

PARTICLE MEASUREMENT WITH PM10 IMPACTOR AND IMAGE ANALYSIS IN POLYTEST PROJECT

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ABSTRACT

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INKA MÄKIPÄÄ: Particle Measurement with PM10 Impactor and Image Analysis in Polytest Project

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ABSTRACT

The Polytest project was started in 2006 in Tampere University of Applied Sciences. The aim of the project was to develop a device for measuring paper dusting and linting in both laboratory circumstances and in online measurements in paper production lines and in printing houses. Due to the application phase of patents concerning the dust collecting method the earlier thesis have been classified confidential.

There have been many development steps during the project and the focus of this thesis was to get reliable data from different paper grades and from more extensive data sets. The tests in the spring 2013 consisted of statistical data analysis and of PM10 measurements and optical data analysis. The statistical data analysis was to be discussed in further details in the thesis of Taye Hailemariam and this work concentrated on the PM10 measurements and optical data analysis.

The method based on detaching particles from paper surface with sound waves and particles were sucked through a dust collecting unit into the measuring device. In preliminary tests for statistical analysis the particles were analyzed by optical boulder counter. PM10 impactor was used in the measurements for mass and image analysis. The objective was to determine the mass distribution with both methods and the proportion of detached particles that exceeded the scale of optical boulder counter with image analysis. Also the nature of the particles was analyzed with optical methods.

In image analysis the results showed a clear difference between papers both in particle amount and different type of particles. Fiber-like particles could be recognized and counted but further analysis should be made with electronic microscope for distinguishing the nature of smaller particles. In PM10 mass measurements the size of the sample was not extensive enough for conclusive results. There should be more extensive samples used for acquiring reliable data. The results are in correlation to the properties of different paper grades thus the method is suitable for measuring linting and dusting.

TIIVISTELMÄ

Polytest-projekti aloitettiin Tampereen ammattikorkeakoulussa vuonna 2006. Tavoitteena oli kehittää laite, joka mittaa paperin pölyävyyttä sekä laboaratoriossa että reaaliaikaisesti paperitehtaissa ja painotaloissa. Patenttien hakuvaiheessa tehdyt opinnäytetyöt ovat luottamuksellisia.

Projektissa on ollut erilaisia kehitysvaiheita ja tämän työn tavoitteena oli kerätä luotettavaa tietoa eri paperilaaduista kattavalla otannalla. Keväällä 2013 tehtiin tilastollista analyysia, mittauksia PM10 impaktorilla sekä analysoitiin kuvia. Taye Hailemariam keskittyy opinnäytteessään tilastolliseen analyysiin ja tämä työ käsittelee PM10 -mittauksia ja kuva-analyysia.

Pölynmittausmenetelmässä hiukkaset irroitettiin ääniaalloilla ja imettiin pölynkeräysyksikön läpi mittauslaitteeseen. Alustavissa kokeissa ja tilastollisessa analyysissa hiukkaset analysoitiin optisella boulder counter -mittauslaitteella. PM10 impaktoria käytettiin massan mittauksessa ja kuva-analyysissa. Tavoitteena oli selvittää massajakauma molemmilla metodeilla sekä arvioida kuva-analyysilla hiukkasten määrä, joka ylittää optisen mittauslaitteen mittaskaalan.

Kuva-analyysissa havaittiin eroja hiukkasten määrässä ja laadussa eri paperilaatujen välillä. Pienten hiukkasten erottamiseksi ja tunnistamiseksi tulisi kuitenkin tehdä lisätutkimuksia elektronimikroskoopilla. Massamittauksessa luotettavan tuloksen saaminen edellyttää laajempaa otantaa.

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1 INTRODUCTION

Accumulation of loose material onto printing blanket and inking unit is called linting. The fibers origins come from the surface of the paper in the printing unit and the problem is most often seen in offset printing. Dusting is also a problem in printing and the phenomena is caused by loose pigments in the paper surface, such as accumulated particles in the surface from, for example, cutting processes.

Both linting and dusting cause loss of details in the printed image and especially in the sharpness of the image due to disturbed ink transfer between the printing blanket, printing plate and paper. Eventually the accumulation of the particles will lead to interruption of the printing process while the blanket or the inking unit is washed. (Suontausta, 1999, 198.)

The main problem is that with current technology there are no means to test linting propensity during the production of paper or at least it is not possible to do it in real time. This means that the production cannot be adjusted flexibly if problems have been registered.

2 DUSTING AND LINTING IN PRINTING AND PRODUCTION

Dusting and linting cause problems in both printing and production. The majority of the problems are seen in the printing houses but the tools for lessening the problem are found in the paper production phase. Dusting and linting can be seen as a mutual enemy: the printing houses require paper with good quality in order to keep the processes running and the producers need to meet the demands and keep the customers satisfied.

2.1 Different methods of particle release

Linting is a term referring to loose fiber or pigment particles released from the surface of the paper. In printing the particles accumulate in water, inking systems and on the printing plates. Linting is a serious problem in the printing process, as it causes the need to stop printing for cleaning purposes at regular intervals. The breaks in the process lead to production losses and decreasing the quality of prints. Uncoated, non-surface-sized wood containing offset paper grades are typically prone to linting. Newsprint and SC offset are examples of these paper grades. (Oittinen & Saarelma, 2009, 118-120.)

In addition to linting, picking is a paper property of very similar type. The difference between linting and picking is that the particles released in picking are at least partly bonded. Picking problems are caused by tacky ink and the particles accumulate onto the printing blanket. Picking problems are caused by either poor surface strength or fast setting of the ink or both at the same time. Picking can be detected by particle size and quality with fewer amounts of papers and in sheet fed printing the results can be analyzed with about 200 paper sheets. By counting the number of picks on the printed sheets the coating and fiber pick can be analyzed. (Suontausta, 1999, 198.)

Dusting is quite similar phenomena but the particles have been accumulated on the paper in previous process steps, such as in cutting sheet or web. (Oittinen & Saarelma, 2009, 118-120.) In addition to accumulated particles, dust is referred as detaching particles smaller than in linting. The material consists mainly of stone based coating pigments and fillers of the length of 0.1 to 5.0 micrometers. (Aittamaa, 2007, 12.)

2.2 Problems caused by dusting and linting

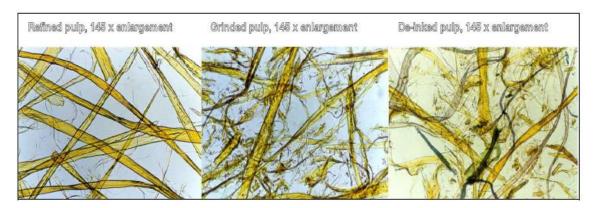
Runnability is an important feature in printing, copying and paper finishing operations. These operations can be seen as a definer of production efficiency. In production the efficiency is measured by number of papers produced per unit of time, but runnability is mostly examined as runnability problems after the product has left the manufacturing site. Runnability problems can be caused by mechanical, chemical or physico-chemical stress, which may lead to changes in the properties of the paper. The amount of the problems usually increases with high running speed. The runnability problems can be divided into three categories: sudden, piling and cumulative problems. Sudden disturbance causes production break and is usually discrete, such as web break. Piling disturbance leads to production breaks at intervals which to some extent can be timed freely. Dusting and linting belong in the last category of disturbances. (Oittinen & Saarelma, 2009, 111-112.)

There are three factors in the mechanism that causes linting. Loose dust particles from the paper surface can detach and adhere to the rubber blanket. The rubber blanket is the first printing unit in offset printing. The stickiness of printing inks may result the upraising of fibers or releasing fiber bundles or the formation of the paper during the printing process (Knowpap, 2012). The splitting resistance caused by the ink can detach fibers and fiber bundles which gather in the printing nip. This mechanism has also biggest effect in the first printing units. The material accumulates on the edges of printing surfaces such as halftone dots and characters. Depart from the previous mechanisms dampening water has biggest impact in the last printing units. Dampening water weakens the fiber bonds and breaks the fibers. Linting is cumulative nature which makes the measurement in laboratory complicated. (Oittinen & Saarelma, 2009, 119.)

In offset printing the problems are biggest in middle tone and half tone areas. The lintaccumulating capacity is caused by the length of the edge surface between the printing and non-printing surface. In addition to offset printing also electrophotography and flexo printing have problems with surface strength. In electrophotography printing the stress is caused by the fixing of a hot roller. In flexo printing the mechanism of drying ink partly on the printing plate causes mechanical stress on the paper surface. (Oittinen & Saarelma, 2009, 118-119.)

2.3 Raw material and linting

Sufficient paper strength properties are required for good runnability. The surface strength equals to the surface layer's ability to withstand the stress caused by tacky printing ink. The raw material composition and different processes can be used to increase the strength properties and decrease linting and dusting propensity. The composition of some pulps with different processes are presented in picture 1. (KnowPap, 2012.)



PICTURE 1. The composition of few pulps (KnowPap, 2012)

Bonding strength is affected both by processes and raw material used in the paper. In the paper production surface sizing is applied to fine papers, coated base papers and paper boards to improve surface strength and thus decrease dusting. In surface sizing the bonds between fibers are reinforced with a water soluble binding agent, usually starch. The binders decrease the movement of the particles. (KnowPap, 2012.)

Paper strength is caused by hydrogen bonds between fibers. When fiber material is replaced with a filler of poor bonding capacity, the paper strength can decrease and cause linting in the dry end of the paper machine and also in printing machine. With flat filler distribution of fillers slightly descending from the surface the linting problems can be decreased. Coarse grained fillers are more prone to create linting than fine grained fillers. Nevertheless, fillers are used for a reason of the lower cost than the fiber raw materials and they also improve optical properties of the paper. Most common fillers in use are calcium carbonate, kaolin, talc, titanium dioxide and synthetic silicates. (KnowPap, 2012.) In newsprint the fiber length needs to be high to ensure sufficient strength. The share of fines needs to be high and the amount of splinters reduced as thoroughly as possible to reduce web breaks in the production and also to decrease linting propensity in the printing phase. (KnowPap, 2012.)

2.4 Production processes and linting

Most common additional process in paper production is calendering. In calendering the paper web runs through one or more nips made of steel or iron. The purpose of calendering is to adjust paper thickness, improve surface properties and obtain smooth and even rolls. Calendering improves printing specific properties, but at the same time it can decrease other paper properties such as runnability and usability. Bonds between fibers can be increased with sufficient moisture content in calendering. From SC offset paper a non-dusting surface is required. In super calendering with sufficient moisture content the fibers of mechanical pulp are bonded. The optimization of calendering process needs to be done according to the end use, that is, to make sure that the smoothing and compacting does not impair critical properties while enhancing the others. (KnowPap, 2012.)

The primary objective of bleaching is to improve the purity of pulp and to increase brightness. In addition, the alkalinity in bleaching process improves the strength of the pulp by increasing sheet density, bonding strength, smoothness and wet strength. (KnowPap, 2012.)

The drying section has also an effect on the fiber detaching. Dryer fabrics support and transport paper web through the dryer section of the paper machine. Uneven support of the fabric against the hot cylinder can often result dusting as well as fabric weaving and heat treatment. If the first cylinders in drying sections have too high surface temperature, there can be technological problems, burning and linting. Too high temperature causes the paper to stick onto the cylinder and causes the appraisal of fibers when the web is detached from the cylinder. (KnowPap, 2012.)

3 MEASURING LINTING AND DUSTING

Different methods are used for measuring linting and dusting but there is no standardized method available. Some methods are simple and quick as the others demand more time and sophisticated methods and devices. During the Polytest project some of the samples have been tested also with other methods. In 2007 Janne Heinilä was evaluating the measurements and the different methods in his thesis. The methods tested were IGT Fluff test, Heidelberg GTO offset printing press and Veitsiluoto device (see chapter 3.6).

3.1 General

Printing process variables affect the printability of the paper. Printability depends on the interaction between the ink and the paper. A good paper grade is not sensitive to the variations of different process variables. The final use determines the print quality definitions. Usually evenness, resolution and print density defines print quality but also many other qualities are in the interest. (Suontausta, 1999, 198.)

