbox it is sprayed on the forming wire. The forming wire is porous material and due to that water is removed by gravitation from the fibre suspension. Dewatering boxes are increasing dewatering process and depending on the set underpressure material gets denser. Between the headbox and the first heating unit most of the water is removed and the nonwoven sheet is formed on the wire. Right before the first heating unit a binding unit is placed. Binding unit sprays liquid binder, usually latex based binder, on the product. In heating unit

thermal and chemical binding occurs. After the first heating unit material is solid enough for the next process stages. (Chapman 2010, 6-7.)

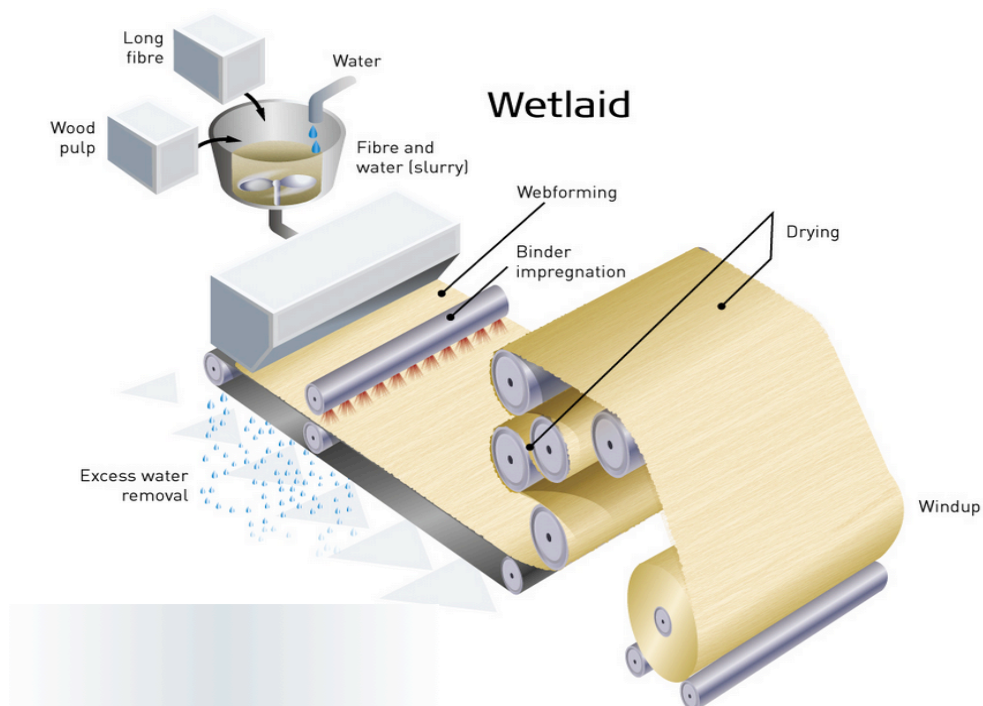


Figure 5. The wet laid process machine (Formation. Discover nonwovens.)

Comparing to the paper process in wet laid process concentration of fibre suspension has to be lower than 0,5 % (fibre-water ratio) which is quite normal for paper process. (Chapman 2010, 7.)

Depending on velocity of the fluid flow most of the fibres are going to set either in a machine direction or in a cross direction. Adjusting the headbox settings (changing the angle of the former) flow velocity of the fluid can be manipulated. This machine direction:cross direction ratio (MD:CD) is important factor effecting on the material properties such as tensile strength, elongation and tear strength. (Chapman 2010, 7.)

3.1.3 Spunlaid

Spunlaid process (Figure 6.) method is developed from polymer extrusion where raw material is in a form of polymer granules. Polymer granules are melted and the molten polymer phase is extruded through spinnerets producing continuous filaments. The filaments are deposited on a conveyor and some of the filament may adhere to one another because of the remaining heat from the extruder. The products made by spunlaid process are materials for hygiene applications. (Chapman 2010, 8-9; Formation. Discover nonwovens.)

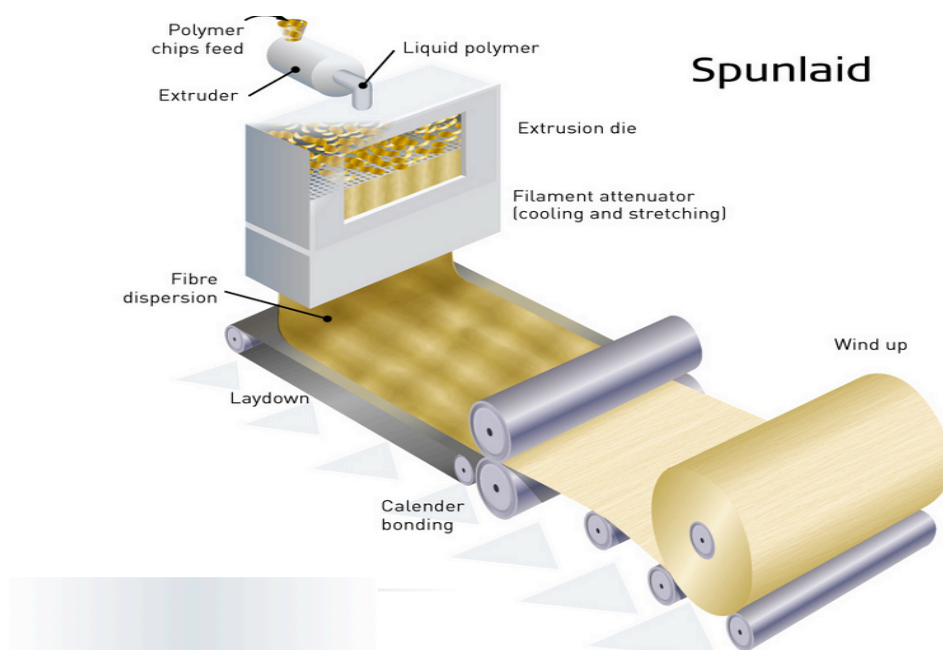


Figure 6. The spunlaid process machine (Formation. Discover nonwovens.)

3.2 Web bonding processes

3.2.1 Thermal bonding

Thermal bonding occurs when a web consisting of thermoplastic and non-thermoplastic fibres is heated to the softening or melting temperature of the thermoplastic fibre and then cooled down. Thermal bonding requires a thermoplastic component and it may be in a form of homogeneous fibre, powder or for example bi-component fibre. When

thermal energy is applied to the web thermoplastic fibres becomes viscous and melted polymer flows to the fibre-to-fibre crossover points. The bonding areas are formed on the crossover points. (NPTEL 2009-2011. Thermal Bonding.) In a Figure 7. illustrates thermal bonding in crossover points.

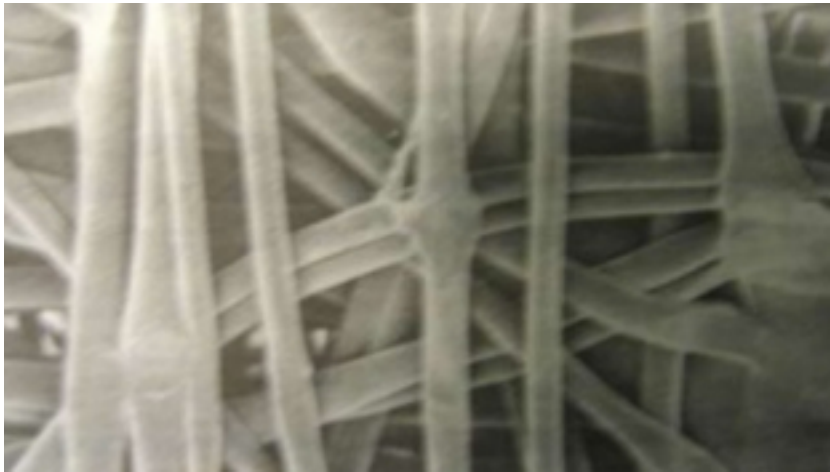


Figure 7. Thermal bonding of thermoplastic fibres (Krishnan 2013.)

Due to the heating of the thermoplastic polymers crystal regions of the chains are partially melted and that is one of the requirements for the formation of a bond. Another requirement is the temperature used in this process. The temperature needed for bonding varies depending on the used polymer. (NPTEL 2009-2011. Thermal Bonding.) Different melting points for thermoplastic polymers are shown in the Table 1.

Table 1. Melting points for thermoplastic polymers (NPTEL 2009-2011. Thermal Bonding.)

Fibre	Melting Temperature °C
PET	245-265
PP	160-175
PA	210-230
PE	115-135
PE/PET	130/250
PE/PP	130/175
CoPET/PET	110/250

3.2.2 Chemical bonding

In chemical bonding method an adhesive bonding chemical binds the web of nonwovens together. The bonding agents are polymers that are formed by emulsion polymerisation. These binders are usually water-based latexes that are formed from few components. Those components are:

- Monomers as building blocks
- Water as a medium
- Initiator which helps the polymerisation process to begin
- Surfactant aim to prevent the particle attraction
- Chain transfer agent to control the final polymers molecular weight. (NPTEL 2009-2011. Chemical Bonding Process .)

The viscosity is near to viscosity of water and that is why chemical binders can easily penetrate into nonwoven web. (NPTEL 2009-2011. Chemical Bonding Process.)

The latex polymers are the most commonly used for several reasons. Latex binders are versatile and because of their variety there can be found suitable binders for different applications and properties required. Other important reasons are ease of application and cost- effectiveness. (NPTEL 2009-2011. Chemical Bonding Process.) Figure 8 illustrates film forming between fibres.

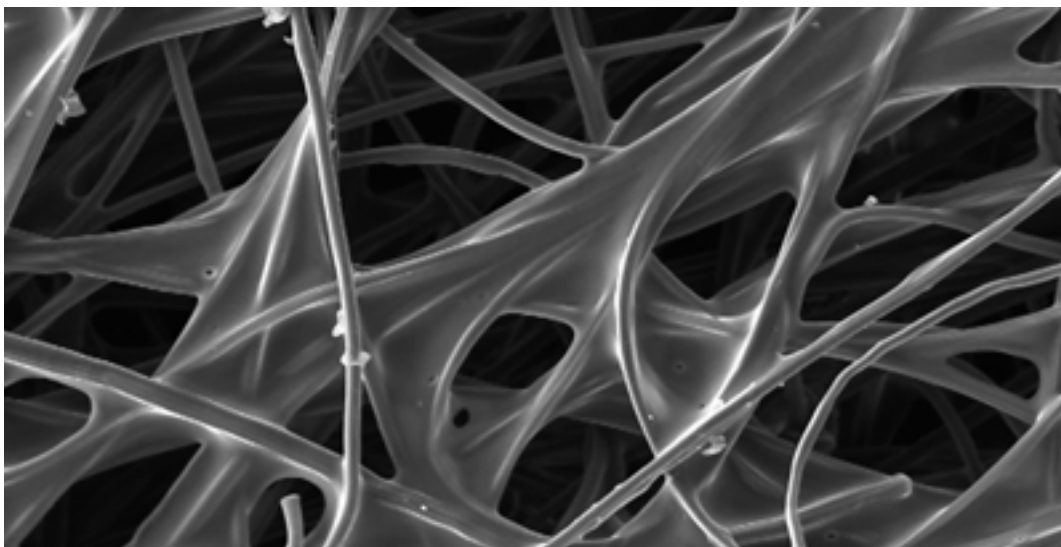


Figure 8. Chemical bonding between fibres (Discover nonwovens. What are nonwovens .)

There can be found several different ways to apply chemical binders to nonwovens. The mostly known methods are spray bonding, immersion or saturation bonding, print bonding or foam bonding. In each method the main idea is to apply the binder evenly on the nonwoven fabric and then remove extra water either from water emulsion or from foam. (NPTEL 2009-2011. Chemical Bonding Process.) After evaporating water phase by drying the binder forms an adhesive film between and across the fibres (see the figure 8.). (Milin Patel & Dhruvkumar Bhrambhattach 2011, 14)

The schematic figure of film forming process is presented in the figure 9. When the binder is applied on the web and water phase is evaporated the polymer particles in dispersion are getting closer to each other. When coagulation occurs strong capillary forces are activated and those forces deforms the particles and merge them together. When the film is formed the process becomes irreversible. (Milin Patel & Dhruvkumar Bhrambhattach 2011, 14.)