Surface strength means the ability to resist the force pulling out fiber bundles or single fibers. If surface strength is low the propensity of linting increases. For testing the strength usually high speed and tacky oil or ink is used. IGT has developed a tester that is frequently used, but generally all these tests are hard to reproduce. (Suontausta, 1999, 198.)

3.2 Common methods

There are various methods in use to measure linting propensity in the paper mills. Few of the most used tests are MB lint tester, Prüfbau pick resistance, IGT pick, Fixpro 1000, black cloth and Apollo press. In Gratton and Wigon's release the commonly used tests are evaluated. Some of the tests available are simple and quick and they are based straightly on the amount of white dust collected from the paper surface. More detailed tests require special laboratories, operators and equipment and are thus rather costly and also time consuming. The simple and quick tests are usually unreliable while the complex tests take too long time to make changes in the production based on the results. From the manufacturer's point of view a suitable test method would be fast, simple and user friendly and it should be repeated easily and performed at the mill without special equipment or arrangement of testing conditions. (Gratton & Frigon, 2006, 1-3.)

In Gartton and Wigon's release there are few aspects brought up considering the commonly used methods. IGT pick test is operator dependent in preparing the samples and grading the results which makes the method time consuming. The picking phase is aggressive and the method is mostly recognized useful for predicting sheet laminating propensity instead of linting propensity. MB Lint tester has not been adopted widely because it does not always predict lint. Some manufacturers have found correlation for linting propensity and have been using the method for years. The Fixpro instrument (FRT 1000) measures fiber rising, fiber roughness, fiber length and amount of fibers in minutes. These characteristics are related to linting, gloss reduction and tissue softness. It was designed mainly for testing the fiber detaching phenomenon. This method is considered to give different results with different paper grades. In Prüfbau pick resistance test the tests are made in laboratory where the printing process is simulated by laboratory press. This test is time consuming and relies on an operator. Also the Apollo press simulates the printing process in laboratory conditions and requires time and operator to conduct the testing and analyzing results. (Gratton & Frigon, 2006, 1-3.)

3.3 LintView method

A new test has been developed by LintView Inc. for testing the linting potential from samples taken off the reels or paper rolls. The test is simple and quick thus the results are gained almost in real time. The tester is portable which makes it easy to carry to sites or rolls that have been anticipated to have problems. The tester can provide data for the producer for assuring that lint sensitive customers do not receive products with high linting propensity and also provide data quickly for making altering in the production line. (Gratton & Frigon, 2006, 1.)

3.4 Metso method

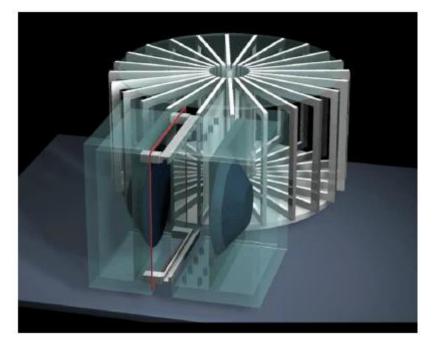
Metso paper has made a study in 2006 concerning the propensity of paper to linting and dusting. The performance in printing is degraded by linting and dusting. The offset presses need to be stopped, washed and started again when linting and dusting reach a critical point. There has been developed a testing device and procedure that are directly in relation to the offset printing process. Before this the paper manufacturers have not had any means to confront these problems on fast schedule and the results from printing lab or print trials have needed to be waited for long time. The long waiting time does not give the paper manufacturers time to react and make needed changes in the machine chemistry and update strategies. The new technique measures linting and dusting and also relative humidity on both sides of the paper, analyses the results and compares them with the production lines related to the production. The objective is to optimize paper quality, determine the linting and dusting propensities and to minimize the problems with linting and dusting in the printing houses. (Metso Paper, 2006.)

3.5 Acoustic Dust Tester

A method quite similar to Polytest Project has been developed in Austria. Mondi research and development team has made a patent of a method for collecting quantitative and qualitative information concerning the particle emissions from paper under mechanical stress. Mondi R&D team has created lab conditions where they have tested the emissions from particulate matter. (Kornherr, Achatz & Drexler, 2011, 79-80.)

The method in use is called Acoustic Dust Tester (ADT). It uses acoustic waves to create mechanical stress on clamped papers. The particles detached from the paper are tested in controlled conditions and they are analyzed in accordance to the total particle distribution, structural and chemical properties of the particles and particle number size distribution. In this method the paper is clamped on a metal frame from the sides and inserted into the measurement chamber (picture 2). The chamber is sealed and first flushed with purified air to clean the air from outside particles. After the cleaning of the air the tested paper is introduced to a five minute mechanical stress created by acoustic waves. The outlet of the air from the chamber is led to different analyzers, for example, to a condensation particle counter and to electro-mobility spectrometer. These analyzers can detect not only micro

but also nano sized particles. The waves created by two loud speakers on both sides of the paper can be changed and the amplitude and shape of the acoustic waves can be modified in accordance of the properties of the paper. This method can be used for papers from low to high basis weight and also for textiles, paper boards and plastic foils. (Kornherr & al. 2011, 79-80.)



PICTURE 2. Scheme of the ADT chamber (Kornherr & al. 2011, 80)

A number of paper grades have been tested with the ADT. The achieved results have shown that the method can provide reliable results even with such a small number of samples as 15 to 20 paper sheets. The method is directed for laboratory use and it gives the advantage for testing all types of recipes containing, for example, different amount and types of filler, sizing agents, binders or fibers. In addition to the changes to the mechanical and optical properties ADT offers a mean to measure the overall dusting propensity. (Kornherr & al. 2011, 79-80.)

Compared to the methods that have been used in dusting measurements, the internal studies show that ADT method gives reliable correlation of the dusting propensity in printing and copying process. Based on the results ADT team is convinced that the method will provide accurate data of the dusting and linting propensity which is important especially in printing processes. The team also sees that the method can be used as a substitute for conventional methods that require up to hundreds or thousands of sample sheets. Thus the costs and time can be reduced with ADT. The aim is to develop a method that interests not only paper industry, but also has an impact on product development and quality control. To accomplish the goal Mondi started a cooperative project with Austrian Kunststoff-cluster -Plastics Cluster (KC) and Austrian Research Institute for Chemistry and Technology (Österreichisches Forschungsinstitut OFI). (Kornherr & al. 2011, 79-80.)

3.6 Methods tested during Polytest Project

3.6.1 IGT Fluff Test

IGT fluff test is based on collecting loosely bound particles from paper surface with a steel disk. The disk is made tacky with medium viscosity pick test oil. The disk was attached to a holder rod which was rolled over 25 paper sheets one revolution per sheet. A layer of fluff test ink was spread onto the disk surface and the disk was placed into the IGT printability tester. The image was transferred onto a standard art paper strip. A pressure of 250 Newton and printing speed of 0.2 meters per second was used. The paper strips can be evaluated with a standard IGT contamination scale. The cleanliness of equipment and air affect the results and also the operator has great impact while performing the test and evaluating the results. (Heinilä, 2007, 26-27.)

3.6.2 Heidelberg GTO offset printing press

In UPM Kaipola mill the Heidelberg GTO offset printing press was used for evaluating linting and dusting tendency. In standard test 2000 sheets are printed with the press and lint is collected from the blanket cylinder with a tape. First 1000 samples are used for measurements from top side and another 1000 from the bottom side. 500 papers are used for optimizing the color distribution in 14 different sectors. The mass of the tape is measured before and after the collection to determine how much particles have been detached. The disadvantage is that the ink is also collected with this method. (Heinilä, 2007, 28.)

3.6.3 Veitsiluoto device

Veitsiluoto device is used by Stora Enso for the measurements of dusting tendency. In this method normally 500 sheets are placed inside a metal chamber. A pressurized air flow is lead through nozzles and it travels around and between the papers. The filter in the bottom of the chamber collects the detached particles. The device is designed for measuring dusting and linting of copy paper. (Heinilä, 2007, 30-31.)

3.6.4 Results

Paper grades tested in 2007 measurements were Kaipola A, B and C. The results from PMMA4 measurement (presented in chapter 5.2), IGT Fluff test, Heidelberg GTO and Veitsiluoto device were presented in the same unit. However, the values were on a different scale because the principle of the measurement methods was different. The comparison was made by giving points to each method by ranking the results from best to worst. The results were taken separately from top and bottom side of the paper samples and the results are presented in figures 1 and 2. (Heinilä ,2007, 46-47.)

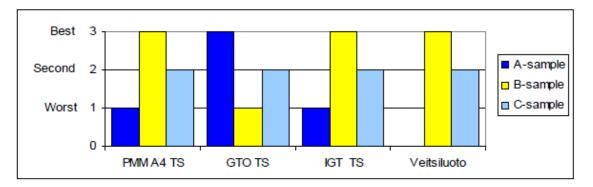


FIGURE 1. Top side measurements from PMMA4, Heidelberg GTO, IGT Fluff test and Veitsiluoto device with Kaipola A, B and C samples (Heinilä, 2007, 46)

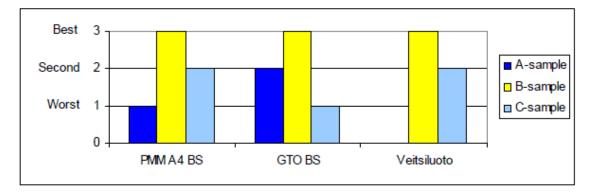


FIGURE 2. Bottom side measurements from PMMA4, Heidelberg GTO, IGT Fluff test and Veitsiluoto device with Kaipola A, B and C samples (Heinilä, 2007, 47)

4 PAPER GRADES AND PRINT MEDIA

4.1 Paper grades and raw materials

The paper grades can be divided into two groups; paper grades where dominating pulp is either mechanical or chemical. Paper grades have different qualities and the end use and cost define the paper grade for which it is used (figure 3). Mechanical pulp containing papers are newsprint grades, super calendared (SC) and coated mechanical paper grades. Usually they contain more than 50% of mechanical pulp and they are also referred as wood containing paper grades. To ensure, for example, printing machine runnability, chemical pulp is added to mechanical pulp as well as minerals are used as fillers and in the coating. Mechanical pulps are economical and have good printability. Mechanical paper grades are prone to turn yellow over time thus the end products have usually a short life cycle. (Haarla, 2000, 24-26.)

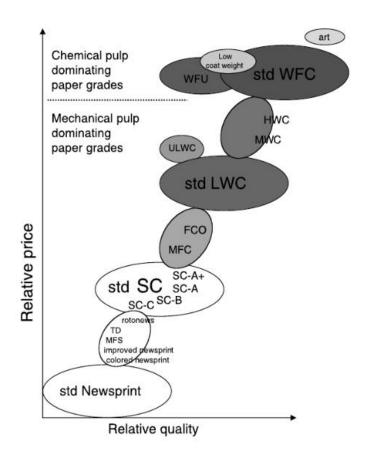


FIGURE 3. Printing and writing papers range (Haarla, 2000, 21)

In addition to the use of virgin material, recycled fiber (RCF) has become an important raw material in paper production. The availability of RFC affects its use and the availability is dependent on the population density and organization of collection. Recycled fibers increase the basis weight of the paper. The lower the basis weight is the lower the costs of transportation, handling and storage are. In Europe, for example, newsprint can be made of 100% recycled material but it can also be made of 100% mechanical pulp. If RFC is used the share of fillers can increase up to 15%. The runnability of the paper depends on the raw materials and treatments made in the production. This means that the printing houses cannot change paper grade without adjusting the printing line. Higher strength and smaller linting tendency can be achieved with soft calendaring in high temperature and linear loads. (Haarla, 2000, 20-25.)

4.2 Trends for print media

The end use of printing and writing papers consists of newspapers, magazines, catalogs, books, commercial printing and copying for laser and digital printing. There are challenges created by financial aspects and material resources and also electronic media has evolved to be a new competitor. The environmental demands have increased which affects the production and use of traditional paper items. The challenges facing printing and writing paper industry are presented in table 1. (Haarla, 2000, 14-18.)

Low building costs of new capacity per	Mixed use of various paper grades within one end use		
roduct unit	Branding		
Low fiber cost per product unit	Newcomers into the industry especially in Asia		
Low energy consumption per product unit	Cyclicality		
High production efficiencies	Challenge from an electronic media		
Supplying global markets from transnational manufacturing	Increasing consumer awareness in terms of environ- mental matters ²		
Timely service and logistics	Competition on intellectual capital		

TABLE 1. Challenges in printing and writing paper industry (Haarla, 2000, 15)

Electronic media has been benefitting from the development of new technologies more than print media. Nevertheless, print media has been developing and has become both cheaper and of higher quality. The choices of the final consumers determine the future development among these two media. At the first stage of development electronic media seems to have reached position in the market and the users' popularity on the field of traditional business forms, manuals and encyclopedias. Especially the magazines of specific field of interest and commercial printing have still been favored in printed form. (Haarla, 2000, 49-50.)

Both media have their strengths and weaknesses. Electronic media provides real time and easily accessible data. The accessibility is strongly related to the access to different machines and networks. This means that developed countries benefit from electronic media more than developing countries. Electronic media can be tailored easily, searched through search engines and the integration of several media is possible as well as interactivity. The weaknesses for electronic media are the price of mass distribution of specific products, lack of safety and the quality of products available in the electronic media. Also the lack of standards is a disadvantage for electronic media. (Haarla, 2000, 49-50.)

Over time there have been changes in end use, paper grades and their characteristics as well as in raw material use in print media (figure 4). The advantages of print media are that it is portable, low cost and it generally has good quality of information. It is also a familiar medium and can be seen as a satisfier of higher needs of human beings. Environmental standards have increased, but despite the use of recycled material the environmental aspect is one strong factor affecting the development in the future of both media. (Haarla, 2000, 41.)

new end uses	
new paper grades	
ightarrow difficulty to classify paper grades	
changing paper characteristics	
higher brightness	
lower basis weights	
polarization of properties	
changes in raw materials	
increasing use of RCF	
expanding use of minerals	

FIGURE 4. Trends for print media (Haarla, 2000, 41)

5 POLYTEST PROJECT

There has been several bachelors' thesis made during the development of Polytest Project. Due to the innovative nature of the project and at that point pending patent applications the earlier thesis are confidential. The progress of the project and the development of the prototypes as well as used methods are explained in the following chapters.

5.1 **Progress of the project**

The first prototype for collecting dust was built in spring 2006. In the autumn 2006 the project started using name Polytest Project and since then there has been several prototypes and different particle analyzers in use. In the beginning the collection of particles was done with Electrical Low Pressure Impactor (ELPI) which later on was changed to ARTI optical particle counter. Before introducing boulder counter the optical particle analyzer used was Dust Trak. PM10 Impactor has also been tested in the project.

Finnish Polytest patent was accepted in 2011 and application for Euro patent was submitted in 2010. IfDust patent application was submitted in Finland in 2010 and also an international patent concerning IfDust has been submitted. (Niskala, 2012.)

In the report of consulting company Practicon from 2009 few possible partners were considered for further development of the project (see chapter 6.1.3). At this moment there are ongoing negotiations with few partners concerning possible collaboration within the project.

5.2 **Prototype development**

PMM V1

In 2007 there were altogether five prototypes built to measure linting in laboratory conditions. All prototypes included an optical particle counter. In the first version in this phase, PMM V1 (particle measurement method version 1), the particles were detached with a mechanical vibrator that was placed inside a 31 liter dust chamber. The chamber walls were covered with aluminum foil to reduce electricity and the particles were sucked into ARTI hand held optical particle counter which detects airborne particles according to the physical dimensions. The device displays six size categories between 0.5 to 10 micrometers. The size categories in the device are low, thus not all fibers were detected. The paper to be examined was taped to the wall from the other end and to the vibrating element on the other end. Replacement air was lead to the chamber through an air filter. After the measurement a PM10 pump was used for sucking out the air polluted by particles. Problems were that ARTI pump rate was too small compared to the chamber size and also the sealing of the box leaked. In this measurement, however, the effect of particles detaching from the edges of the paper was discovered to be considerably high. (Aittamaa, 2007, 13-18.)

PMM A1

In the subsequent versions the mechanism was based on acoustic waves. In the first acoustic version, PMM A1, the loud speaker was covered with a wooden box with a hole. The hole was covered with a transparency on which the paper sample and a wooden plate with a hole were laid. The hole was to restrict the measuring area and prevent the particles from the edges of the paper to interfere the measurement. A plastic box of 10 liters acted as the dust chamber and measurement was conducted with ARTI. Before the measurements the chamber was cleaned with ELPI vacuum pump. Zero levels could not be achieved, as the room air leaked in to the chamber and the results were not reliable. Also ARTI suction point should have been placed in the middle of the chamber to achieve even distribution. (Aittamaa, 2007, 18-22.) In PMM A2 the sealing problem was solved with a sealing mat instead of gasket. The loud speaker was mounted on acrylic glass covered with closed cell plastic mat leaving a hole for the speaker and set into an aluminum core. Insulation was taken care with closed cell plastic matt and sample sheet was placed between two plates made of plexi glass insulated with same material. ELPI cleaned the air before testing. On top the sealing mat a plastic dust chamber of 3 liters was tightened. Two filters were on opposite sides to ensure even air supply. ARTI suction point was on in middle of the chamber and from continuous measurements with no sound wave and afterwards adding the impulse there were clear results of detached particles from the paper samples. Optimal frequencies and amplitudes were examined and from this version on the parameters of 69Hz and 32V were used. (Aittamaa, 2007, 23-30.)

PMM A3

PMM A3 had a bigger woofer and it was a simplified version of PMM A3. Two of the stabilizing plates were combined as one and a new amplifier was introduced instead of function generator to ensure the power supply for the bigger woofer. Also the computer program Audacity was used to generate the sound waves instead of the function generator. During this phase the amplitude of the paper was measured with a laser measurement device and also the effect of room dust gathering on papers was discovered to be significant in the tests made at this phase. (Aittamaa, 2007, 31-41.)

PMM A4

The biggest enhancement in PMM A4 was that the system was turned upside down to prevent the gravitation force to pull down the particles back on the paper surface. This way all the detached particles would be collected. The woofer was glued onto the sealing plate and set on top. The other sealing plate was attached on a funnel shaped chamber and an extra closed cell plastic ring was used in between as a spacer. ARTI was connected on the bottom of the funnel chamber. Clean air was lead directly into the chamber at the rate of 4 liters per minute. As ARTI's suction rate was 2,8l/min the excess air was lead out through the filters. Surprising results showed that fewer particles were recorded with PMM A4. Metal dust chamber was grounded because it was questioned whether the

chamber was collecting dust instead of ARTI. As a defect in this version, the small particles could have gone out of the filters. (Aittamaa, 2007, 41-47.)

Online measuring device

In further development and testing there was an on-line simulator called Helena in use of developing an online measuring device. The running paper web created the need to change the composition of the measuring device. The web conveyer made in the laboratory consisted of a paper laminator as the drive unit and a roll holder in the other end. The dust chamber was tuned in the middle. The tests showed that the establishment was not stable and particles detached already before entering the measurement area. In this phase the entry of room dust was prevented with introducing air flow that keeps the outside particles away from the measurement chamber. (Kurra, 2008, 14.) A more detailed description of the method is presented in chapter 7.2.1.

Automatic paper feeder

Most recent addition in the device development is an automatic feeder. So far the paper feeding has been done manually and by automating the process the manual work can be minimized. The paper feeder is modified from a domestic printer by adjusting the height and the angle of the paper feed suitable for use together with the dust chamber. A controller was used for switching the feed on and off and it uses separate power supply (GW Instek SPS-3610 DC). The feeder is seen in picture 3 in chapter 7.2.

6 ADVANTAGES OF POLYTEST PROJECT

With an online measuring device there are several advantages. The results from the measurements can be accesses and analyzed quickly and it is possible to make adjustments in the production parameters. One major benefit is the decrease in problems created by linting and dusting in the printing houses. There can also be savings in raw material consumption which originate mainly from the optimized ability to use recovered material.

6.1 Consultant report for Polytest project

A consulting company Practicon Partners Oy made a partner matching initiative for the Polytest project in 2009. The objective was to look into the potential markets for the paper dust measuring device and the additional value the device can bring to the paper manufacturing companies. In addition the project recognized potential partners from the industrial field in Finland to support the technical development of the measuring system and commercializing. (Polytest Partner Matching Hanke, 2009.)

6.1.1 Recognized features

In the report it was recognized that the problem with dusting and linting is mainly with the paper that consists of recycled material. These paper grades are mainly used for news-papers. The papers that are produced from primary fibers are not that prone to dusting and linting. Dusting and linting can be conditioned by technical measures, thus the online measurement for dusting and linting in the paper production line has significant benefits. Because the monitoring and controlling of dusting and linting is done mainly in the production lines, the printing houses are not included the primary partner group. The Practicon's report states that the potential partners are in the field of paper industry. (Polytest Partner Matching Hanke, 2009.)

With Polytest measuring device the measuring of dusting and linting can be done either in the laboratory during the quality control or online in the production line. When the quick response to the variation is taken into account, the online testing allows quicker real time response and enables more efficient production process. The development of the processes can also be enhanced efficiently with a measuring device that reacts immediately to the changes in the production. When the measuring method is in correlation to the specifications of the paper quality needed in the printing houses, the ingredients and their relations can be controlled more precisely. (Polytest Partner Matching Hanke, 2009.)

Paper grades prone to dusting are printed mainly with offset or rotogravure techniques. Most of the problems with dusting occur with heatset offset printing due to the high viscosity of the used ink. With rotogravure the low viscosity ink and the absence of rubber cylinder decreases the dust particle detaching from the paper surface. The main printing process for newspapers is either coldset offset or heatset offset. For SC grades heatset offset and rotogravure are in use. In offset printing the rubber cylinders are equipped with washing devices and, for example, in newspaper printing houses the print run is done during the night and the cylinders are washed during the daytime. (Polytest Partner Matching Hanke, 2009.)

6.1.2 Financial advantages in production and printing

From the financial point of view the benefits of the enhanced process with the online measuring device can be found in the production process and also in the printing houses. The amount of rejected papers can increase 3.3% and at the same time the productivity decreases in same relation if there is a 2 minute washing once per hour in a printing house. In a midsized printing house with 20000 tons of paper annually the estimated cost of rejected paper is between 300000 and 400000 depending on the used paper grade (figure 5 in original language). (Polytest Partner Matching Hanke, 2009.)

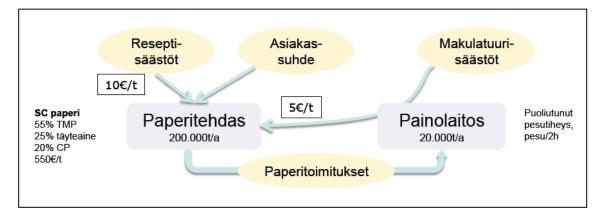


FIGURE 5. Estimation of additional value to paper mills (Polytest Partner Matching Hanke, 2009)

In paper industry there are potential benefits from the use of online paper dusting measuring device. The amount of fibers and fillers can be optimized and this can make savings in the ingredients. Also the amount of the rejected paper during the cylinder washing can be decreased. A significant advantage can be found in the customer relations due to the enhanced quality of the paper. For SC paper the financial savings can be estimated to be approximately 10€/t by optimizing the ingredients and approximately 5€ per ton from the decrease of rejected papers. For a paper manufacturer with 200000 tons production per year the savings could reach up to 3 000 000€ annually, not to mention the satisfied customers and possible new customers due to the high quality paper. As mentioned, the printing houses can make savings by increasing the cylinder washing interval up to 2 hours from the earlier 1 hour interval. The costs from the washing could be halved, from 3000000€ - 400000€ to 150000 - 200000€. (Polytest Partner Matching Hanke, 2009.)