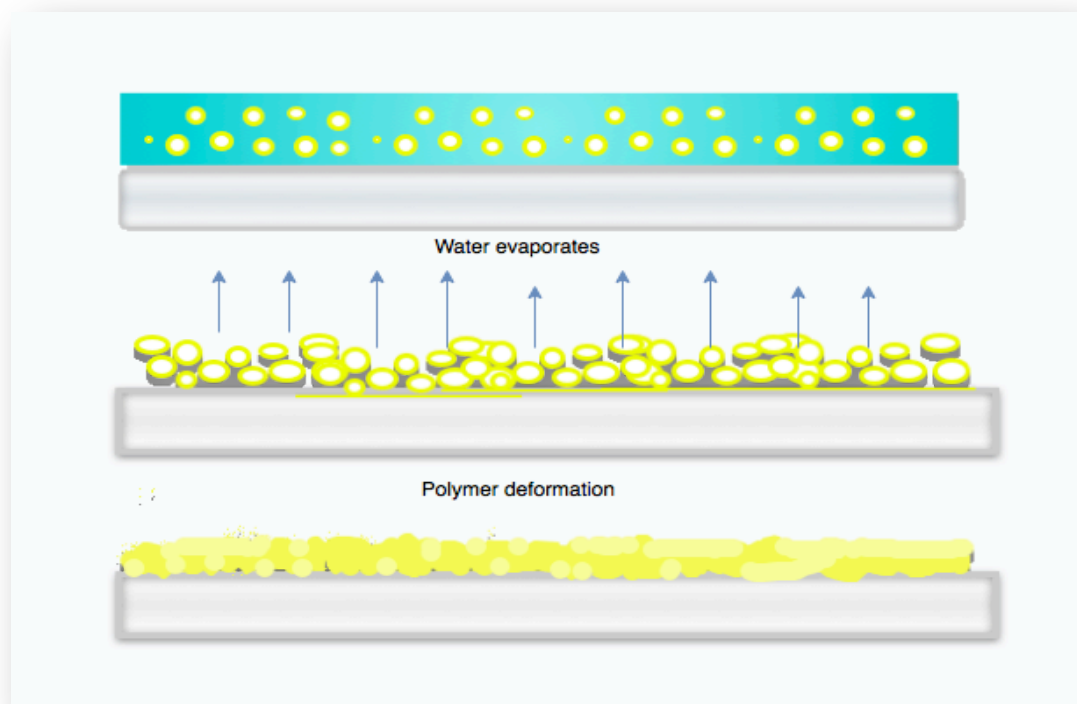


Figure 9. A Schematic figure of film formation. Reformed. (Milin Patel & Dhruvkumar Bhrambhattach 2011, 14.)

3.3 Raw materials

A great variety of raw materials in the market allows production of different nonwoven fabrics. The choice of fibres, the bonding process, the bonding agent and technology to be used defines the properties of nonwoven fabrics. Some of the properties that are defined by the end use product are: high or low tear strength, softness, roughness or crisp touch. Nowadays a great value is given to disposable materials but also durability is a valued feature. (Milin Patel & Dhruvkumar Bhrambhatt 2011, 5.)

The goal of nonwoven manufacturing is to obtain desired fabric and it can be obtained with the right type of raw materials and the right process method. In theory all types of fibrous material can be used as a raw material for nonwoven fabrics. Fibres physical, mechanical and chemical properties need to be considered when choosing the fibre type. From physical properties crimp, fibre length, finish and deniers per filament are important factors that effect to the product properties. (Milin Patel & Dhruvkumar Bhrambhatt 2011, 5; Textile learner 2012.)

3.3.1 Rayon (viscose)

Viscose is a man made fibre based on naturally occurring organic polymer called cellulose. Viscose is renewable fibre with various molecular structures. The fibres are mechanically and chemically stable but when needed, viscose can be physically and chemically modified. Viscose fibres are skin friendly and hydrophilic and due to that the material provides good moisture management. (Roggenstein 2011, 72).

Other factors to the popularity of viscose are low cost and the ease of processing on all types of equipment. Viscose fibres can be found as crimped and un-crimped, bleached and unbleached, range of deniers, range of lengths and range of finishes. (Roggenstein 2011, 72-73; Milin Patel & Dhruvkumar Bhrambhatt 2011, 6.)

3.3.2 Acetate

Acetate is a man-made fibre (processed from wood pulp and cellulose) in which the fibre forming substance is cellulose acetate. Characteristics for acetate fibre are the ability to fast drying and resistant to shrinking and mildew. The material has a good durability and softness. (Acetate fiber 2016.)

3.3.3 Polyamides, polyester

Polyamides (also called Nylon) and polyester fibres are widely used in many applications and are produced in high quantities. Polyamide was the first commercially successful synthetic polymer. (Textile tuts 2014.)

Polyamides are defined as fibres composed of linear macromolecules having amine linkages in the structure. Also either aliphatic or cycloaliphatic groups can be found. (European man-made fibres association 2016.) A figure 10. shows the chemical formula of an amide group and polyamide molecule. The positive properties of polyamides are good resistance to soiling, abrasion and chemicals, good heat processability and good wet strength. The negative features are high price and high pilling tendency. (Milin Patel & Dhruvkumar Bhrambhatt 2011, 7)

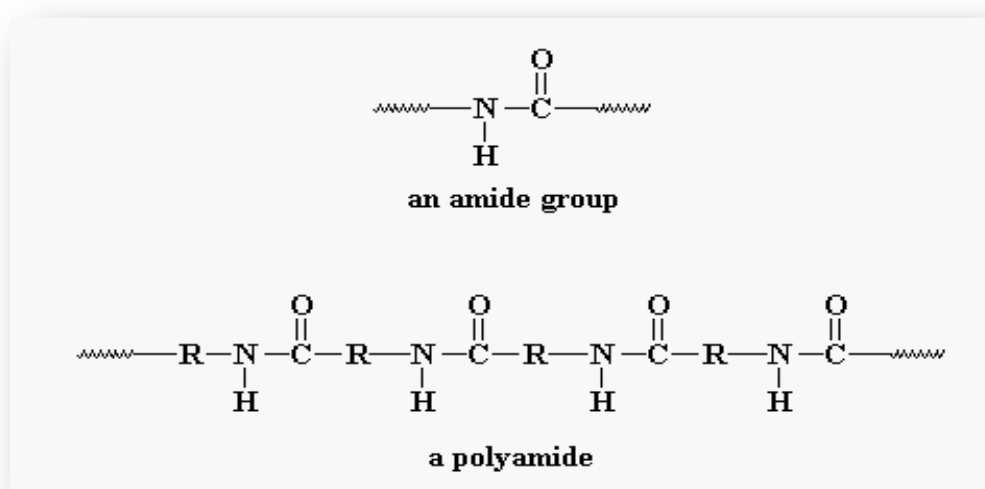


Figure 10. Chemical formulas for an amide group and polyamide molecule. (Textile tuts 2014.)

Polyesters have good properties including a good heat setting, high elasticity and wet strength. As negative properties it has a tendency to formulate static charge and a high pilling tendency. (Milin Patel & Dhruvkumar Bhrambhattach 2011, 7)

3.3.4 Acrylic fibres

Acrylic fibres are composed of linear macromolecules having at least 85 % of acrylonitrile repeating units in the chain. Acrylic fibres are flexible and soft and therefore are used in knitted clothing. (European man-made fibres association 2016.) Other properties are excellent chemical and moisture resistance, high bulk and good elastic recovery. Negative features are high price, low abrasion resistance and tendency to pilling. (Milin Patel & Dhruvkumar Bhrambhattach 2011, 7)

3.3.5 Bonding agents

The bonding agents have a huge influence on properties of the nonwoven fabrics. Factors influencing nonwoven performance are the backbone structure of the binder, functional groups of the main polymer chain, and the process that applies the binder (Milin Patel & Dhruvkumar Bhrambhattach 2011, 14.) Often the properties of the end use product determine the choice of binder. The bonding agent works as a glue that binds the fibre web together. Some important characteristics that defines the suitable bonding agent are:

- Strength as resistance to tearing, pilling and ripping
- Elastic tenacity and bend-ability
- Resistance to chemicals and resistance to air and oxygen
- Resistance to light and heat
- Washing and dry cleaning ability
- Hydrophilic or hydrophobic properties (Milin Patel & Dhruvkumar Bhrambhattach 2011, 11.)

The used bonding agents might be in liquid form or in solid form. Solid binders are usually thermoplastic fibres or powders or pastes made from thermoplastic polymers. The

liquid binders are polymer dispersions. The binders are usually classified according to their chemical structures and the most frequently used binders are butadiene copolymers, acrylates, vinyl copolymers. (Milin Patel & Dhruvkumar Bhrambhata 2011, 11.)

Butadiene copolymers

The butadiene polymers are cross-linked by poly-sulphides (disulphide molecule where two sulphides are bonded together by a covalent bond.) and their properties can be modified by different copolymers. Copolymers used with butadiene polymers are styrene and acrylonitrile monomers. (Milin Patel & Dhruvkumar Bhrambhata 2011, 14.)

Styrene-Butadiene binders have a good combination of flexibility and toughness and the range reach from very soft to very firm. This binder can be very hydrophobic and durable when cross-linked. Styrene-Butadiene has a tendency to oxidize and that is why they are affected by light and heat. (Milin Patel & Dhruvkumar Bhrambhata 2011, 16.)

Acrylates

Acrylic binders are the most versatile binders and they are available with various modifications. The properties of these binders can be modified by copolymers. Acrylic binders are usually copolymerized with styrene, acrylonitrile, vinyl chloride or vinyl acetate. The properties as hardness comes from styrene, flame resistance from vinyl chloride and solvent resistance from acrylonitrile. The benefit of vinyl acetate is low cost. (Milin Patel & Dhruvkumar Bhrambhata 2011, 14.)

Vinyl copolymers

Vinyl chloride and vinyl acetate are the mainly used vinyl copolymers as binders. Vinyl acetate binders are firm and they offer good toughness and dry strength but they also have a tendency to yellow when heated. In vinyl chloride binder the chloride provides flame retardance. (Milin Patel & Dhruvkumar Bhrambhata 2011, 15.)

3.4 Nonwovens as a filtration media

The type of a filter media needed depends on the particles to be filtered and the surrounding conditions. When choosing filter media the basic knowledge required about particles are particle size, concentration of particles and possible tend to agglomeration. The surrounding conditions that will be necessary to know are for example temperature, flow velocity, pressure, humidity, mass flow etc. (Zobel & Gries 2010, 160-163.)

As a filtration material nonwovens are versatile and adjustable. Because of a wide range of raw materials in market nonwovens offer also a wide range of end use products for different filtration purposes and surroundings. In nonwoven filter media fibres form a thick web. This structure is increasing the filtering surface area and permeability and by folding or pleating filtering surface area can be enhanced. (Zobel & Gries 2010, 160-163.)

3.5 Filtration process

Filtration process is a separation of particles from fluid-solid suspension. The fluid may be a gas or a liquid and the particles may be solid, liquid or gaseous. The fluid-solid suspension goes through a septum or a membrane that captures most of the solids on or within itself. The separating septum is called filter medium and the equipment that holds the filter medium is called filter. (Zobel & Gries 2010, 163.)

Filters and filtration processes can be classified as dry and wet filtration, surface filters and depth filters. Depending on the particle size to be filtered filter media are classified as micro- and ultra-filtration. (Zobel & Gries 2010, 163.)

3.5.1 Dry and wet filtration

In dry filtration the medium to be filtered is either gaseous or solid and the particles to be separated are solid, liquid or gaseous. When separating solid or liquid particles from gaseous phase filter fabrics are widely used. (Zobel & Gries 2010, 163.)

The air and gas filtration is used in domestic, industrial and automotive filtration purposes. Domestic filters are used in vacuum cleaners, cooking and in heating, ventilation and air conditioning (HVAC). In industrial use there is a requirement for high efficiency filter media that separates fine particles. For that purpose high efficiency particulate air filter (HEPA) and ultra low penetration air (ULPA) filters are used. (Zobel & Gries 2010, 163.)

3.5.2 Surface filtration and depth filtration

The classification into surface and depth filters is often made but in many cases these both ways occurs in filter medium. In surface filtration the particles have usually greater diameter than the pores in the filter medium and the particles are deposited on the filter surface. The particles can block the pores and that will lead to increased fluid resistant and eventually the filter has to be cleaned. (Zobel & Gries 2010, 164-166.)

An ideal situation in surface filtration is when substrates in a fluid form a porous layer on the filter surface. This layer is often called a filter cake. Filter cake has a smaller pore size than the actual filter media has and that facilitates the filtration process. The filter cake functions as a depth filter when particles are mechanically held or adsorbed into the cake. (Zobel & Gries 2010, 164-166; Sutherland 2008, 18-20.) Both surface filtration and depth filtration are presented in a figure 11.

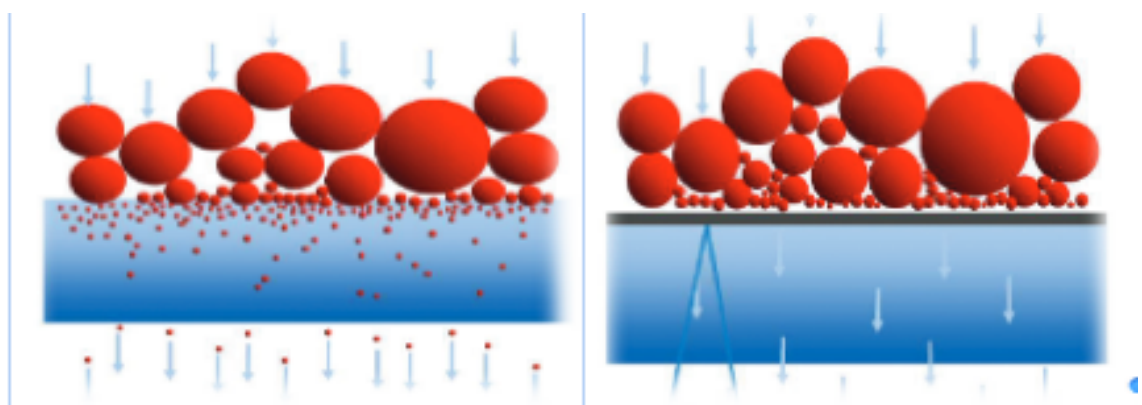


Figure 11. Schematic picture of depth filtration (on the left) and surface filtration (on the right) (Donaldson filtration solutions 2013, 1.)