6.1.3 Partners

The potential markets for the use of commercial paper dusting measuring device are limited both in national and European level. In Finland newsprint and SC paper are manufactured in 5 places with 10 machines. In Europe the estimated amount of the machines is approximately 75. In the future the amount of machines will probably not increase and the trend for the demand for newsprint has also been decreasing. (Polytest Partner Matching Hanke, 2009.) The quarters benefitting most from the online dust measuring device are the paper manufacturers. Despite this fact the manufacturers do not usually participate in the development process of measuring systems, but mostly do only test runs and experiments. The possible partners are the manufacturers of measuring devices for industry and producers of machine automation and control systems. In Finland there are few companies in the field of product development which are the most potential for partners in Polytest project development. (Polytest Partner Matching Hanke, 2009.)

The domestic partners recognized in Practicon's report were ACA systems (measuring devices for paper industry), Dekati Oy (online fine particle measurements), Metso Automation (automation and control systems for processing industry) and Tapio Technologies (measuring solutions for paper industry). One partner could also be Stora Enso mainly in the field of test runs. The main objective for introducing an industrial partner is to ensure further development and commercializing the product. The discussions concerning partnership in the project were started with ACA Systems and Metso Automation. The estimated market price for an online measurement device was from 50000€ to 100000€. (Polytest Partner Matching Hanke, 2009.)

6.2 Use of recovered paper

Recovered paper (RP) is rather good and reasonably priced raw material and it has a very important part in paper and board industry. The share of both utilization and use of RP is expected to grow but virgin fiber is still needed to keep the production running. In 2008 the share of the world's recovered paper supply used for container board and carton board production was 65%. The share of RP as raw material has increased from 41% in 1997 up to 52% in 2007. The global paper recovery rate was 52%, however there are great regional differences. Generally the countries producing paper and board that have good resources of virgin material have used less RP as raw material. Despite the availability of virgin material the collection rates can be good and the over carried RP is be exported to countries where the material is needed. (Ervasti, 2010, 17-18.)

In Finland there is an obligation for producers to organize the reuse recovery or other suitable treatment of disposal concerning their products and the waste derived from them. The related costs are covered by the producers. The obligation is based on the Waste Act

(646/2011). The obligation covers many product types, printing paper, packaging and paper for manufacturing other paper product among the other types. (Finnish Environment Institute, 2013.)

There are two accepted producer communities in Finland, Paperinkeräys Oy and Suomen keräystuote Oy. The producers can pass on the baton by joining in the producer communities. On behalf of the producers the producer communities have arranged a nationwide collection network for paper and they are liable for achieving the target of reclaiming 75% of the recovered paper obligated by law. Individual real estates are liable for arranging a place and a collection bin for paper in population centers. The producer communities are responsible for collection and the costs resulting from organizing the collection. In sparsely populated areas the real estates are not liable to establish collection points but the producer communities have to arrange local reception centers. (Finnish Environment Institute, 2013.)

Over 90% of the used paper is recovered in Finland (Paperinkeräys Oy, 2012). The majority of recovered paper is used in UPM Kymmene's paper mill in Kaipola. Over two thirds of the recovered paper from households is processed in Kaipola mill. In addition, a small amount of RP is delivered from nearby countries. Nowadays Kaipola mill is the only mill in Finland producing newsprint. According to the Kaipola mill's environment manager Pia Siirola-Kourunen, the most important thing in using recovered material is that the amount of virgin material can be decreased. Also the energy consumption in the manufacturing process is significantly lower compared to processes using virgin material. The water used in paper production is collected, purified and reused in the production of recovered pulp. (Vainio, 2013.)

7 MEASUREMENTS WITH STATISTICAL METHOD

7.1 Paper grades

Paper grades used in the measurements were mainly the same grades that were used in the previous stages of Polytest project. Kaipola A, B and C were stored in the original delivery packages which were covered in order to prevent external particles contaminating the samples. Shotton, Sappi and Stora Enso Varkaus were delivered to the laboratory during the measurements. A sample of commercial copy paper (CCP) was obtained from an unopened copy paper package. Extra care was taken that the samples would not be affected by room dust. The effect can be significant over time as was discovered in Janne Heinilä's thesis A comparison of the paper linting and dusting measurement methods used in the POLYTEST –project. In his thesis the room dust effect was tested for one to 10 days and after one day the results gave clear indication that the papers collected dust from the room air. (Heinilä, 2007, 37.)

The properties of CCP were not measured as detailed as the other samples were measured in previous stages of the Polytest project. During the project Jani Kurra who studied International Pulp and Paper Technology made analysis of other paper grades in the paper laboratory and the properties are presented in table 2.

Properties	Kaipola A	Kaipola B	Kaipola C	Shotton
Basis weight (g/m ²)	35,3	37,7	33,4	46,5
Thickness (um)	51,3	54	50,3	68,5
Density (kg/m ³)	688	698	664	678
Roughness us (ml/min)	82	55	67	105
Roughness bs (ml/min)	71	67	59	125
Air permeability (ml/min)	187	119	215	213

TABLE 2. Measured paper properties (Kurra, 2008, 24)

Kaipola A, B and C paper grades are made in UPM Kaipola paper mill and they are telephone directory papers (TD). These paper samples were also measured with their own Heidelberg GTO method and in addition, with IGT Fluff test and Veitsiluoto device. These tests were evaluated in Janne Hienilä's thesis and explained in more details in chapter 3.6. The sample named Shotton is made in the UPM Kymmene paper factory in Shotton, Great Britain. It is a newsprint and known to have high dusting density. It also contains high proportion of recycled fibers. Based on the evaluation of Janne Heinilä the samples of Kaipola paper grade are very similar according their properties. However, Kaipola B has higher basis weight and thickness which improves strength and toughness. (Heinilä, 2007, 44.)

Two other paper grades were used in some of the measurements with boulder counter but they were left out from the further analysis. The other grade is base paper Sappi made in Sappi's Kirkniemi mill and the other is a TD paper from Stora Enso factory in Varkaus. The decision of abandoning the grades was done based on the results that these grades were less prone to dusting than, for example, Shotton. Also the objective was to gain results that could be compared to the measurements done in the previous stages of the Polytest project. CCP was maintained in the tests mainly to ensure in every stage that the recordings were successful. The effects of changing parameters were easiest to monitor with CCP copy paper due to the high amount of detached particles.

7.2 Laboratory equipment

A sound wave was created with Audacity computer program, amplified with Peavey PV-8.5C amplifier and lead to the loud speaker which acted as the acoustic vibrator. The sound wave detached particles from the paper that was placed between the acoustic vibrator and the dust collecting unit. The particles were sucked into the optical boulder counter (see chapter 7.2.2) through the dust collecting unit. Purified air was lead between the layers of the dust collecting chamber to prevent the external particles entering the collecting unit. The schematic picture of the laboratory equipment is presented in figure 6.

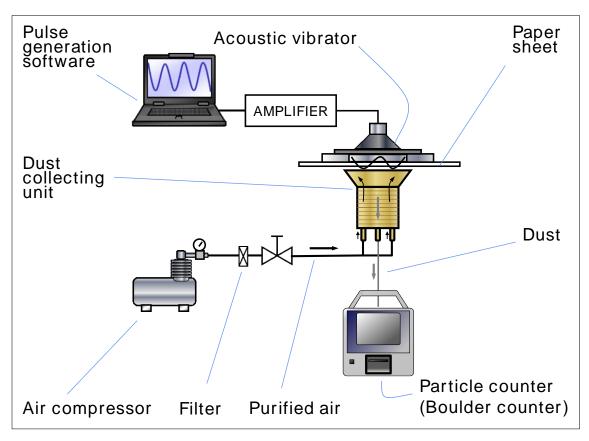


FIGURE 6. Schematic overview of the laboratory equipment with boulder counter

The laboratory device setup is shown in picture 3. The devices are numbered as follows:

- (1) Pulse generating software (computer)
- (2) Amplifier
- (3) Acoustic vibrator
- (4) Dust collecting unit
- (5) Boulder counter
- (6) Air flow system
- (7) Automatic feeder
- (8) Power supply for automatic feeder
- (9) Oscilloscope for amplitude checking



PICTURE 3. Laboratory equipment

7.2.1 Dust collecting unit

The dust collecting chamber is made of three layers. Pressurized air is conveyed in the round metal can which acts as the outer layer and to the funnel shaped middle layer. The middle layer is perforated from the sides and the small pinholes make a laminar air flow against the paper. The inner layer is also a funnel and there is a gap between the inner and middle layer, which is also approximately 2mm higher on the edge next to the loud speaker. This way the air flow can both create an air curtain to prevent the room air entering the chamber and also partly help to transfer the detached particles into the measuring device (see figure 7). The dust output pipe is joined with the inner layer with aluminum bushing.

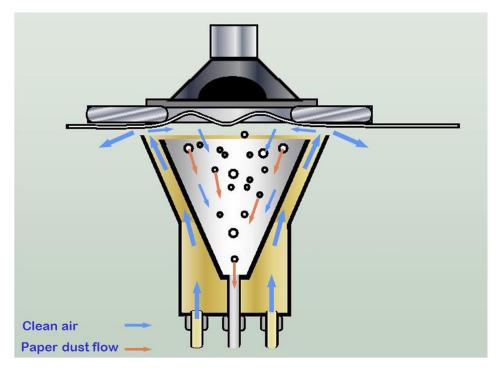


FIGURE 7. Scheme of the air flows inside the dust chamber

7.2.2 Boulder counter

Lighthouse Worldwide Solutions' LMS Express 7 is an optical particle counter that collects and displays data and it has been used in the project since summer 2012. The boulder counter has the inlet speed on 281/min and it records the particles by sizes in 6 categories: 5, 10, 25, 40, 50 and 100 micrometers. The sizes and number of particles are recorded by an optical reader.

The recordings can be saved directly in worksheet form or in graphs in linear or logarithmic scale. The data can be downloaded into computer either automatically or manually. In this experiment the manual downloading was used. The data sets are automatically generated when there is data downloaded from the particle counter. With the program it is possible to generate data tables and graphs from the recordings according to the chosen time period. A specific feature which is not commonly in use is that the particle counts are added from the smallest size to the subsequent size. The data is rough data after downloading and needs to be processed further in order to acquire data that can be compared and divided, for example, according to size of the particles. In the table the data is presented according to the size and also standard deviation, average, maximum and minimum data points are calculated and displayed automatically. In the graphs the sizes are presented in different colors and the y-axis presents the counts of each size and the x-axis presents the time. Also in the automatic graphs the smallest particle counts consists of all the larger particle counts.

7.3 Device parameters and preliminary tests

The parameters had to be chosen before starting the actual tests with boulder counter. The parameters were studied already in previous tests over the years but the changes in the laboratory devices in use and the current laboratory settings required preliminary testing. The method and parameters needed to be powerful enough to detach particles from the surface but not too powerful in order to prevent the paper structure collapse. In the pre-liminary tests the parameters were monitored by changing the values and recording the results in order to see which will have an effect on the results. The paper used in preliminary tests was CCP.

There are few factors affecting the parameter choices. One is the inlet speed of boulder counter. The rate of the air flow needed to be more than the suction rate of the counter in order to keep the outside air particles from being sucked into the counter as well. The other factor is the nature of the particles detaching from the paper. Not all the particles are spherical and the assumption was that also bigger fibers will detach. The composition of the particles needed to be analyzed with a different method and it was done with PM10 impactor and optical analysis (see chapter 8).

7.3.1 Air flow

The effect of the air flow coming from the inlet was tested with several papers and with the flow from 251/min to 401/min. The generic result of the values close to the boulder counter inlet suction rate (281/min) showed that they were too low. The amount of particle counts increased significantly when the air flow was decreased closer to the boulder counter suction. The air flow was not sufficient to keep the outside air away from the chamber and there was a great probability that external particles were sucked into the dust chamber. The tests were made with 10 paper samples and in addition, the higher air flow values were tested in combination with higher amplitudes. Based on the results air flow was set to 401/min.

7.3.2 Burst duration

The first tests were conducted with a two second sound wave (burst). The impact of longer burst was tested for assuring the most effective way to detach the particles from the paper surface. The burst duration was tested with four seconds and the results were compared with the two second results. No significant effect was recognized thus the burst duration was set to two seconds in the final testing. In table 3 there are the average counts of CCP, Kaipola B and Shotton from 3 burst testing with 20 papers. The averages of 10-18 volt are calculated. In Kaipola B and Shotton there is a slight increase noticed.

TABLE 3. Effect of burst duration

Order of	CCP 2s	CCP 4s	Kaipola B	Kaipola B	Shotton	Shotton
burst			2s	4s	2s	4s
1	840	780	100	130	83	95
2	360	420	23	27	32	40
3	230	220	16	18	23	35

7.3.3 Multiple burst testing

The objective of testing three bursts with the same paper was to determine how much of all the particles could be detached from the paper surface with one burst and how much is detached relatively after 2 and 3 consecutive bursts. The tests were executed with different amplitudes for monitoring the variation in the results between p-to-p value of 10V and 18V. The paper grades used were Kaipola B, CCP and Shotton and two tests with 20 papers were conducted with each paper grade. In figure 8 there is an example of the three burst test with CCP. The parameters used in this experiment were amplitude of 14V, frequency of 100Hz, burst duration of 2 seconds and air flow of 40l/min.

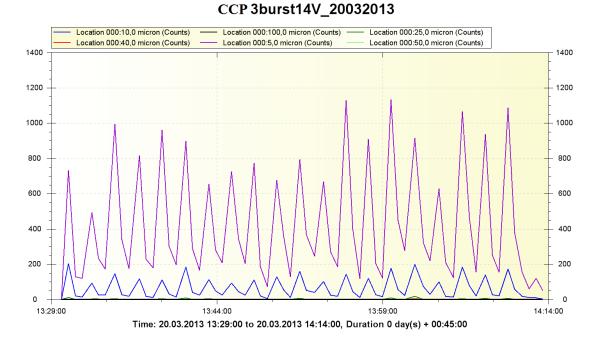


FIGURE 8. Example of three burst experiment with CCP

As seen in figure 8, there is a clear pattern and the amount of particles decreased significantly from first burst to second burst. The decrease was not that high between second and third burst. The overall results are presented in table 4 where the decrease between bursts is presented as an average of all particle sizes and all the amplitudes from all the grades. The results indicated that there were great variations in the particle counts between paper grades. Yet, in all amplitudes and all paper grades there was a notable and regular pattern among first, second and third burst.

Decrease (%)	ССР	Kaipola B	Shotton
between 1st and 2nd	50,0	79,0	60,0
between 1st and 3rd	68,3	86,2	72,5

TABLE 4. Decrease in the amount of particles between 1st, 2nd and 3rd burst

7.3.4 Amplitude

The preliminary testing was started with low amplitudes with p-to-p values between 4V and 8V. These levels were not high enough to detach the particles from the paper surface. The levels were tested up to 18V to determine whether there was a point where the paper

would start to break and release more particles from the tattered edges of the tears. Amplitude testing was done simultaneously with three burst testing and in the results the total amount of particles from the first burst was taken into account. The amplitude was tested with 2V intervals and the results of three paper grades (Kaipola B, CCP and Shotton) were compared with each other (figure 9). CCP had a much higher amount of particles and in figure 10 the comparison is made only with Kaipola B and Shotton which had a result more of the same size. The general observation was that all the grades showed quite close values within the grade with 14V when compared to 12V and 16V. In other intervals the variations were higher. Thus the amplitude was set to 14V in the final testing.

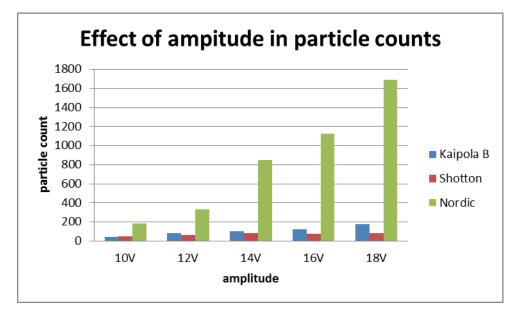


FIGURE 9. Effect of amplitude with CCP, Kaipola B and Shotton

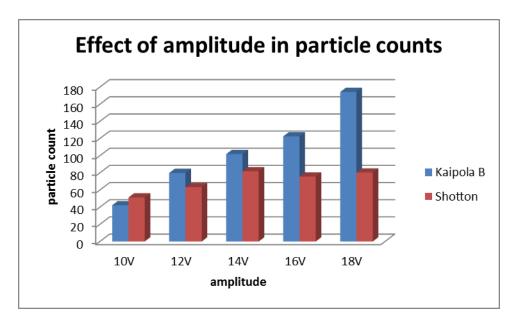


FIGURE 10. Effect of amplitude with Kaipola B and Shotton

7.3.5 Other parameters

Frequency was set to 100Hz according to the instructions from the supervisors and based on the previous tests made in earlier phases of the Polytest project. The latest recorded tests made with the dust chamber were from summer 2012. In comparison to the external factors during the summer time, relative humidity (RH) was lower during this experiment. Relative humidity in the laboratory in the summer time was estimated to be around 50%. During this experiment the measured RH values varied between 10% and 14%.

The laboratory circumstances could not be modified due to lack of control in the room conditions, the various devices attached to each other and the need of space for them. However, the effect of humidity was superficially tested by keeping the papers in the control room overnight and in another test for three days. In the control room the temperature and relative humidity were kept constant, in 20 degrees Celsius and RH 50%. The paper used in the test was CCP and four piles of ten papers were tested in each test. The results did not show any significant change compared to the tests made with papers kept in lower humidity.

7.4 Final tests

The final tests were made with CCP, Shotton, Kaipola A, B and C. The sample size was 100 papers and frequency used was 100Hz, amplitude 14V, air flow 40l/min and burst duration 2s. For each paper grade the average particle counts and standard deviation per size are presented in tables 5 to 9. The results are used as an overview in comparing the results with data obtained from PM10 measurements and optical analysis. A more detailed analysis concerning data obtained with boulder counter is made in the thesis of Taye Hailemariam.

In the result tables there are few changes that need to be taken into account. One test result is removed from Kaipola A measurement because it clearly stood out from the rest of the results. In Shotton and Kaipola B results three outliers were removed from each. In Kaipola C there were some peaks but they were not that clear thus all the results were recorded in the final results. With CCP there was a regular pattern with high variation between results. In general it can be seen that CCP has a high amount of small particles ($5\mu m$) and compared to the other grades, less particles that are $25\mu m$ or larger. Also Kaipola A stands out from the paper grade group with higher average with particle sizes $5\mu m$ and $10\mu m$.

ССР	5µm	10µm	25µm	40µm	50µm	100µm
sum	51882	7557	152	3	5	0
average	520,4	75,5	1,5	0,0	0,1	0,0
st.dev	157,8	26,4	1,8	0,2	0,2	0,0

TABLE 5. The average count and standard deviation per particle size with CCP

TABLE 6. The average count and standard deviation per particle size with Kaipola A

Kaipola A	5µm	10µm	25µm	40µm	50µm	100µm
sum	10638	7038	393	3	17	0
average	107,5	71,1	4,0	0,0	0,2	0,0
st.dev	50,1	52,6	4,8	0,2	0,5	0,0

TABLE 7. The average count and standard deviation per particle size with Kaipola B

Kaipola B	5µm	10µm	25µm	40µm	50µm	100µm
sum	5745	4478	545	10	20	0
average	59,2	46,2	5,6	0,1	0,2	0,0
st.dev	21,7	20,4	3,9	0,3	0,5	0,0

TABLE 8. The average count and standard deviation per particle size with Kaipola C

Kaipola C	5µm	10µm	25µm	40µm	50µm	100µm
sum	6267	5043	535	9	21	0
average	62,7	50,4	5,4	0,1	0,2	0,0
st.dev	24,9	37,0	5,6	0,3	0,5	0,0

Shotton	5µm	10µm	25µm	40µm	50µm	100µm
sum	4660	3481	295	2	14	0
average	48,2	36,0	3,0	0,0	0,1	0,1
st.dev	30,8	32,2	3,2	0,1	0,4	0,3

TABLE 9. The average count and standard deviation per particle size with Shotton

8 PM10 MEASUREMENTS

8.1 Laboratory equipment

In PM10 measurements the laboratory settings were the same as in statistical method (see chapter 7.2) with few exceptions. Boulder counter was replaced with PM10 impactor and an external Dekati PM10P30 pump. The scheme of the equipment is presented in figure 11.

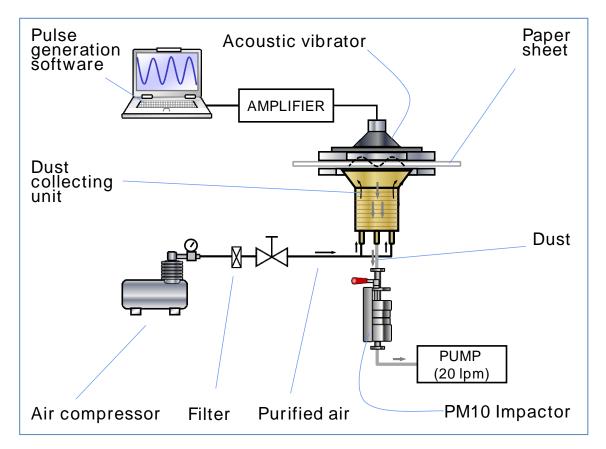


FIGURE 11. Schematic overview of the laboratory equipment with PM10 impactor

Dekati PM10 impactor is a cascade impactor which classifies the airborne particles in four stages. The particles are classified according to the aerodynamic diameter, thus the impactor operates based on inertial classification and gravimetric analysis. The air is pumped into the impactor with Dekati PM10P30 pump and it is used in conditions close to atmospheric pressure. Each stage of the impactor is compiled with a jet plate (filter), a collection plate where the aluminum foil (collection substrate) sprayed with silicone is

placed, and a support ring to secure the aluminum foil on the collection plate (picture 4). The last stage has a filter holder where the filter is placed.

The first stage (PM10) removes particles that are larger than 10 microns. Subsequent stage (PM2.5) collects particles of size between 2.5 and 10 microns and last stage (PM1) collects particles that are smaller than 2.5 microns and larger than 1 micron. The filter stage collects particles smaller than 1 micron.

8.2 Procedure

The measurement setup started with disassembling the impactor according to the stages. The collection plates were made of thin aluminum foil of 25mm diameter and the filter was the size of 47mm diameter. The PM10 impactor was disassembled stage by stage to retrieve the collection substrates (aluminum foils) where the different size particles would then be collected through the filter on the collection plate. The impactor was positioned vertically on the table and the two clamps compressing the impactor vertically were released (picture 4). The first jet plate was joined with the inlet tube of the impactor and the subsequent stages could be accessed after removing the previous stages.



PICTURE 4. The disassembled stages in lines from PM10 stage parts on the left to the filter stage on the right

Four aluminum foil collection substrates were placed in a mask plate. When the mask plate lid was attached it was taken to the ventilation chamber. There it was delicately sprayed with Dekati DS-SIS collection substrate spray to prevent particle bouncing and left to evaporate for few minutes. The collection substrates were then placed into a plastic container and transferred to the control room for measurements. The room temperature is normally set to 23°C and room humidity of 55,3% and they should stay constant, thus normalized. The room suffered some malfunctions at the time of the experiment and the conditions varied. Despite of this, all the measurements were conducted in relatively similar conditions and the collected samples were stored in the room in same conditions while waiting for picture analysis to be started.

During the handling of the collection substrates it was important not to touch the greased area and tongs were used for careful handling. In the control room all the foils were weighted with a balance (Sartorius CP225D). Before every weighing the balance had to be zeroed. The initial foil mass was noted down according to the size or stage where the foil would be placed in the impactor. The measurements were done five times for each collection substrate and the average was used for calculations of the starting and ending weights.