In depth filtration (Figure 12.) particles are mechanically held or adsorbed into the filter media. Often these types of filters are used in applications where a variation in particle size is large. The filter media can have a progressive density of pore structure. For example the filter media can have an open structure on the surface (meaning bigger pore sizes and greater air permeability) and below the surface filter media has a thicker structure. The benefits in depth filtration are high filtration efficiency over a wide range of particle sizes and its suitability for the filtration of more difficult solids for example particles of different dimensions. (Zobel & Gries 2010, 164-165.)

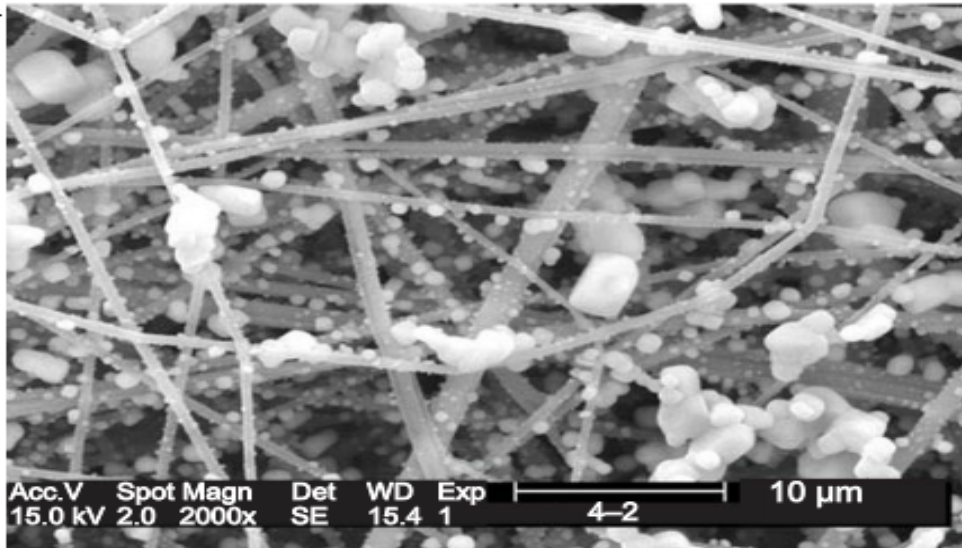


Figure 12. Depth filtration of sodium chloride (NaCl) particles in a meltblown fabric. (Tsai & Yan 2010, 40.)

3.5.3 Filtration mechanisms

The mechanism that collects particles from carrier fluid by a filter medium is shown in Figure 13. The picture 13. on a page 27 presents a cross-section of a single fibre in a fluid flow. Four main mechanisms occurs:

- direct interception
- inertial impaction
- diffusion

- mass of fibres wrapping smaller particles (Sutherland 2008, 16-18.)

In absence of electrical charges on fibres or on particles all particles will be attracted to the fibre. This happens when particles are brought close enough to the fibres. These attractive forces (van der Waal's forces) are weak but still strong enough to hold the particle on the fibre. When this attachment happens the particle creates an extension of the fibre and can trap other particles. (Sutherland 2008, 16-18.)

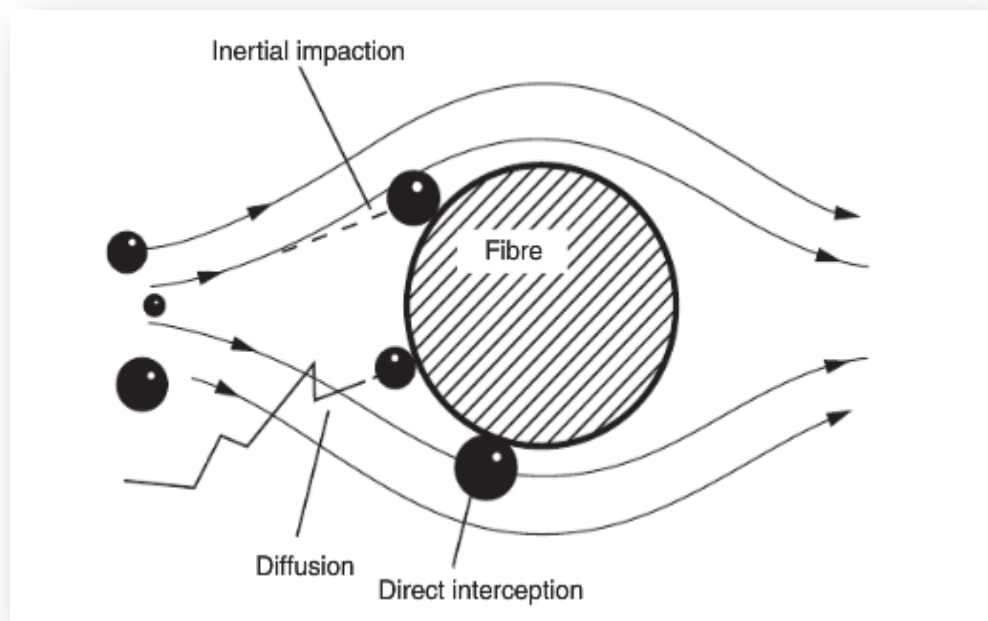


Figure 13. Main filtration mechanisms. (Sutherland 2008, 16.)

The fluid flow in the filter medium can be considered as a laminar flow. The fluid flows in smooth streamlines around the fibres and the particles follow the stream if they are not disturbed. The fluid streamlines bend around obstacles. When the particle is taken close enough to the fibre surface (to distance of less than half of its diameter from the fibre surface) direct interception happens and the particle is trapped. This mechanism is defined to happen on the flanks of the fibre. (Sutherland 2008, 16-18.)

Inertial impaction happens when a larger particle or a particle moving too fast cannot follow the streamlines. These particles will carry too much inertia to make the turn and

then they will cross the streamline and be trapped on the fibre. (Sutherland 2008, 16-18.)

Some particles, mainly small ones, meander across the streamlines. When the particle pops out the streamline near the fibre it will get trapped. This is called diffusion mechanism. One important way of catching small particles (Figure 14.) (smaller than the pores in fibre web) is the massive amount of fibres and the randomly oriented structure of the web. (Sutherland 2008, 16-18.)

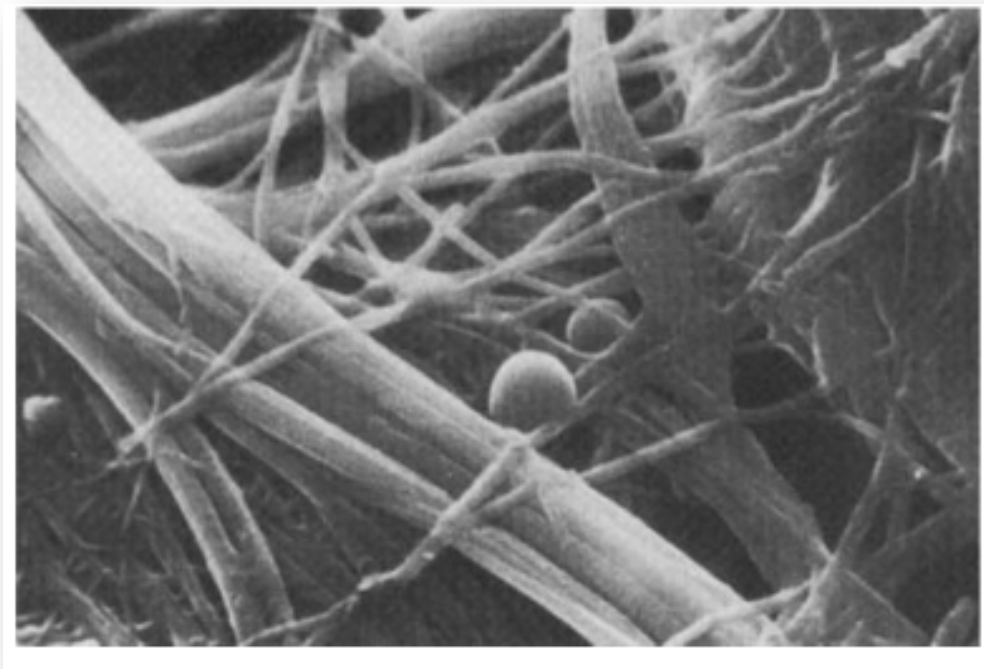


Figure 14. Small particles trapped by fibre web. (Sutherland 2008, 18)

3.6 Important chemical and physical properties of filter media

The used nonwoven application determines the properties required for the material. The most important properties to be considered are first of all the fibre properties. The fibre properties are the fibre size, fibre shape, fibre surface roughness and surface chemical functions and these effects on the performance of the filter media. The fibre properties are effecting on the products pore size, packing density and fibre orientation in the web.

All these properties together determine the nonwoven's physical, mechanical and chemical performance attributes such as:

- Thermal and acoustic insulating properties
- Barrier properties
- Mechanical strength
- Filtration efficiency (FE) (Tsai & Yan 2010,18.)

The used fibre size effects on the packing density and these are two critical factors when determining the filtration efficiency and the dust holding capacity. Fibrous materials have high fabric porosity, resulting in reduced air resistance, and a large fibre surface area that contributes to high filtration efficiency. Minimizing the fibre size a maximized porosity is obtained and that way the desired filtration efficiency is achieved. In an air filtration particles are captured on the surface of the fibres and therefore packing density and increased fibre surface area are in important role. Then again in a liquid filtration the idea is to make the medium with small pore size, smaller than the particles to be captured. (Tsai & Yan 2010, 19.)

In oil filtration oleophilic fibres, (such as polypropylene (PP) or polyethylene (PE)), that have a good affinity with oil are preferred. In the filter media oil occupies the pores and that is why the amount of oil that can be absorbed is determined by porosity. The fabric should have a structure that can provide enough capillary force to hold the absorbed oil. (Tsai & Yan 2010, 19.)

Nonwovens mechanical strength is obtained from the fibre strength and also from the bonding strength among the fibres. As anisotropic material (meaning that the strength of the fibre differs based on the direction of the measurement) nonwovens directional strength is firstly determined by the fibre modulus and secondly by the fibre orientation distribution in the web. (Tsai & Yan 2010, 20.)

3.6.1 Pore size

Pore size controls filtration and fluid permeability through the medium. Pores can be treated as a circular channel (Figure 15. on a page 30) and pore size can be calculated by the modified Hagen-Poiseuille equation (1.) (Tsai & Yan 2010, 21-23.)

$$\frac{\Delta P}{L} = \frac{32 \mu v}{D^2} \quad (1.)$$

where ΔP is a pressure drop , L is the length of the channel, D is the diameter of the channel, v is the velocity and μ is the viscosity of the fluid.

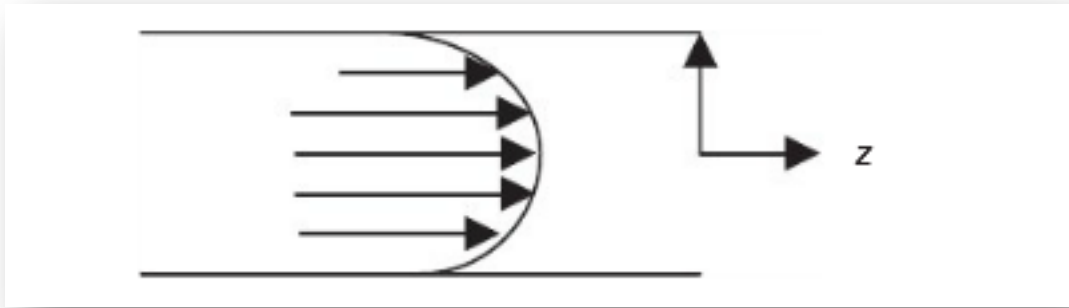


Figure 15. Velocity profile of a fluid flow in a channel. (Tsai & Yan 2010, 21.)

Considering that the channel in actual filter media is not straight some correction to the length of the channel has to be made. Because of the pores tortuosity the fluid goes longer path than in a straight channel. The tortuous path l can be obtained from equation 2. (Tsai & Yan 2010, 21-23.)

$$l = LT \quad (2.)$$

Where L is the length of the straight channel and T is the path tortuosity.

The velocity is increased in the medium by a reduction in the available cross-sectional area. Therefore some correction to the velocity term is needed (Equation 3.). (Tsai & Yan 2010, 21-23.) The new velocity in the medium becomes:

$$v = \frac{v_0}{\varepsilon} \quad (3.)$$

Where v_0 is the velocity at the entrance of the medium and ε is the porosity of the web. After correcting the fluid travelling length and the velocity in the media the equation (1.) becomes:

$$\frac{\Delta P}{tT} = \frac{32 \mu v_0}{\bar{D}^2 \varepsilon} \quad (4.)$$

In equation (4.) channel length L has been replaced with web thickness t . The term \bar{D} is the average circular-capillary-equivalent pore size (ACCP). (Tsai & Yan 2010, 21-23.)

3.6.2 Packing density

Packing density indicates the compactness of the web and it is important factor in determining the pore size. Packing density is defined as the ratio of the volume occupied by the fibres to the whole volume (Equation 5.). (Tsai & Yan 2010, 20.)

Packing density, α

$$\alpha = \frac{V_f}{V_{web}} = \frac{\frac{W_f}{\rho_f}}{tA} = \frac{\text{Basis weight}}{t\rho_f} \quad (5.)$$

Where V_f is volume of fibres, V_{web} is volume of the web, W_f is weight of fibres equals weight of the web, ρ_f is or polymer density of fibres, t is thickness of the web and A is area of the web. (Tsai & Yan 2010, 20.)