The assembling of the impactor was done in the opposite order than disassembling, thus starting from the bottom with the filter stage. The subsequent stages were placed on to the previous and finally the impactor was locked with the clamps. The impactor was attached to the dust chamber system with the inlet tube. From the bottom of the impactor a hose was lead to the pump. In this analysis the air flow used was 20 liter per minute. Also the air flow of purified air lead to the dust chamber was set lower than in boulder counter experiment to maintain similar air flow difference. The clean air flow was set to 321/min instead of previous 401/min. The pump automatically recorded the air flow and the actual air flow could be calculated by subtracting the initial recording from the final recording in the end of the measurement. The tangible air flow rate varied between 18 and 19 liters per minute.

8.3 Mass measurement

The first samples were collected from CCP. This paper grade had been the most prone for dusting in the boulder counter experiments and it was important to start with a grade that would demonstrate the functionality of the setup. Based on the measurements with boulder coulter there was estimation done of the needed amount of papers to achieve needed amount of mass to be measured with the most accurate scale available (Sartorius CP225D). The mass estimation was based on formula 1 and presented in figure 12 for CCP. The balance could measure masses of 10 micrograms, thus the deviation was +/-10 micrograms. The required amount of papers needed to exceed dozens of micrograms to achieve a mass that was measurable with the balance and based on figure 12 it was decided to start with 100 papers.

$$M_{tot} = \sum_{i=1}^{6} N \rho \pi D_{p,i}^{3} / 6$$
 (1)

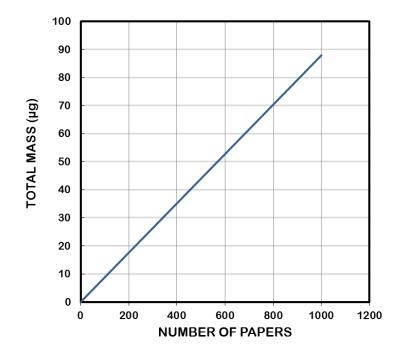


FIGURE 12. Total mass estimation (linear regression) for CCP

The first collection was done with 100 papers and the parameters were the same than used in the final tests with boulder counter, that is, frequency 100Hz, amplitude 14Vand duration of the burst 2s. The results indicated that a bigger sample was needed to make conclusions and the next experiments were done with a sample of 300 papers and with a sample of 100 papers combined with amplitude of 18V. The altering with the parameters did not make a remarkable change and the availability of time and especially the amount of available paper samples for different paper grades set the limit to experiments with 100 papers per paper grade.

8.3.1 Results

The results from PM10 impactor weighing measurements are presented in appendix 1. CCP results are taken from sample of 300 papers and all the other grades are from samples of 100 papers.

8.3.2 Errors

The variation was calculated per each sample and stage before and after the measurement. The variation per stage is also presented in the result tables in appendix 1. As an example of the calculation of initial variation with CCP results for stage PM10

$$\Delta m = \frac{mhighest - mlowest}{2} = \frac{mhighest - mlowest}{2} = 0.015 mg$$

Total error includes the variation and also the scale readability which was ± 10 micrograms. Total error for the previous example with initial and final error and readability was calculated as follows

$$\Delta m = \sqrt{\delta_1^2 + \delta_2^2} = \sqrt{(0.015mg)^2 + (0.02mg)^2 + (0.010mg)^2} = 0.027mg$$

8.3.3 Discussion

The size of the sample was too small to make any definite conclusions. In some of the samples the error was bigger than the subtraction from initial and final weight. With some grades the initial weight was bigger than the final even though it should have been the opposite. Also the weight of blank sample and filter varied.

The errors may have been caused by that the collection substrate spray that did not evaporate thoroughly before the initial weighing. It might have resulted some of the grease still evaporating before putting the foil into the impactor. The impactor accuracy was also close to the expected deviations in the weight and no definite results could even have been waited with the sample size used. The differences in the mass were also close to the scale limits and only a small contamination from outside particles could have affected the results. Human error is also possible with such a small readings. The scale did not always stop into the readings it already had showed as final readings. It was noticed that the scale reading changed still after many seconds even though it had been stable for longer time.

9 Optical analysis

9.1 Analysis from Olympus SZX9 pictures

The optical analysis was done with two microscopes and two cameras. The overall pictures were magnified with a microscope Olympus SZX9 and the camera in use was Olympus Camedia Digital Camera C-3030 Zoom. Each sample was photographed as one image covering the whole sample area (appendices 2 to 6). After taking the pictures with adjusted settings the scale was also photographed and it was later attached to each picture of the sample with image editing software ImageJ. The numerical data obtained from ImageJ was transferred to an excel template made by PhD Jarmo Lilja to model the distribution of different size particles and to determine the amount of fiber like particles.

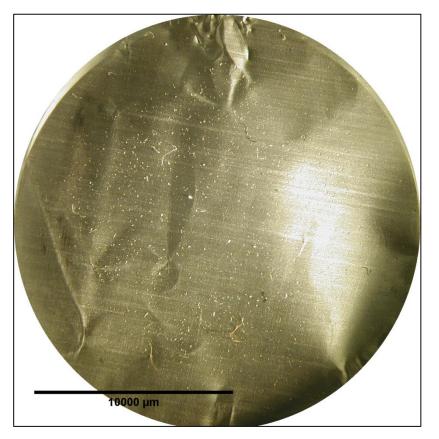
9.1.1 ImageJ software

ImageJ is an open source software for image processing and analysis. It is written in Java and the software and the code it is freely available either as an online applet or as downloaded application. With ImageJ it is possible to display, edit, analyze, process, save and print 8-, 16- and 32-bit images and it reads many different image formats. Time consuming operations can be performed simultaneously with other operations. The application measures distances and angles and calculates area and pixel value statistics. Scaling, rotation and flips, contrast manipulation, sharpening, smoothing, median filtering and edge detection belong to the standard image processing functions. The open source makes it possible for the users to develop Java plugins for solving almost every image processing or analysis problem.

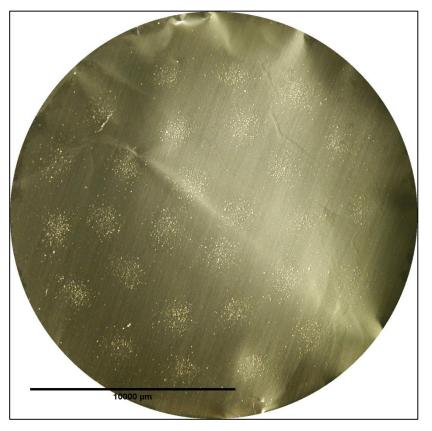
The features used in the work were mainly constrained to few features, such as area selection, scaling and subtracting background. The latter used a sliding paraboloid or a legacy rolling ball algorithm for correcting uneven illuminated background. Image processing was developed further by adjusting window (range of minimum and maximum) and level (position of that range in the grayscale intensity space), brightness and contrast. For the analysis the image needed to be converted to binary form, that is, in black and white. With the analysis tool it was possible to obtain wanted data from the images such as particle area, center of mass, shape descriptors, area fraction, median, feret's diameter, perimeter and centroid.

9.1.2 Results

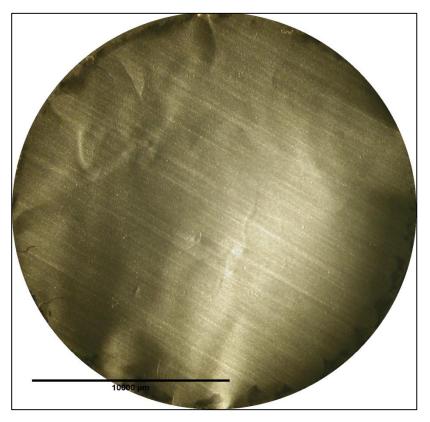
As an example there are pictures of Kaipola A sample from stages PM10, 2.5 and 1 in pictures 5, 6 and 7. These example pictures were not handled in any way thus it is clearly seen that the illumination is very uneven. From the images it was clearly seen that the magnification and optical properties of the camera were not sufficient for making detailed analysis with ImageJ software for stages smaller than PM10. In stage PM2.5 it was noticed that the particle sizes are so small that the program cannot distinguish the individual particles. Stage PM1 seemed virtually untouched by any particles in these pictures. The further analysis concentrated on the results from stage PM10.



PICTURE 5. Kaipola A aluminium substrate from stage PM10



PICTURE 6. Kaipola A aluminium substrate from stage PM2.5



PICTURE 7. Kaipola A aluminium substrate from stage PM1

All the pictures were handled with similar procedure to the point where the illumination conditions came across. The steps of the image processing from each sample are presented in appendices 7 to 11. The images start from the original image that has been selected to the center of the image, then the background was subtracted and the window, level, brightness and contrast were adjusted. In the fourth image the color threshold was adjusted. In the fifth image the sample was turned into binary form and the last image presented the results from the particle analysis with ImageJ.

The results obtained from ImageJ were used in an excel application created by PhD Jarmo Lilja especially for analyzing the particle sizes, distribution and also made it possible to classify the particles in different categories. The results obtained from the different samples from stage PM10 showed differences according to the sample both in the total amount of detected particles and also in further analysis of fiber like particles with excel application (table 10). The results presented in the table are given as an average of five different processing sessions with all five samples. In the results both the total amount of particles and the amount of fiber like particles is presented with the variation.

Sample	ССР	Kaipola A	Kaipola B	Kaipola C	Shotton
total count,	4300+/-90	3000+/-970	1200+/-140	1500+/-160	1900+/-150
ave					
variation in	2	32	12	11	8
per cent					
fiber like	175+/-10	132+/-80	140+/-30	130+/-20	190+/-10
count, ave					
variation in	5	57	21	14	6
per cent					

TABLE 10. Average of total and fiber like particles

As seen in table 10 the results with CCP and Shotton have very low variation and with Kaipola B and C it is a little higher. The illumination conditions with the picture of Kaipola A were very demanding and also the results were versatile. The results with the total amount varied between approximately 2300 and 4200 particles but only in one take the result with fiber like analysis was clearly different from the others. This take was close

to the average total particle count but compared to the other fiber analysis it differed immensely. As for the other four takes the average was 104 fiber like particles but the differing take resulted for 243 counts.

The distribution of particles are presented in figures 13 to 17 both in total number of particles and as fiber as non-fibers (linear scale). The examples are taken from fourth experiment where Kaipola A results were very divert from the other experiments. The amount of smaller particles is much higher and it implies that there might be background noise which is interpreted by the ImageJ software as particles even though they would actually be reflections from the foil. All the samples have the similar pattern of having particles approximately size 25µm and continuing from 40 but lacking sizes in between.

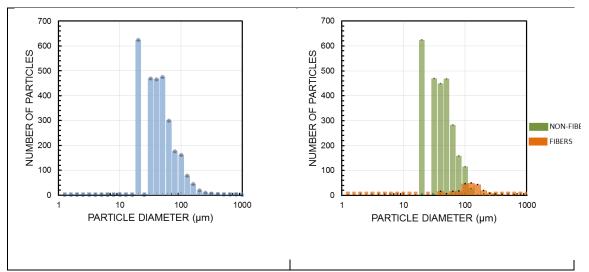


FIGURE 13. Distribution of total (left) and non-fiber and fiber like particles of PM10 stage of Kaipola A

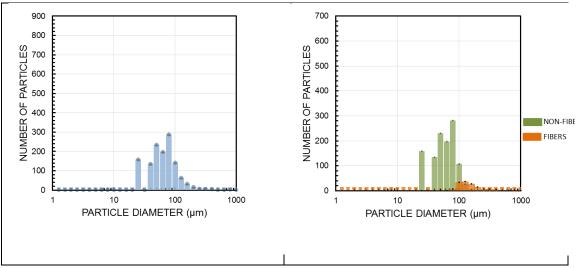


FIGURE 14. Distribution of total (left) and non-fiber and fiber like particles of PM10 stage of Kaipola B

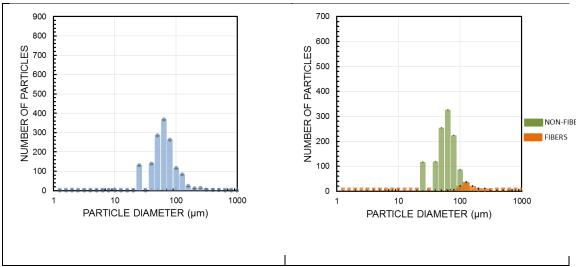


FIGURE 15. Distribution of total (left) and non-fiber and fiber like particles of PM10 stage of Kaipola C

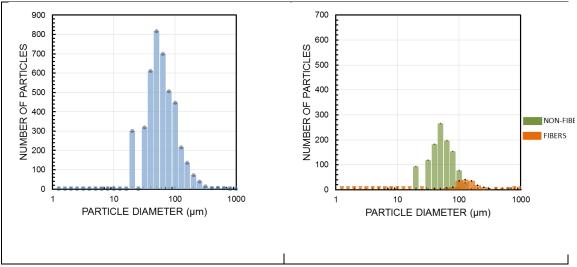


FIGURE 16. Distribution of total (left) and non-fiber and fiber like particles of PM10 stage of CCP

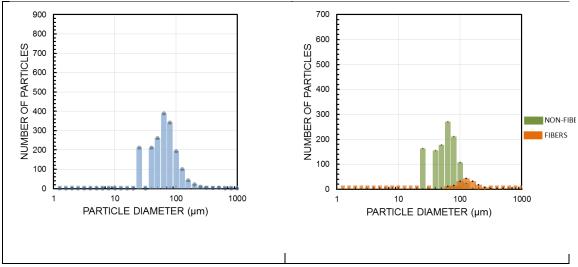


FIGURE 17. Distribution of total (left) and non-fiber and fiber like particles of PM10 stage of Shotton

9.1.3 Discussion

In all the pictures taken from PM10 level there are clearly visible fiber-like particles. There are also smaller particles which are harder to identify and a more precise microscope and camera would be needed. From the overall pictures it is clear that the sample size makes a clear difference. The amount of particles in CCP sample of 300 papers is clearly higher than in the other samples. In comparison to the results from earlier measurements with Kaipola paper grades (see chapter 3.6.4) there are clear similarities. The measurements were made with these grades in 2007 with the Polytest project prototype PMMA4, Heidelberg GTO method, IGT Fluff test and Veitsiluoto device from each side of the paper. Based on the ranking, generally the best grade, that is the grade that has the lowest dusting index, was Kaipola B from both sides. In average the second best grade was Kaipola C. In the measurements made during this thesis Kaipola B and C were also the least dusting paper grades from these three grades with only a small difference in the results. The most recent test gave results with high variation with Kaipola A most likely due to the relatively poor quality of the pictures.

The boulder counter measurements showed clear variations between individual papers which may have affected the results even with a bigger sample. The paper properties measured in 2007 did not show any remarkable difference in the properties of Kaipola grades. Even though, Kaipola B had higher basis weight and thickness which improves toughness and strength properties. These aspects explain the differences in the results to some extent. The 2007 measurements also included Shotton sample. It has considerably higher basis weight and thickness than the Kaipola grades but density on the other hand is not as high.

For stages PM2,5 and PM1 it was harder to recognize any fiber-like particles although in some of the samples there were few particles resembling longer fibers. It may be assumed that the bigger particles had appeared to the foils either by passing the filter stages or by transferring to the foil from external sources during the handling of the foils after the measurement.

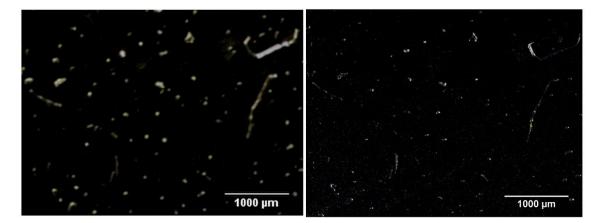
The aluminum substrate made it difficult to adjust the microscope light in a way that the particles in different areas could be seen clearly. The foil itself reflected the light and also it was uneven horizontally. The elevations left part of the areas in shadows and other parts very bright. The clarity of the photos was not as good as expected due to the lacking properties of the camera in use.

9.2 Analysis from Nikon Eclipse E400 pictures

More detailed pictures were magnified with a microscope Nikon Eclipse E400 and pictures were taken with camera Q Imaging Micro publisher 5.0 RTV. There were several pictures taken from each aluminum foil and more attention was paid to areas where bigger or deviating particles were found.

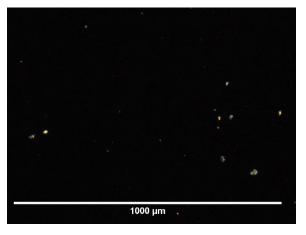
9.2.1 Results

The example of the comparison with the two analysis is given in picture 8 with Kaipola B PM10. The left picture is a processed enlargement from the picture taken with Olympus and the right one is a processed image directly from a picture taken with Nikon. There are many similarities to be distinguished but the illumination with samples taken with Nikon also showed details that are clearly from the background aluminum foil.



PICTURE 8. Kaipola B PM10 details with enlargement from Olympus picture (left) and directly processed image taken with Nikon (right)

In the more detailed pictures there are also illuminated particles, of which origins is not clearly decided. In picture 9 there is a sample of Kaipola C blanco aluminium foil. This shows that the foil only treated with spray has some particles or particle type matter on it without going through the measurements in the PM10 impactor.



PICTURE 9. Kaipola C blanco aluminum foil

The pictures taken with higher magnification were processed with ImageJ software to some extent but the main objective was to store the samples and all the taken pictures for further analysis. Samples were left for storage in scale room with controlled temperature and humidity and they were labeled accordingly. The objective was to continue the work with the samples later with an electron microscope to achieve more detailed data of the particles.

9.2.2 Discussion

The fiber-like particles were quite easy to distinguish and the details were clearer in the pictures taken with higher magnification. There were quite extensive amount of small particles that were illuminated and showed off from the background. It was not clear what these particles represented and as seen picture 8 from the blank sample there were also similar particles found in this sample. One possibility is that these particles have something to do with the collection spray to line the small particles and result as bigger particles with illuminative feature. Other possibility is that there are more particles gathered together and they look like one single particle. Further analysis should be made with electron microscope to distinguish the origins of these particles. The electron microscope was not available at TAMK premises thus some arrangements for further analysis needs to be made.

10 DISCUSSION

The mass measurements with PM10 impactor was made with too small amount of papers to make any definite conclusions. The mass measured was not high enough to exceed the errors thus bigger samples will be needed to make definite calculations and comparisons.

In the optical analysis the objectives were achieved with some exceptions. With the bigger particles at stage PM10 there were clearly visible objects and also identifiable fiber-like particles by naked eye. The detailed analysis with a microscope and camera made it simple to use ImageJ software to further process and analyze the results. Within some grades there were quite extensive variations in the results, especially with Kaipola A paper grade. The errors most probably occurred due to the poor quality of the sample pictures. However, the majority of the results were consistent with the earlier results made with some of the tested paper grades. Also the properties of the tested grades implicate that the results can be considered reliable.

Most of the problems arose from the lack of optical properties with the installations of microscopes and cameras. The illuminative feature with both tested assemblies would have needed more time to be adjusted properly. Also the type of the base, the aluminum foil, was not well suited for taking such a detailed pictures.

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APPENDICES

Appendix 1. The results of PM10 impactor weighing measurement

Stage	m _{initial}	$\Delta_{ m initial}$	m _{final}	$\Delta_{ ext{final}}$	m _{remainder}	Result (mg)
	(mg)	(mg)	(mg)	(mg)	(mg)	
PM10	19,274	0.015	19,296	0,02	0,022	0,022±0,027
PM2.5	19,24	0,015	19,248	0,01	0,008	0,008±0,021
PM1	19,238	0,005	19,234	0,02	-0,004	-0,004±0,023
Filter	311,608		311,586	0,0075	-0,022	
Blanco	19,192		19,180	0,0125	-0,012	

CCP, sample size 300

Kaipola A, sample size 100

Stage	m _{initial}	$\Delta_{initial}$	m _{final}	$\Delta_{ ext{final}}$	m _{remainder}	Result (mg)
	(mg)	(mg)	(mg)	(mg)	(mg)	
PM10	19,542	0,01	19,536	0,005	-0,006	-0,006±0,015
PM2.5	19,366	0,005	19,374	0,005	0,008	0,008±0,012
PM1	19,300	0,01	19,302	0,01	0,002	0,002±0,017
Filter	292,084		292,078	0,0075	-0,006	
Blanco	19,22		19,218	0,01	-0,002	

Kaipola B, sample size 100

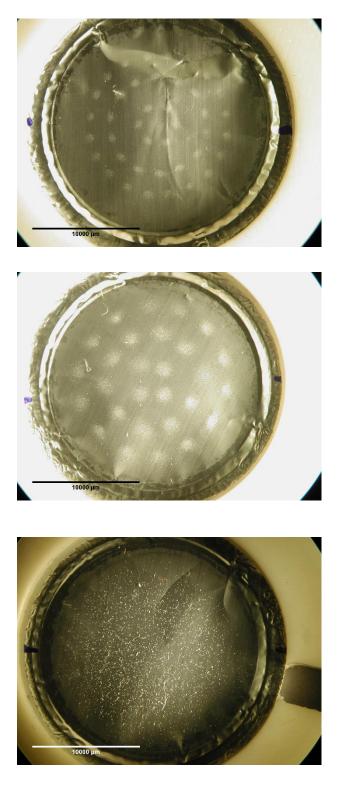
Stage	m _{initial}	$\Delta_{initial}$	m _{final}	$\Delta_{ ext{final}}$	m _{remainder}	Result (mg)
	(mg)	(mg)	(mg)	(mg)	(mg)	
PM10	19,058	0,01	19,096	0,015	0,038	0,038±0,021
PM2.5	19,322	0,01	19,348	0,01	0,026	0,026±0,017
PM1	19,152	0,01	19,166	0,01	0,014	0,014±0,017
Filter	281,974		281,988	0,0075	0,014	
Blanco	19,284		19,292	0,0125	0,008	

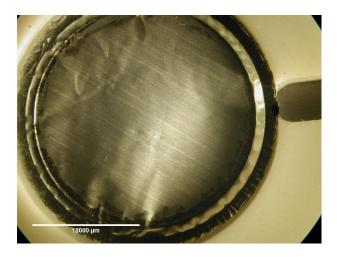
Kaipola C, sample size 100

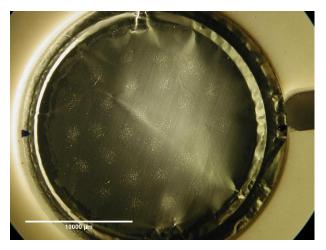
Stage	m _{initial}	$\Delta_{initial}$	m _{final}	$\Delta_{ ext{final}}$	m _{remainder}	Result (mg)
	(mg)	(mg)	(mg)	(mg)	(mg)	
PM10	19,094	0,01	19,114	0,01	0,02	0,02±0,017
PM2.5	19,202	0,01	19,21	0,01	0,008	0,008±0,017
PM1	19,222	0,01	19,234	0,005	0,012	0,012±0,015
Filter	284,346		284,374	0,01	0,028	
Blanco	19,208		19,214	0,01	0,006	

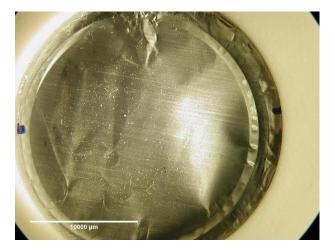
Shotton, sample size 100

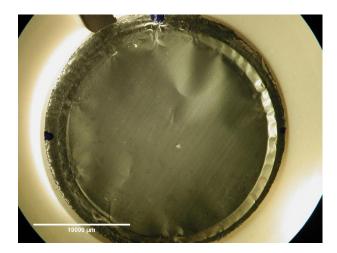
Stage	minitial	$\Delta_{initial}$	m _{final}	Δ_{final}	m _{remainder}	Result (mg)
	(mg)	(mg)	(mg)	(mg)	(mg)	
PM10	19,212	0,01	19,25	0,01	0,038	0,038±0,017
PM2.5	18,986	0,005	19,014	0,005	0,028	0,028±0,012
PM1	19,539	0,01	19,558	0,01	0,02	0,02±0,017
Filter	285,414		285,40	0,01	-0,014	
Blanco	18,976		18,974	0,0075	-0,002	