When packing density (α) is known porosity of the web can be calculated. Porosity (ε) is fraction of the void volume to the volume of the web (Equation 6.).

Porosity, ε

$$\varepsilon = 1 - \alpha \quad (6.)$$

3.6.3 Air permeability

Air and fluid permeability is determined by the medium's properties such as thickness, fibre diameter and packing density. Fluid properties including the fluid viscosity and the fluid velocity are also effecting to the permeability. (Equation 5.). (Tsai & Yan 2010, 32.)

4 A THIN FILM DEPOSITION TECHNIQUES

In this chapter some of the advanced surface treatment methods and their applications are introduced briefly. First technique is atomic layer deposition, which is abbreviated as ALD and second introduced technique is liquid flame spray (LFS) and finally the third is self-assembled monolayers (SAM).

4.1 Atomic layer deposition

An atomic layer deposition is a technique that can be used for different surface treatment purposes. In atomic layer deposition (ALD) a solid thin film is formed layer by layer on a surface. ALD is more advanced version of chemical vapour deposition, which was developed in 1920s. In chemical vapour deposition a mixture of gases flows over a heated surface forming a thin film on a substrate. The risk in this method is reaction in a gas phase. The unwanted gas phase reactions forming particulates can cause uneven layer. (Kääriäinen, Cameron, Kääriäinen, Sherman 2013, 1-2.)

ALD technique uses gas reactants separately instead of gas mixture and this way the risk of gas phase reactions is eliminated. In ALD method a heated substrate is placed in a chamber where the film forming is going to take place. In a figure 16 on a page 34 a typical reactor configuration is illustrated. The stationary substrate is placed in the end of the chamber and in the other end two valves for reactants and a vacuum pump. (Kääriäinen et al. 2013, 2.)

In a step one the first reactant flows to the chamber and the gas reacts with the heated substrate. After the monolayer forming the first reactant gas is pumped away and the second reactant is introduced into the chamber. The second gas will react with the first monolayer and one complete layer is formed. These steps can be repeated as many times until the desired thickness of the film is achieved. (Kääriäinen et al. 2013, 2.)

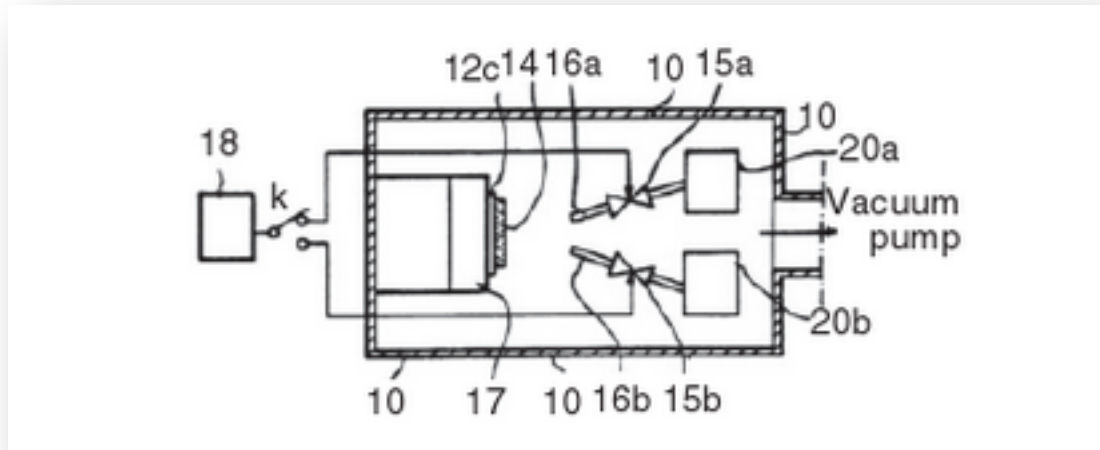


Figure 16. Schematic figure of an ALD reactor. (Kääriäinen et al. 2013, 11.)

The growth rates are depending on the reactants reactivity and the temperature used in the process. Figure 17. shows growth rate as a function of temperature. The more staple the reactant molecules are the higher temperature has to be used to result higher growth rate. In the figure 17 the silicon tetrachloride (SiCl_4) molecule is the most staple and requires high temperature to achieve relatively low growth rate. When choosing the reactants in CVD-method this factor has to be considered carefully since more reactive reactants can result in unwanted gas phase reactions. (Kääriäinen et al. 2013, 3-4.)

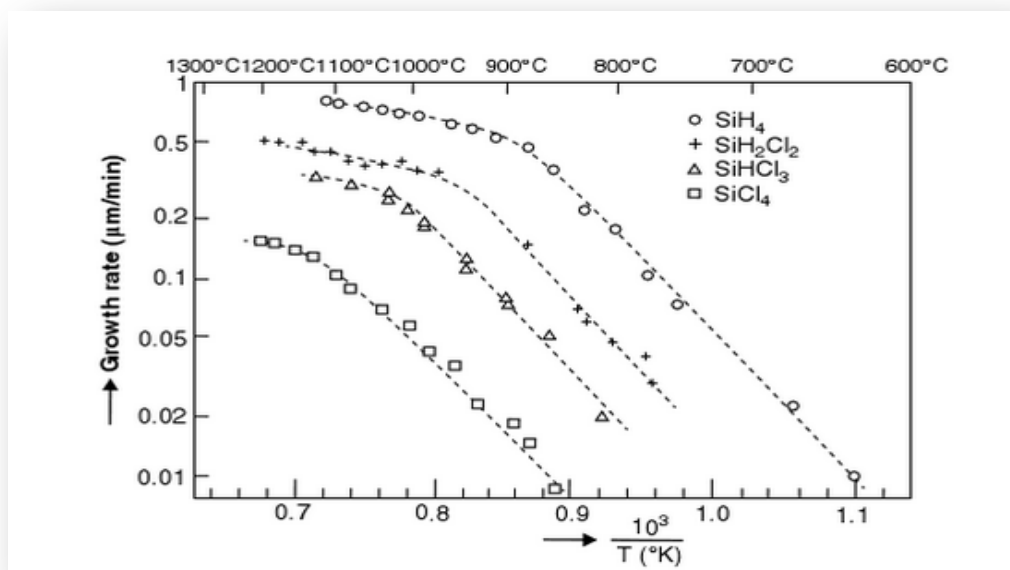


Figure 17. Growth rate as a function of temperature. (Kääriäinen et al. 2013, 3.)

In ALD more reactive reactants can be used and the film deposition temperatures can be lower. A minor disadvantage is that the film deposition rate can be slow but thin and uniform film can be formed. (Kääriäinen et al. 2013, 2.)

For successful particular deposition in the ALD process there are temperature constraints that has to be noted. The “Temperature window” is shown in figure 18. This schematic picture shows deposition rate on the vertical axis versus temperature on the horizontal axis. The temperature window explains possible outcomes for different temperature conditions. For example if the temperature is too low the result could be more than one monolayer adsorbed per cycle or a condensation of a liquid or solid layer on the surface. In this case the deposition rate would be higher than expected. On the other hand when the temperature is too low, the reaction rate may be so slow that the reaction time would be longer than the cycle time. Then there wouldn't be enough time for a complete monolayer to form and the deposition rate would be too low. (Kääriäinen et al. 2013, 12-13.)

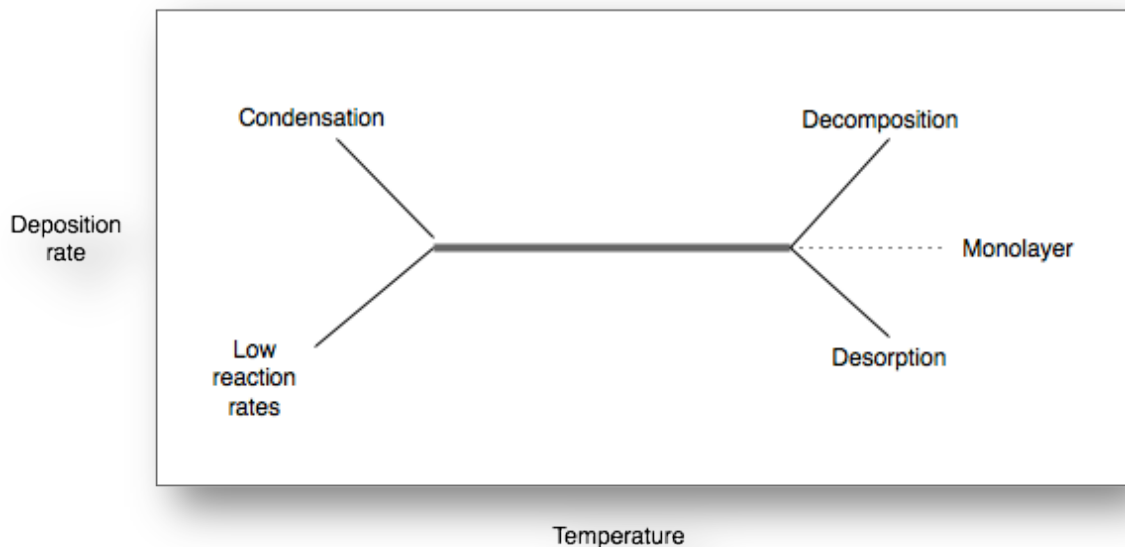


Figure 18. Temperature window for ALD process. (Kääriäinen et al. 2013, 12.)

In the case of a too high temperature decomposition of the first chemical reactants may occur. The result would be higher deposition rates than normally in ALD process. Then again if the first reactant is very stable it may desorb from the surface before reaction with the second reactant and that would result to lower deposition rate. (Kääriäinen et al. 2013, 12-13.)

4.2 Layer adsorption

During the first step in ALD the first reactant has to form a self-limiting monolayer on the surface. Self-limiting monolayer is important factor when forming a uniform film. The first reactant molecule bonds with the surface molecule forming a monolayer. The bonded reactant molecules prevent furthermore bonding of the other reactant molecules (the reactant molecules of the same species) on the formed monolayer (self-limiting monolayer). (Kääriäinen et al. 2013, 6-7.)

The molecules may be attached on the surface by relatively weak forces such as physical adsorption (physisorption) or by strong chemical adsorption (chemisorption). Molecules, which are bonded with chemisorption tend readily to form a self limiting monolayer and the monolayer is also stable at moderate temperatures. The monolayer bonded with physical adsorption can be easily desorbed if the temperature rises too high and because of the high process temperatures bonding with chemisorption is more likely to happen. With low enough temperatures a self-limiting layer can be formed by physisorption as well. (Kääriäinen et al. 2013, 6-9.)

In both ways the adsorption rate will be finite and the bond strength that binds the gaseous molecules to the surface varies depending on the type of the surface and on the nature of the gas molecules. (Kääriäinen et al. 2013, 7.)

4.2.1 Physisorption

Forces called van der Waals forces are acting in physical adsorption. Those forces are usually quite weak (< 20 kJ/mole) but the rates of adsorption are fast. (Kääriäinen et al. 2013, 7.) Under the correct pressure and temperature conditions all gases will physisorb a self-limiting monolayer. At low pressure if the temperature is too low more than one monolayer will physisorb. When the temperature is lowered even more a liquid or solid film is formed. If the temperature is raised the formed layers will desorb. The formation of complete monolayers depends on the pressure and the concentration in the gas phase. (Kääriäinen et al. 2013, 8.)

A gas particle is adsorbed when it hits a bare surface but will be reflected back to the gas phase when it strikes an occupied site on the surface. When there are fewer sites available the surface coverage approaches the unity and the rate of adsorption slows down. (Kääriäinen et al. 2013, 9.)

4.2.2 Chemisorption

In chemisorption the reactant forms a chemical bond with the atoms on the surface. The forces holding the formed layer in the place are on the order of chemical bonds between the atoms. (Kääriäinen et al. 2013, 10.) Because the adsorbed molecules are attached to the surface by valence bonds, they will occupy certain adsorption sites and that is why only one layer of chemisorbed layer is formed. (Chemisorption and physisorption 2002.)

The surface reactions between the two reactants may require high temperature (in order to achieve a fast reaction) and therefore the bonding forces have to be high enough that the layers sustain bound until the reaction is completed. (Kääriäinen et al. 2013, 9.)

4.2.3 Applications

The applications for ALD process include different surface treatments for the field of microelectronics (dielectric, conductor and semiconductor materials). In the field of nanotechnology applications such as coating on nanoparticles, nanomaterials and porous objects have a promising future. Optical coatings, solar cells and thin film Electroluminescent displays, anti-corrosion layers are also ideal applications for ALD. (Kääriäinen et al. 2013, 216-218, 226-227, 228-233.)

4.3 Liquid flame spray coating

The liquid flame spray (LFS) coating method is a new advanced coating method that can be used for heat sensitive materials. In this flame spraying process a high temperature and high velocity flame is produced using either electrical or chemical energy. Liq-

uid feed is injected in to the flame where the material is atomized. Due to the heat the liquid phase is evaporated and via thermochemical reactions fine particles are produced. (Tikkanen et al. 1997, 210.) A schematic figure of the LFS equipment can be seen in a figure 19.