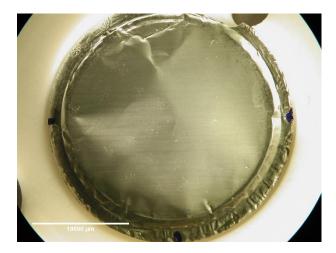


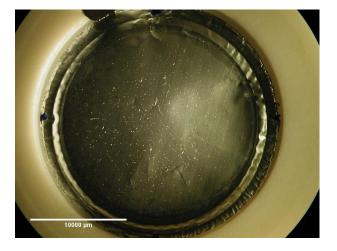


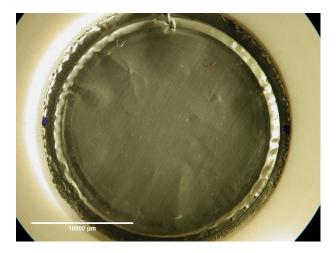


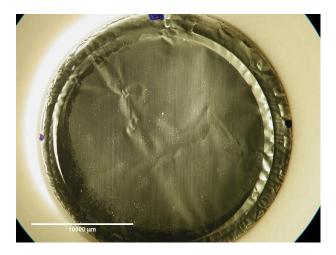


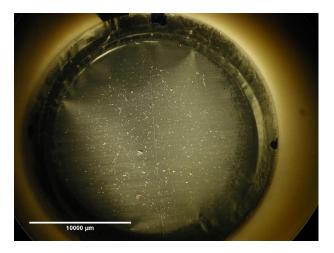




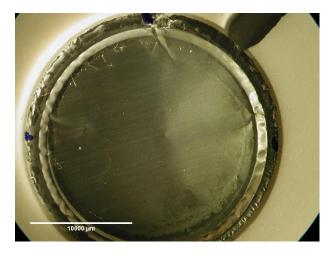


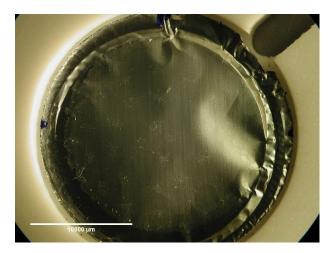


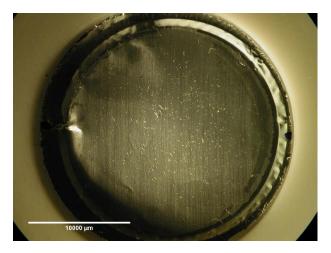


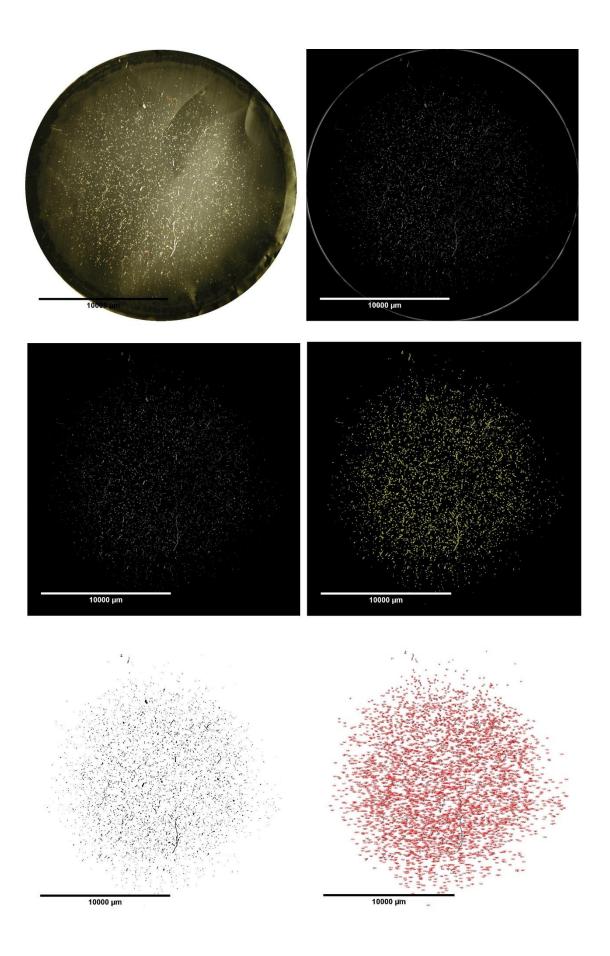


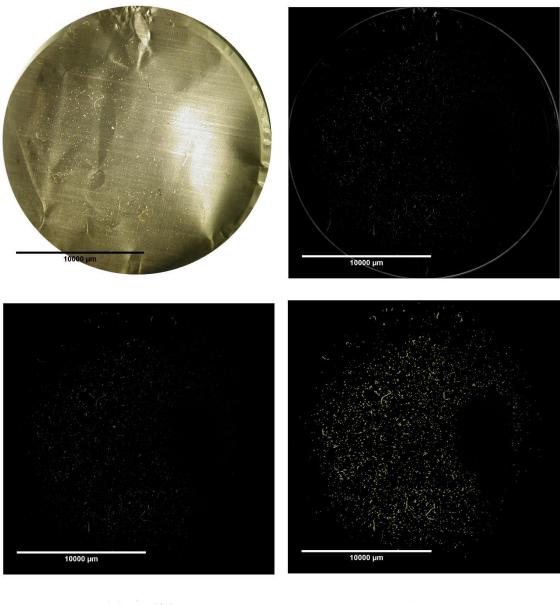
Appendix 6. Shotton pictures (PM1, PM2.5, PM10)



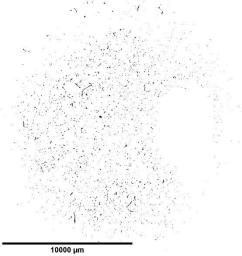


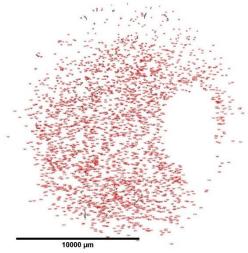


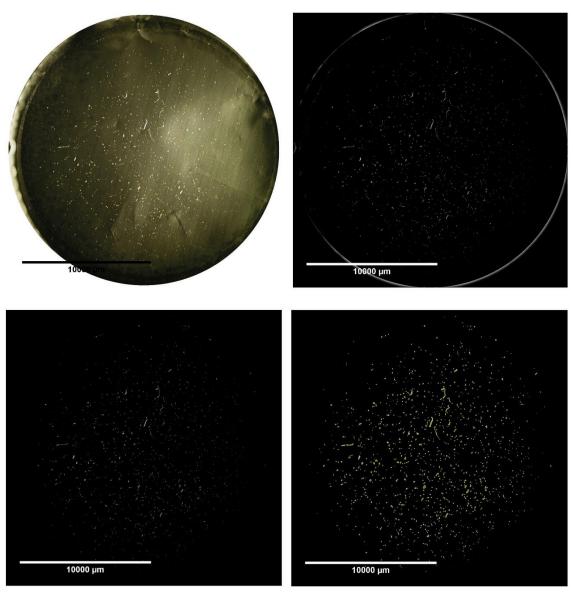




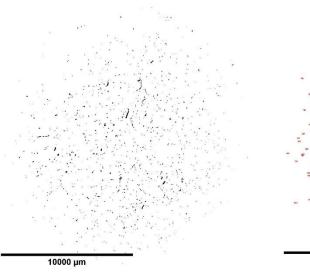
Appendix 8. Kaipola A ImageJ processing steps from stage PM10

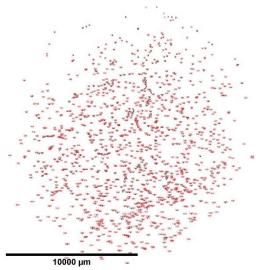


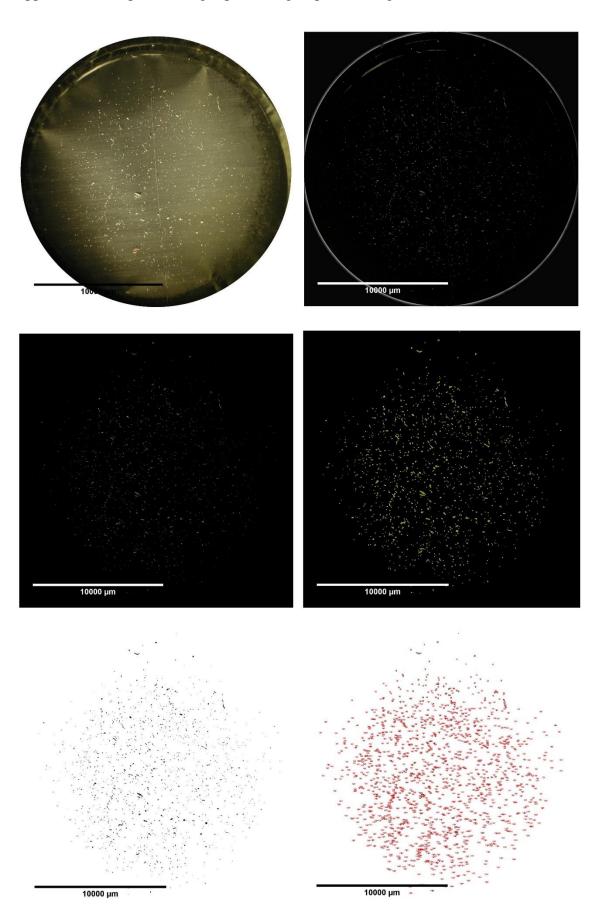




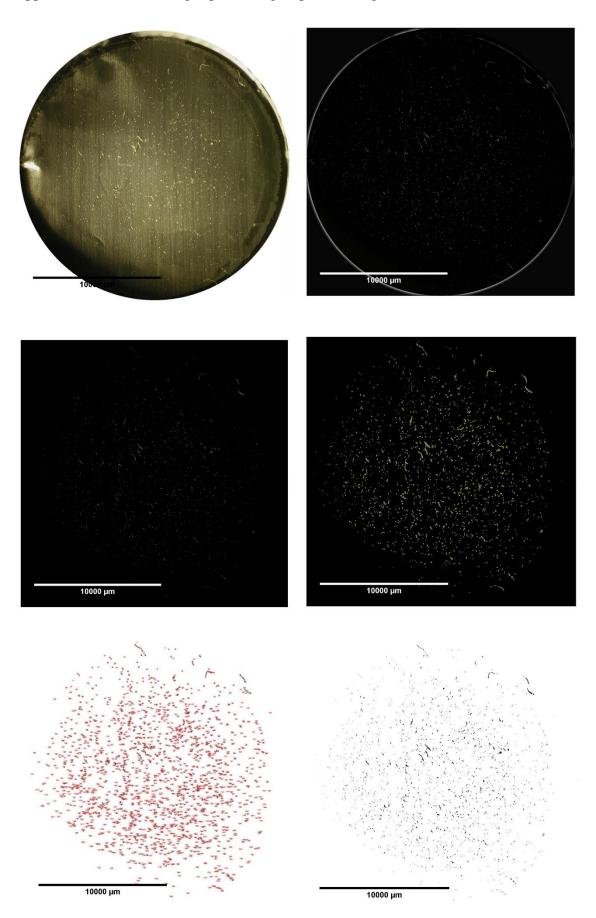
Appendix 9. Kaipola B ImageJ processing steps from stage PM10







Appendix 10. Kaipola C ImageJ processing steps from stage PM10



Appendix 11. Shotton ImageJ processing steps from stage PM10