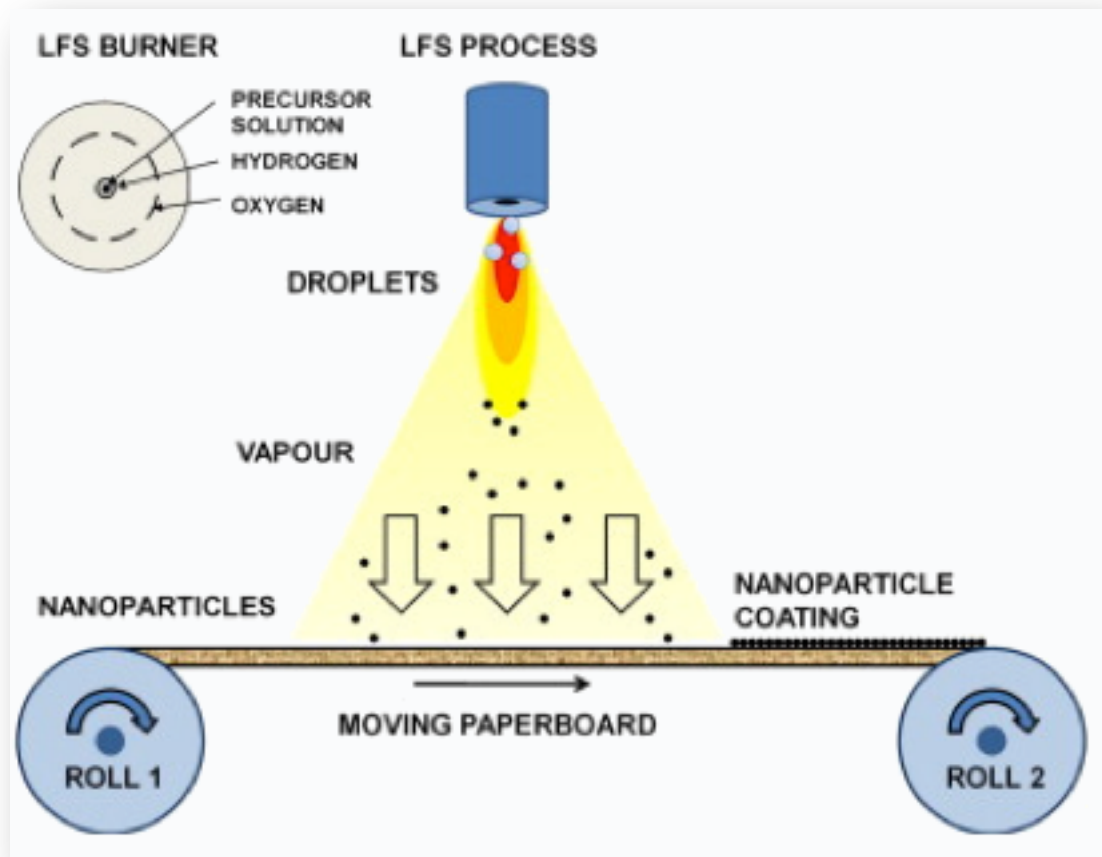


Figure 19. A schematic figure of the liquid flame spray process on paperboard. (Teisala 2013, 47)

The precursor chemicals in the feed material can be dissolved either in water or organic solvents such as alcohol and acetone. It is noticed that aqueous solutions allow higher concentrations than organic solvents. Aqueous solutions are also cheaper to produce and safe to handle and store. (Tikkanen et al. 1997, 212-214.)

A various atomizers can be used to produce micron-sized droplets:

- An ultrasonic atomizer
- A centrifugal atomizer
- A single fluid and a two fluid atomizer (Tikkanen et al. 1997, 212-214.)

During the process the droplet size distribution can be varied by changing the parameters such as atomizing gas flow rate, liquid feed rate and liquid type. The flame length can be controlled by the gas flow rates and by the used solvent (organic solvents extends the flame length). (Tikkanen et al. 1997, 216.) The LFS process is illustrated in the figure 20.

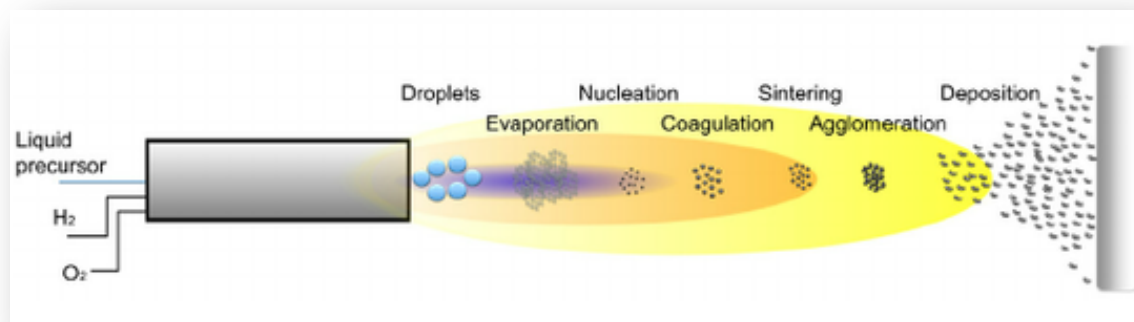


Figure 20. The aerosol process in the liquid flame spray method. (J Haapanen et al. 2010)

4.4 Self-assembled monolayers

Self-assembled monolayers (SAMs) and self-assembled multilayers (SAMTs) are molecular assemblies formed spontaneously by adsorption of an active molecular precursor onto a solid surface. The molecules to be attached onto the surface are usually dissolved in solvents. For the deposition the substrate is immersed in the solvent and after sufficient time the monolayer will be formed. (DiBenedetto, Facchetti, Ratner, & Marks 2009.) The exact structure of the monolayer depends on the chemistry of the chain (Boeckl & Graham 2006.)

The important components, the precursor molecules of SAMs (Figure 21.) are the anchoring group, the head group and the linking group. The anchoring group attaches the molecules to the surface and the head group defines the nature and functions of the new outer surface. The linking groups, between the anchoring and the head group, generally consist of hydrocarbon or fluorocarbon chains with more than 8 carbon atoms. Via van der Waal forces these linking groups can create a certain degree of order in the system

(more stable and uniform layer). With more carbon atoms in the chain the van der Waals interaction increase and so does the degree of order in the system. (Spencer. 2011, 2-3.)

The interaction between the alkane chains causes the thiol chains to tilt. The tilting maximizes the interaction between the chains and it also lowers the over all surface energy. (Boeckl & Graham 2006.)

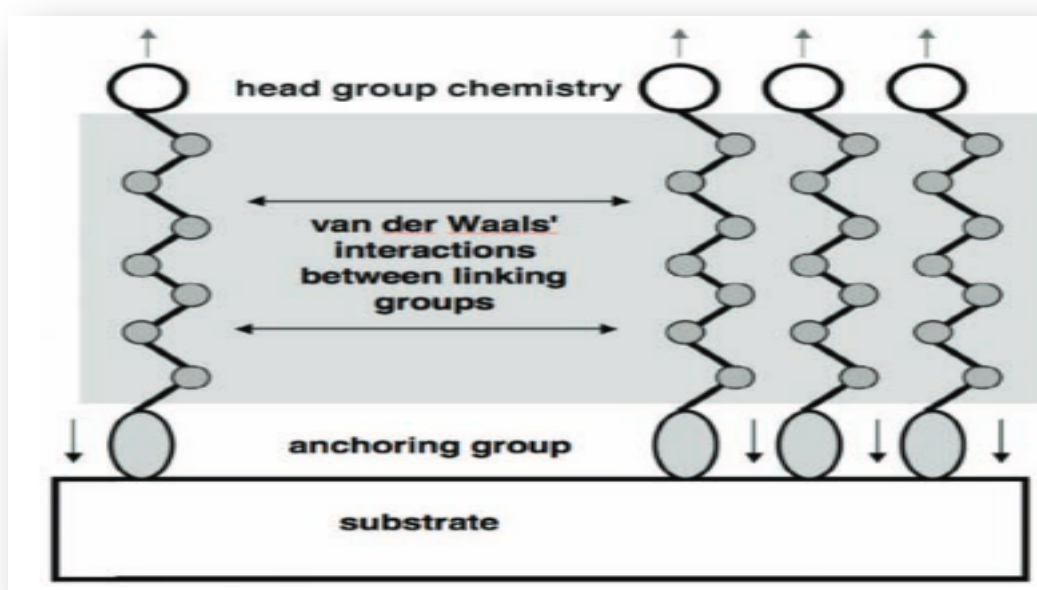


Figure 21. Diagram of the self-assembled monolayer and the interactions between the molecule chains. (Spencer. 2011, 3.)

The chose of the anchoring group depend on the substrate type. The most studied SAMs are alkanethiols (Figure 22 on a page 41) on gold, silver, copper, palladium or platinum. Gold is most commonly used for few reasons:

- It is easy to deposit as planar thin film
- it is reasonably inert to oxidation
- gold readily binds organothiols. (DiBenedetto et al. 2009.)

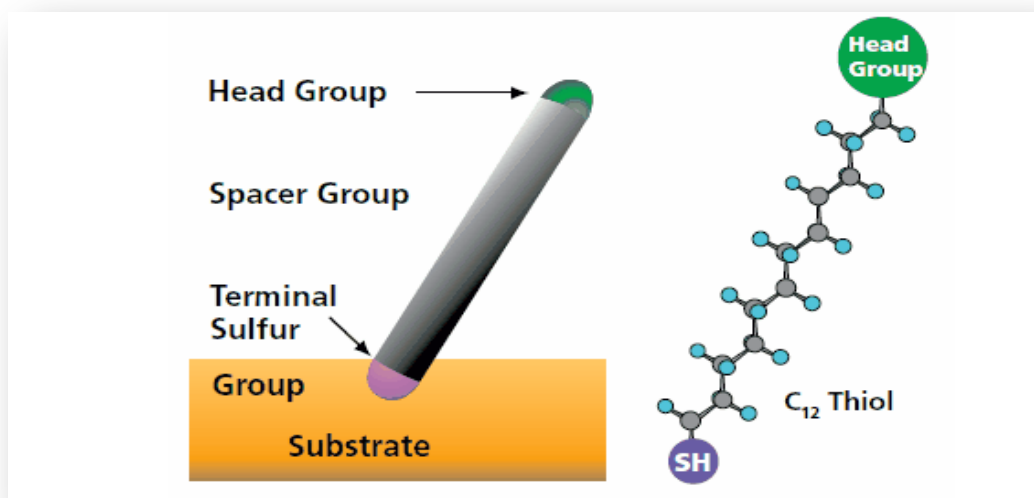


Figure 22. Schematic figure of a thiol molecule (Boeckl & Graham 2006.)

To the structure of the mono- or multilayer can be affected by the choice of the solvent, solution temperature, concentration and the immersion time. Changing the head group provides different properties:

- A hydrophobic surface with methyl head group
 - Hydrophilic surface with hydroxyl or carboxyl head group
 - Protein resistant head group with ethylene glycol
 - Surface that allows chemical binding with carboxyl or amine head groups.
- (Boeckl & Graham 2006.)

There can be found many applications for self-assembled monolayers:

- Surface wetting
- Non-fouling property
- Surface passivation
- Protein binding
- Electrochemistry
- Corrosion resistance
- Molecular electronics (Boeckl & Graham 2006.)

4.5 Aerosol Film coating

An aerosol film coating is a method for a wet film coating. The technology has a high coating quality and it can be operated in atmospheric pressure and room temperature. Among a laboratory scale the aerosol coating method can be used in an industrial scale. The equipment for continuous large-area and high-volume production or batch processing is possible. The application varies from coating of vertical substrates, horizontal surfaces like continuous substrates or separate substrates. (Beneq aerosol thin film coatings 2005-2016)

The method generates ultrafine droplets that are set on the product in the most natural way, by gravitation. This aerosol wet film coating is developed for deposition of wet-based chemistries. The nFOG™ method allow deposition of structured and curved materials and also highly sensitive material because the process is contact free. (Beneq aerosol thin film coatings 2005-2016) Figure 23. below shows the structure of the nFOG™- equipment. The streams of carrier gas and liquid mixture are impacted in the nozzles forming an aerosol fog.

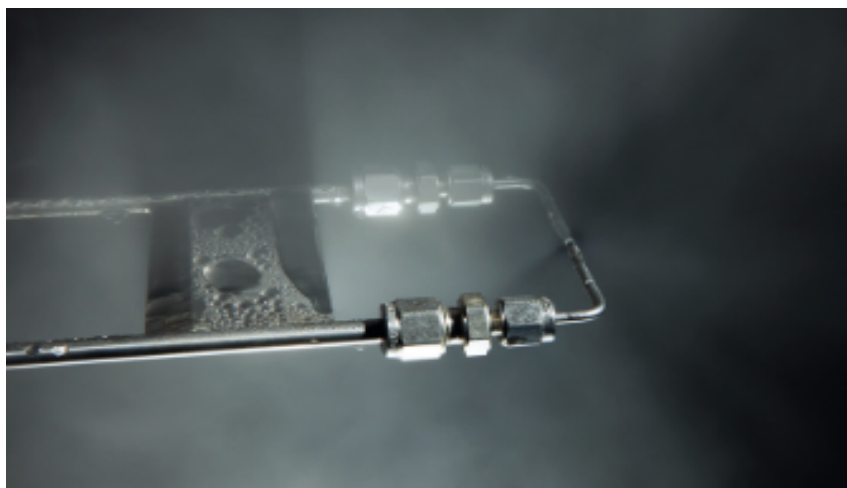


Figure 23. The nozzles of the nFOG™- equipment (ACS 200 2005-2016.)

5 TRIALS

This chapter includes report of the pilot runs and conclusion of the trials. The numeral trial information is presented in a table 2.

5.1 Pilot runs

In the pilot runs different combinations of binders and additives were tested. The main focus in this trial was to find out if the aerosol coating equipment was able to spray material (binder emulsion) with relatively high solid content. Secondly the interest was on the coating quality.

The first part of the trial information is shown in a table 2. Rest of the information can be found from appendices 1 and 2. The table 2. is presenting information about chemicals and their solid content for each trial. The chemicals were different polymer emulsions binders (chemicals 1-3) and a flame retardant chemical (chemical 4). Some of the binder chemicals have high viscosity.

Table 2. Trial information. The binders used in each trial and the solid content values

Number	Liquid l/h	Gas l/min	Liquid pressure/ bar	Material	Solid content %
1	1.8	30	4.8	Chemical 1 (SC 21 %)	
2	1.8	30	6.5	Chemical 1 (SC 42 %)	
3	2.2	30	7.1	"	
4	3	30	8.1	"	
5	3	35	8.9	"	
6	3.5	35	9.7	"	
7	1.8	30	5	Chemical 1 60 % + Chemical 2 40 %	15
8	2.2	30	5.2	"	15
9	3	30	5.8	"	15
10	1.8	30	5.5	"	25
11	2.2	30	5.8	"	25
12	3.5	30	7.1	"	25
13	3.5	35	8	"	25
14	1.8	30	6.8	"	35
15	2.2	30	7.3	"	35
16	3.5	30	9	"	35

Before each trial the binder volumes were calculated in order to the instructions. The variable parameter was solid content of the binder mix and the ratio between the chemicals was settled (see table 2.). The binders were mixed in a mixer (Figure 24.).



Figure 24. The Mixing of the binder solution.

The trials were made with the pilot scale wet laid machine of Ahlstrom Tampere plant. The aerosol coating equipment was set on the sidebars of the forming wire just after the headbox (in figure 25 on a page 45). The gas used in the trial was nitrogen gas from a gas bottle and the flow rate of the gas was controlled with a rotameter.

In this trial the base material was formed earlier in the production so the headbox was out of use. The base material was fed manually in to the aerosol chamber where it was coated. The speed of the forming wire was 3 m/min during all trials.

In the trial the aerosol binder condensed on the ceiling and on the walls of the chamber and eventually that caused droplets on the product. The droplets can be seen in a figure 26 on a page 45. A temporary gutter was build to avoid the droplets. After coating the material was dried in a drier unit and rewound (Figure 26.).



Figure 25. The aerosol coating unit on a forming wire.

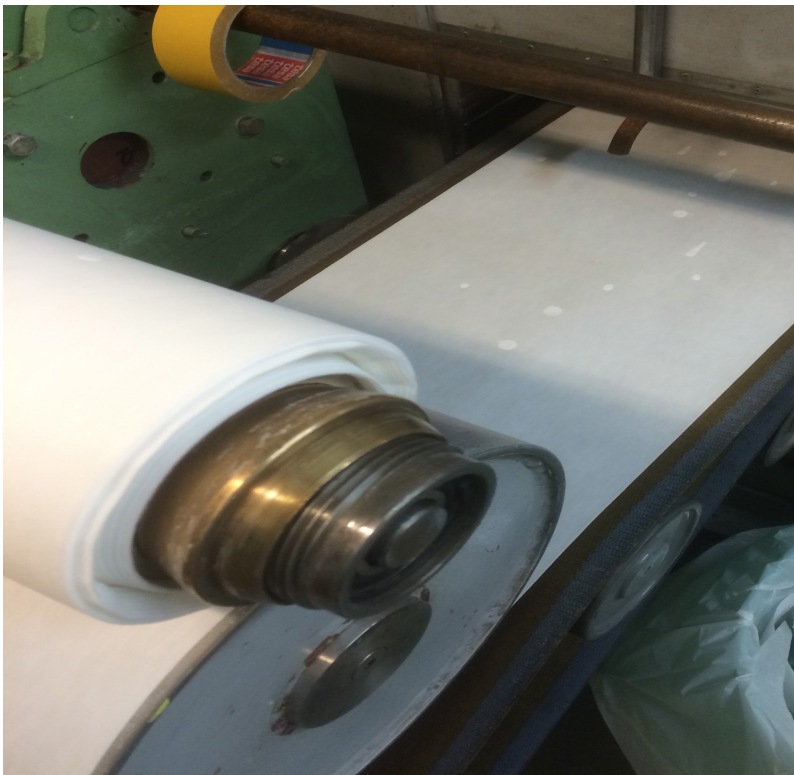


Figure 26. Finished product

5.2 Conclusion of the trials

The trials were successful and everything worked as planned. The trials lasted two days, which was the original plan but all the most important trials were done during the first day. Overall 37 trials were done.

The aerosol equipment worked well. It was able to spray material with high solid content (after small variations were made) and the coating quality was satisfying in all cases. The coating equipment had a small filter before the spraying nozzles and with the highest solid contents that filter was blocked. The filter had to be removed but it did not effect on the coating quality.

During the trials binder fog condensed on the walls of the coating chamber and that caused droplets on the product. A temporary gutter was build from foil tape and that reduced formation of the droplets. The aerosol coating chamber was set on the side bars of the forming wire and two small dewatering boxes were under the forming wire. The under pressure of the dewatering boxes was high enough to keep the aerosol binder in side of the chamber.

The chamber was placed unevenly on the sidebars and a small slit was left in the other side of the chamber. The small slit caused turbulent airstream in the chamber resulting somewhat asymmetric coating. More symmetric placement of the chamber would have solved this problem.

6 THE TESTS AND RESULTS

In this chapter the test methods for the trial materials are introduced as well as some of the results. The rest of the results and other information concerning the trials are presented in appendices 1-3.

6.1 The test methods

After the trials several test were done to examine the quality of the coating and the effects on some basic features for example thickness of the material, basis weight and air permeability. The following test were done for all test materials:

- Air permeability
- Thickness measurement
- Basis weight
- Pressure drop across the filter media
- Colour indication test for the binder chemical

The trial material that was coated with volume flow rate 1,8 l/h had porosity and pore size tested. The pore size was tested with the Mean flow pore size (MFP) equipment, which automatically determines maximum, minimum and the average pore size. The results from MFP are shown in the table 4. Flammability test was done for the material that had flame retardant in the coating.

6.2 Base material results

The base material was fully synthetic filter media. The structure of this material is less dense and the air permeability is rather high. Basis weight was around 71 g/m² and thickness ~ 600 µm. Pressure drop (DP in the table 3.) was also measured and for this material type pressure drop is low. For less dense materials the pressure drop will be lower. When determining collection efficiency pressure drop is quick way to determine

material properties. The results of the base material are shown in a table 3. Table 3. shows also the minimum, maximum, average and the standard deviation value.

Table 3. Results of the base material

	Basis weight g/m ²	Air permeability l/s/m ²	Thickness nessμm	DP (Pa)
Base material	71.6	1480	562	9
	72.8	1480	589	9
	70.8	1520	578	8
	71.4	1480	598	9
	71.6	1530	601	9
Min	70.8	1480	562	8
Max	72.8	1530	601	9
SD	0.73	24.9	15.9	0.4
Avg.	71.6	1498	585.6	8.8

6.3 Test results for coated materials

The first trial complex (trial numbers 7-19) comprehends from materials that were coated with two synthetic binders (chemical 1 & 2). The ratio between the binders was constant and the solid content was variable. Results from the tests are shown in a table 4. and the values are average values. The last column in the table shows solid content values. When the solid content gets higher value the basis weight also increases and so does thickness. This is reasonable change since the material content versus the water content is bigger and also coating volume flow rate effects on the basis weight and thickness.

Table 4. Average results for test materials coated with binder mix (chemical 1&2).

The average test results for each trial. Binder combination: Chemical 1 60 % + Chemical 2 40 %						
Trial number	Volume flow rate (l/h)	Basis weight (g/m ²)	Air permeability (l/s/m ²)	Thickness (μm)	Pressure drop DP (Pa)	Solid content (%)
7	1,8	75.8	1348	566	11,2	15
8	2,2	77.5	1308	567	11,3	15
9	3	78.3	1322	596	11,3	15
10	1,8	77.6	1338	564	11	25
11	2,2	80.6	1252	567	11,6	25
12	3,5	80.4	1273	564	11,6	25
13	3,5	79	1308	594	11,3	25
14	1,8	80.8	1245	566	11,5	35
15	2,2	83	1262	571	12	35
16	3,5	82.9	1253	583	12	35
17	3,5	80.2	1400	591	11	35
18	1,8	79.9	1350	627	11	45
19	2,2	75.9	1452	593	11	45

The following bar charts are presenting the measured values. Each chart shows the test results with different volume flow rate. For example the figure 27 shows results for the materials that had volume flow rate 1,8 l/h. In the x-axis the results are categorized by solid content. The information in the charts includes: basis weight (g/m²), thickness (μm) and air permeability (l/s/m²). In the first chart (Figure 27.) the data of base material is included as a reference.

Figure 27. on a page 50 shows results for material coated with binder chemicals 1 and 2. This flow rate value was the lowest and was easily sprayed. Measured values are quite similar between each solid content group only basis weight is increasing little bit. When compared to the base material we can notice that basis weight is little higher and the air permeability is lower. The coated layer was very uniform and that can explain the change in air permeability.

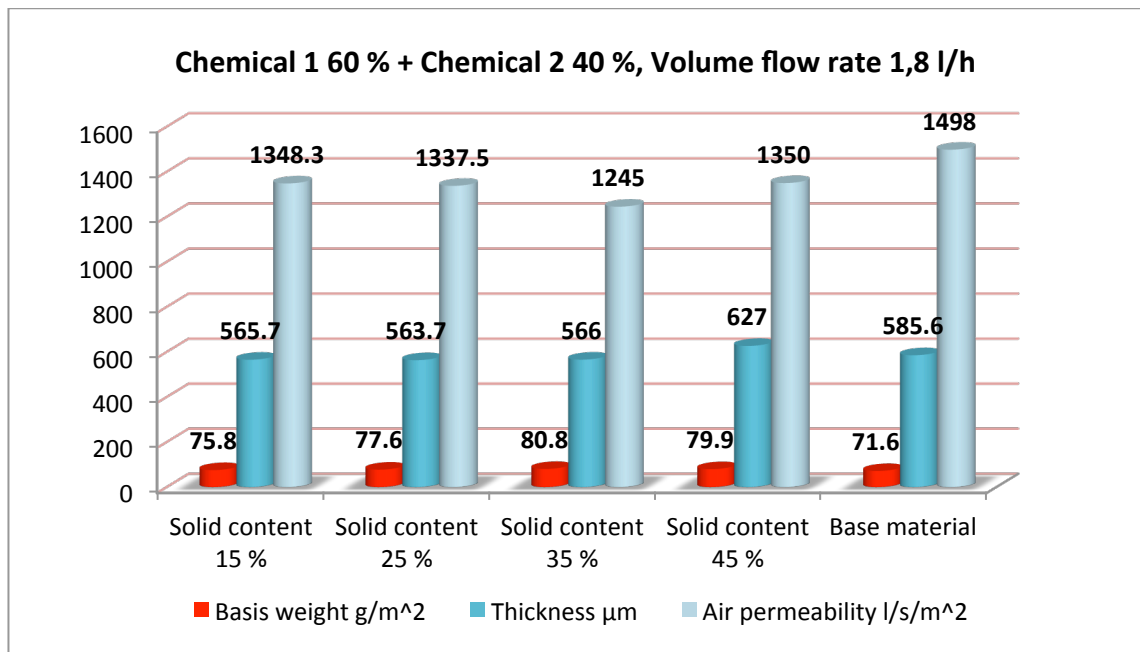


Figure 27. The test results for trial material (Chemical 1 60 % + Chemical 2 40 %. Volume flow rate 1,8 l/h)

Figure 28. shows the results for material that was coated with volume flow rate 2,2 l/h. As well as in earlier figure 27. the basis weight is generally increasing when the solid content increases. The peak in the air permeability of the last group (solid content 45 %) could be explained with particle agglomeration in the coating or with the uneven formation of the base material.

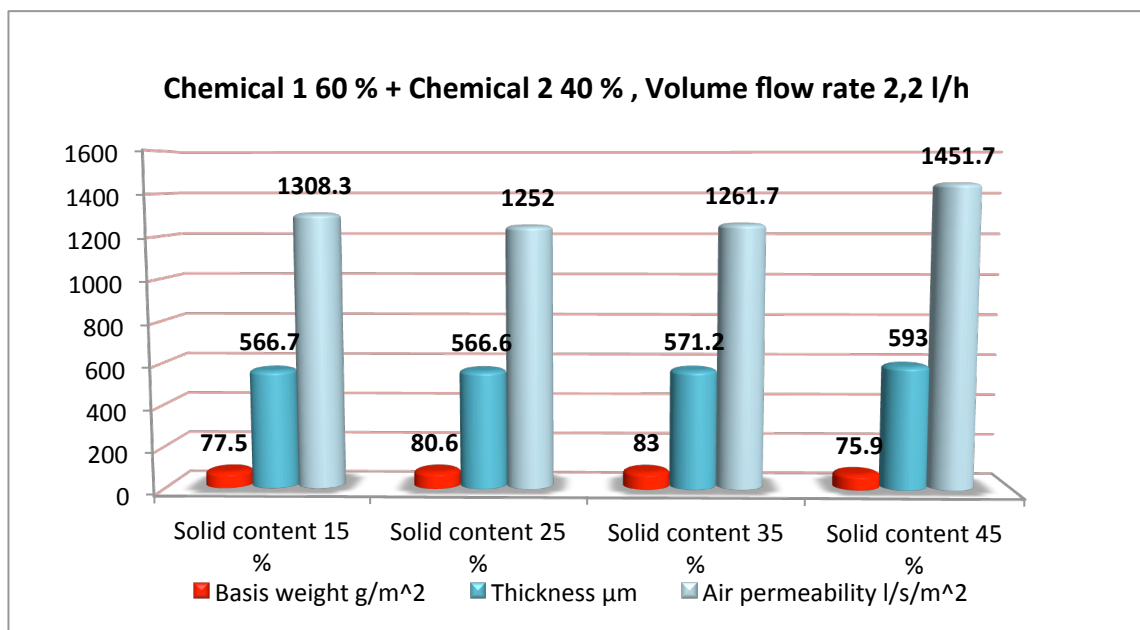


Figure 28. The test results for trial material (Chemical 1 60 % + Chemical 2 40 %. Volume flow rate 2,2 l/h)

The last chart (Figure 29.) presents results for material coated with volume flow rate 3-3,5 l/h. As can be seen from the chart basis weight is higher when compared to the to charts presented earlier (Figure 27. and 28.) and it can be explained with the higher volume flow rate.

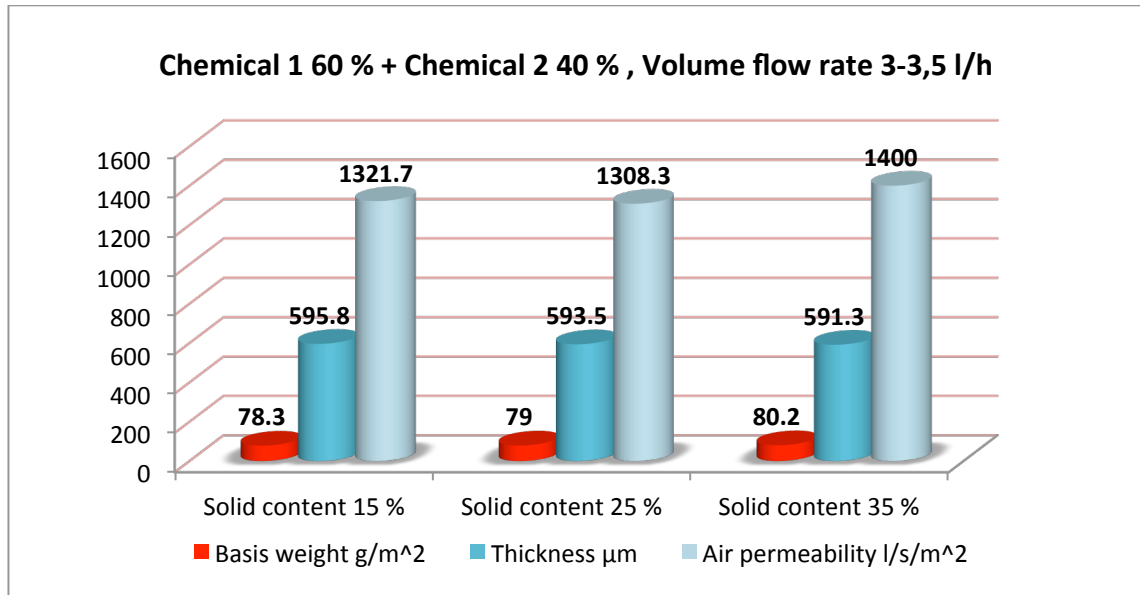


Figure 29. The test results for trial material (Chemical 1 60 % + Chemical 2 40 % . Volume flow rate 3-3,5 l/h)

Table 5. Average results for test materials coated with Chemical 3 50 % + Chemical 4 50 % -binder mix.

The average test results for each trial. Binder combination: Chemical 3 50 % + Chemical 4 50 %						
Trial number	Volume flow rate (l/h)	Basis weight (g/m ²)	Air permeability (l/s/m ²)	Thickness (µm)	Pressure drop DP (Pa)	Solid content (%)
23	1,8	84	1345	609	9,2	25
24	2,2	84,3	1368	603	9,3	25
25	3	84,5	1362	600	9,8	25
26	3,5	84,8	1377	644	9,7	25
27	3,5	78,9	1345	585	10	25
28	1,8	81,1	1365	584	10	30
29	2,2	83,7	1367	622	10	30
30	3,5	86,8	1336	632	10	30
31	3,5	80,4	1382	585	10	30
32	1,8	81,2	1374	585	10	35
33	2,2	83,7	1382	616	9,7	35
34	3,5	74,1	1575	646	10	35

The table 5. on a page 51 shows the average test results for trial material coated with a latex binder (chemical 3) and flame retardant chemical (chemical 4). The ratio was between these chemicals was constant in each trial. The variables were solid content and volume flow rate. The following bar charts (figure 30 & figure 31) illustrate the results presented in the table 5.

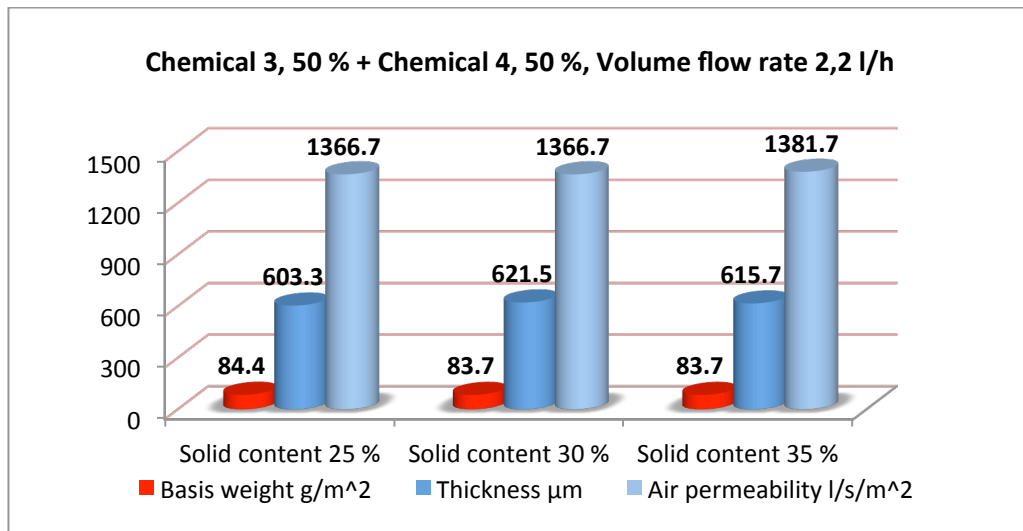


Figure 30. The test results for trial material (Chemical 3 50 % + Chemical 4 50 %, Volume flow rate 2,2 l/h)

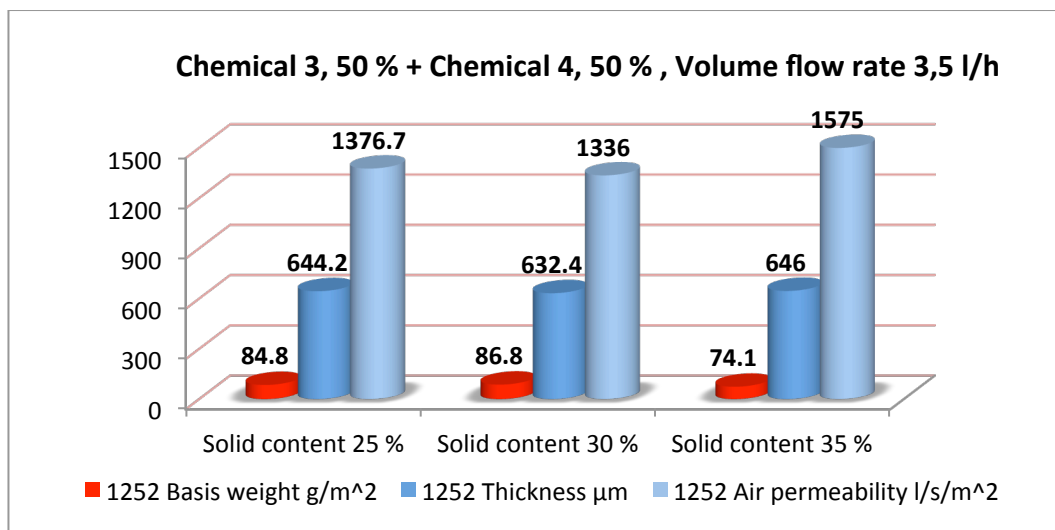


Figure 31. The test results for trial material (Chemical 3 50 % + Chemical 4 50 %, Volume flow rate 3,5 l/h)

Bar charts (in the figure 30. and 31.) shows that the results between each solid content group are nearly equal. This can be seen also from table 5. When comparing the results of table 5. to the results of the table 4., it can be noticed that results in the table 4.

(Chemical 1 60 % + Chemical 2 40 % -coated samples) have a bit more variation in values.

6.4 Flammability tests and results

Material with flame retardant chemical (Chemical 4) had flammability test done. The test was done according to DIN 53438 standard. In this test the test sample is placed to metal holder, which is placed in fume hood. The flame angle is 45 °. In this test the aim is to observe the burning distance (max. 150 mm) and time. Also burning behaviour is observed. The desired result of this test is when the flame does not reach the upper measuring mark or the flame has extinguished (Flame retardants, 6).

Table 6. Flammability test results. Material sprayed with solid content of 25 %

Flammability test results			
23) 1,8 l/h	Burning time / s	Burning distance/ mm	Comments
	5	150	The whole sample burned
	25	110	
	30	150	
	25	130	
	40	140	Burned only from other side
24) 2,2 l/h			
	38	150	Burned only from other side
	45	150	The whole sample burned (brightly)
	50	150	
	40	150	The whole sample burned
25) 3 l/h			
	27	150	
	32	140	
	40	150	
	30	110	
	25	150	
26) 3,5 l/h			
	50	150	The whole sample burned
	22	105	
	93	150	The whole sample burned
	70	150	The whole sample burned
27) 3,5 l/h			
	57	100	
	17	100	Burned only from other side
	35	100	
	59	150	The whole sample burned

Almost all samples burned 150 mm distance and some samples were burned totally. Some samples burned with a big flame in a few seconds and then again some samples

burned very slowly with small flame. Table 6 shows the results for samples that were aerosol coated with solid content of 25 %. Rest of the results are shown in the appendix 4. The used volume flow rate varies between the samples. Only a very small difference can be seen in the burning time when comparing the samples.

The samples burned slowly which was positive result. Normally products treated with flame retardant chemicals are coated from both sides. The results were positive considering that the material was coated only from one side.

6.5 Mean flow pore size test results

MFP –results are shown in a table 7. The pore size was tested for trial materials that were coated with volume flow rate 1,8 l/h. The results were surprisingly equal. The test values and the standard deviation values indicate uniform coating quality.

Table 7. Test results pore size

Mfp measurement				Buble point		
	Min pore side/ μm	Max pore size/ μm	Mfp	Flow/ l/min	Pressure / bar	Air permeability l/m ² /s
4)	14.5	37.4	26.5	1.1	0.017	1210
	14.5	37.9	26.5	0.77	0.017	1250
8)	14.5	37.4	26.5	0.99	0.017	1260
	14.5	37.9	26.5	0.8	0.017	1330
11)	14.5	38.4	28	0.76	0.017	1260
	14.5	38.4	28	0.9	0.017	1280
14)	14.5	37.9	28	0.91	0.017	1290
	14.5	37.4	26.5	1	0.017	1310
18)	14.5	37.9	28	1.1	0.017	1420
	14.5	39	28	0.52	0.016	1380
20)	14.5	37.9	28	0.9	0.017	1350
	14.5	38.4	28	1.2	0.017	1350
23)	14.5	37.4	26.5	0.9	0.017	1390
	14.5	37.9	28	1.2	0.017	1340
28)	14.5	37.9	28	0.84	0.017	1320
	14.5	39	28	0.76	0.016	1310
32)	14.5	39	28	0.75	0.016	1360
	14.5	37.4	26.5	0.97	0.017	1360
Min	14.5	37.4	26.5	0.52	0.016	1210
Max	14.5	39	28	1.2	0.017	1420
Avg	14.5	38.03	27.42	0.91	0.017	1321
SD	0	0.6	0.8	0.2	0.0004	55

6.6 Results from colour indication test

Colour indication test is easy and fast way to examine the samples. Only the acrylate binder (chemical 1) dyes by the blue dye. This can be noted from other samples since they appear lighter than samples with acrylate binder.

The figure 32. below shows colour indication test in action. The sample was sunk in the dye for 15 minutes, rinsed with water and dried. The result can be seen immediately.

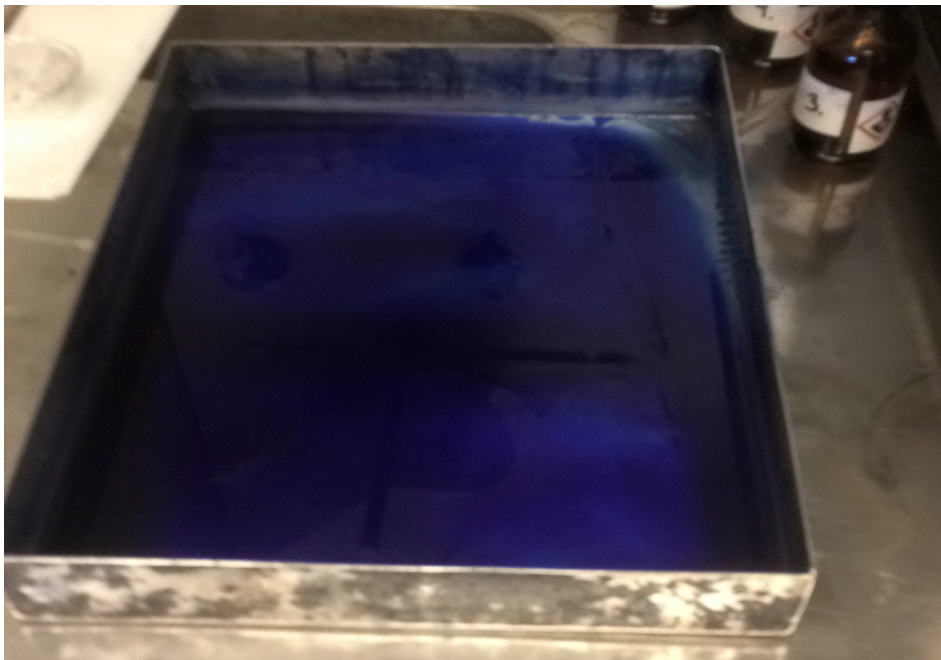


Figure 32. Colour indication test

The next figure (Figure 33 on page 56) shows test samples of colour indication test for base material on the left and the sample coated with binder chemical 1. The binder was sprayed with 42 % solid content. (See table 2.) The blue cloud-like figure in the base material sample indicates acrylate binder. It can be noticed that the binder has set rather unevenly on the material. The sample on the right seems very uniform when compared to the base material.

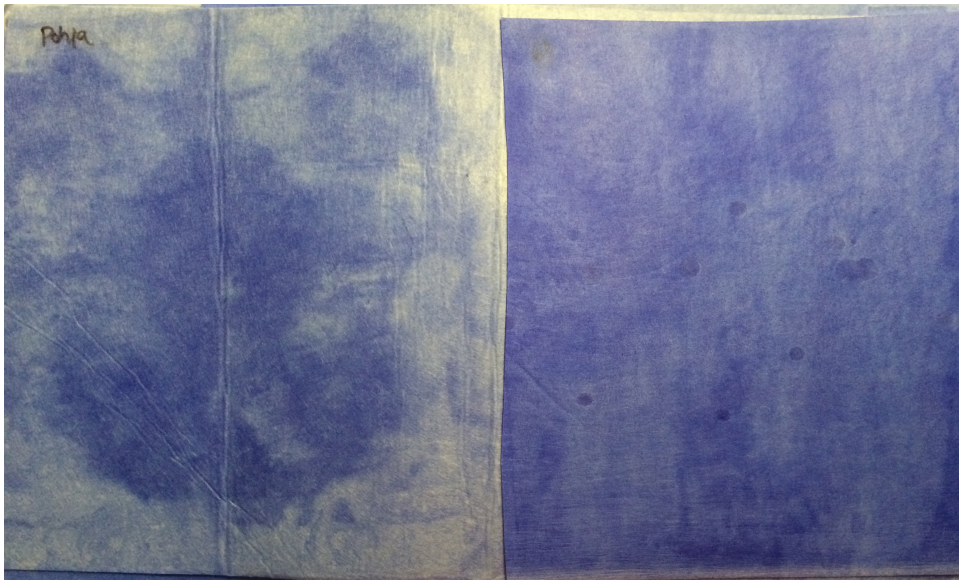


Figure 33. Test samples of colour indication test. Base material on the left and chemical 1 coated sample (trial number 6) on the right

In the next figure (Figure 34.) there is base material compared with sample coated with chemical 2. The sample of trial number 20 seems very light and that is because the used chemical doesn't react with the dye as acrylate chemical does. The uniform white colour indicates uniform coating quality. Some blue colour can be seen from the background.

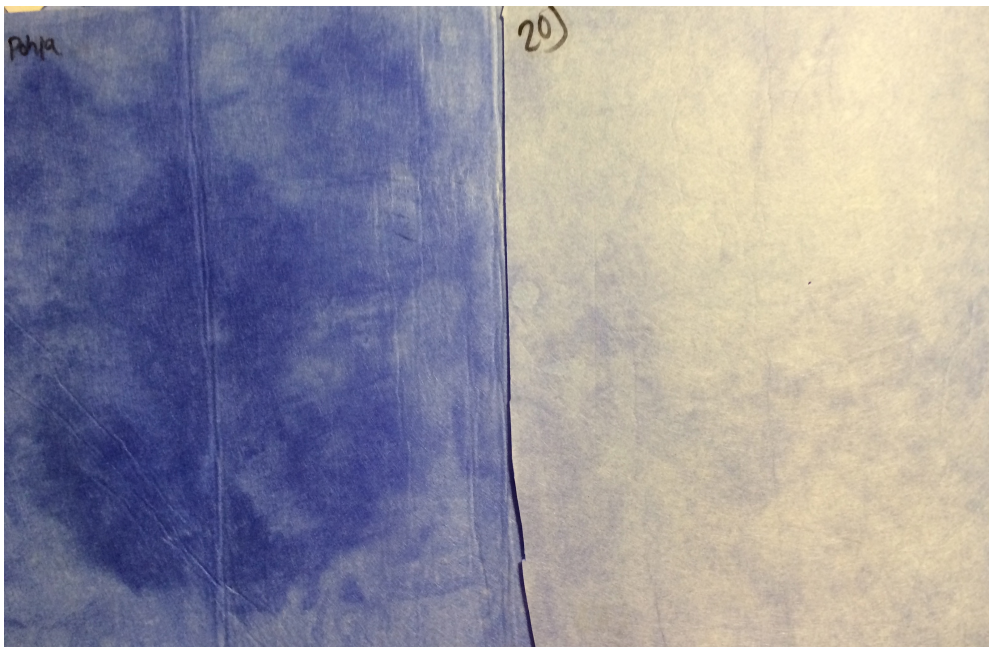


Figure 34. Test samples of colour indication test. Base material on the left and chemical 2 coated sample (trial number 20) on the right

In the figure (35.) is a comparison of samples with binder chemical 2 and an acrylate binder. Again the clear coverage of the base material can be noticed. The spots formed during the trial can be seen on the blue sample.

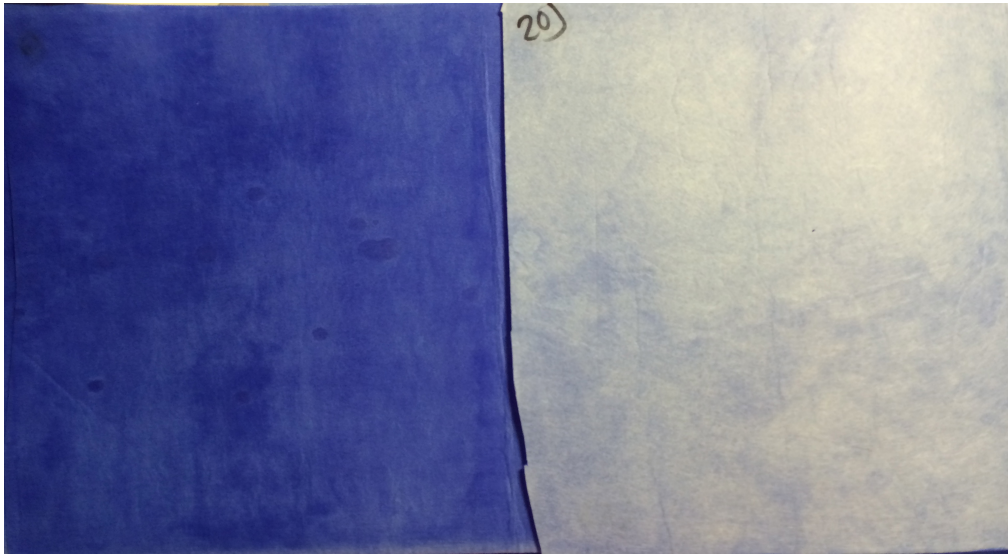


Figure 35. Test samples of colour indication test. The sample coated with chemical 1 (trial number 6) on the left and the sample coated with chemical 2 on the right

In the figure (36.) below is a folded sample coated with chemical 2. The sample is coated on one side and therefore the backside of the sample looks light blue. The blue colour of the backside recovers acrylate binder.



Figure 36. On the left chemical 2 coated surface and on the right background of the sample

7 CONCLUSIONS AND DISCUSSION

The purpose of this thesis was to test new type of equipment for applying binder chemicals and other emulsion chemicals on wet laid nonwoven material. The goal was to find out if the aerosol coating equipment was able to function satisfyingly and produce good coating quality. Several Chemicals with different solid content values were tested.

It was interesting to notice that the equipment worked well in all circumstances. Some small setbacks occurred but the effects of these setbacks were minor and fixable. In a trial session the aerosol fog condensed on the walls of the coating chamber and that caused droplets on the product. This fault was fixed by building a temporary gutter. The formed droplets were not effecting on the measurement of the samples.

The test results were clearly positive. In all samples the coated layer was thick and on some samples the coated layer was almost 20 g/m^2 . The effect of the thick extra layer on air permeability was surprisingly low. The conclusion is that the pores of the filter media were not blocked and therefore air permeability was not reduced enormously. The results also refer to fine and uniform settling of the binder particles and the results of the mean flow pore size test supports this fact.

Colour indication test recovers acrylate binder in samples. The sample of base material and other samples aerosol coated with acrylate binder and acrylic binder were compared. As a result uniform settling of the polymer particles in the aerosol-coated samples were noticed. The trade of acrylate binder in the base material is uneven and the binder forms cloudy pattern.

Results from flammability tests were also very positive. All test samples burned the set test distance of 150 mm but the burning speed was slow. For most samples it took more than 30 seconds to burn totally.

The R&D department of Ahlstrom Tampere and Beneq Corporation will possibly continue in this matter. The next stage would be scaling the aerosol coating method according Ahlstrom's specifications.

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APPENDICES

Appendix 1. The trial information (Part 1)

Date 13.4.2016

Number	Liquid l/h	Gas l/min	Liquid pressure bar	Material	Solid content %
1	1.8	30	4.8	Chemical 1 (SC 21 %)	
2	1.8	30	6.5	Chemical 2 (SC 42 %)	
3	2.2	30	7.1	"	
4	3	30	8.1	"	
5	3	35	8.9	"	
6	3.5	35	9.7	"	
7	1.8	30	5	Chemical 1 60 % + Chemical 2 40 %	15
8	2.2	30	5.2	"	15
9	3	30	5.8	"	15
10	1.8	30	5.5	"	25
11	2.2	30	5.8	"	25
12	3.5	30	7.1	"	25
13	3.5	35	8	"	25
14	1.8	30	6.8	"	35
15	2.2	30	7.3	"	35
16	3.5	30	9	"	35
17	3.5	35	10	"	35
18	1.8	30	9.8	"	45
19	2.2	30	10.8	"	45
20	1.8	30	6.6	Chemical 2 100 %	20
21	2.2	30	7.1	"	20
22	3.5	30	8.9	"	20
23	1.8	30	5.8	Chemical 3, 50 % + Chemical 4 50 %	25
24	2.2	30	6.1	"	25
25	3	30	6.9	"	25
26	3.5	30	7.3	"	25
27	3.5	35	8	"	25

Appendix 2. Trial information (Part 2)

Date 14.4.2016

Number	Liquid l/h	Gas l/min	Liquid pressure bar	Material	Solid content %
28	1.8	30	6.9	Chemical 3, 50 % + Chemical 4 50 %	30
29	2.2	30	7.3	"	30
30	3.5	30	8.8	"	30
31	3.5	35	9.5	"	30
32	1.8	30	7.6	"	35
33	2.2	30	8.2	"	35
34	3.5	30	10	"	35
35	2.2	30	5.1	Chemical 5 100 % (on both sides)	3
36	2.2	30	5.4	Chemical 6 70 %+ Chemical 1 30 %	17
37	3.5	30	6.2	"	17

Appendix 3. Flammability test results of samples 28-33

Flammability test results			
Flammability test results			
28) 1,8 l/h	Burning time / s	Burning distance/ mm	Comments
	42	150	The whole sample burned
	33	130	
	44	85	
	59	150	The whole sample burned
29) 2,2 l/h			
	31	85	Burned only from other side
	47	105	The whole sample burned
	43	150	The whole sample burned
	42	150	
30) 3,5 l/h			
	30	150	
	34	80	
	42	150	The whole sample burned
	23	100	
	60	150	The whole sample burned
31) 3,5 l/h			
	21	105	
	94	150	The whole sample burned
	49	150	The whole sample burned
	71	150	The whole sample burned
32) 1,8 l/h			
	35	120	
	23	100	
	73	150	The whole sample burned
	45	77	Burned only from other side
	25	110	
33) 2,2 l/h			
	53	150	
	48	150	The whole sample burned
	68	150	The whole sample burned