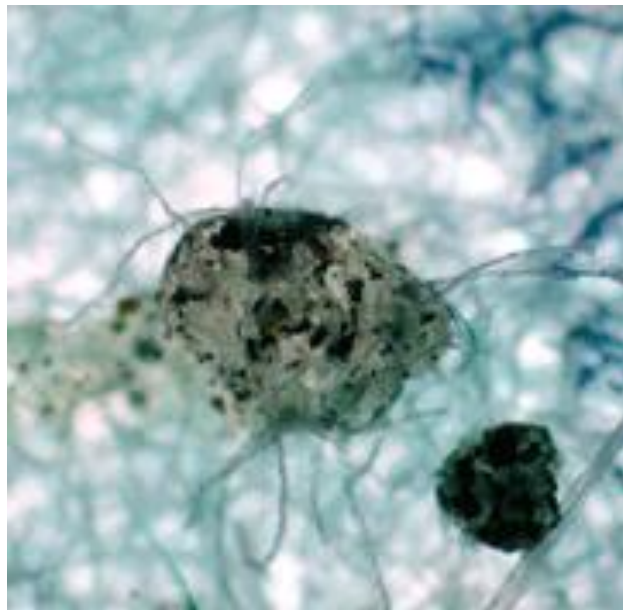


CHALMERS



Sticky Deposits in a Tissue Manufacturing Process

Master of Science Thesis

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Department of Chemical and Biological Engineering
Division of Forest Products and Chemical Engineering
CHALMERS UNIVERSITY OF TECHNOLOGY
Göteborg, Sweden, 2012

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Cover: adhesive and glue stickies contaminants on a paper-web [1].

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ABSTRACT

Sticky deposits are a common problem in mills producing paper from recycled fibers. This issue leads to inferior runnability of the paper machine, specks and holes in a final product as well as production downtime due to cleaning of wires and felts.

The project has been elaborated at SCA Edet mill. The main scope of the project has been to get an insight about the sticky deposits, find suitable methods for monitoring stickies, find out where they are coming from and why this problem occurs.

The first part of the project consist of a literature review where the main theoretical aspects of the problem are examined, it is followed by analysis of historical data where the data from SCA databases have been processed with an aim to find correlations between cleaning of wires and felts of the paper machine and stickies measurements performed at SCA laboratory. Also the effect of wet-strength agent and pulp dosage on the machine was studied.

The experimental part consists of different process surveys where variation in process parameters has been observed as a first step. Process surveys have further been performed where different stickies determination methods have been used in order to monitor stickies in the system.

From historical data it could be seen that cleaning occurs to a larger extent while running toilet paper compared to wet-strong paper. Indications that a mixture of pulp from Tower 2 and Tower 3 contribute to cleanings could be seen. Further no correlations could be seen between the stickies measurements performed and cleanings of wire and felts.

The main conclusions of the experimental part are that there are significant variations in process parameters that might contribute to the formation of deposits. The system was less contaminated while running wet-strong paper compared to toilet paper. The obtained results do not provide clear evidence whether this is due to increased retention when the wet-strength agent is added; pulp dosage or addition of fresh water.

Tower 3 is suspected to contribute to cleaning occasions and further examination is necessary.

Keywords: Macro-stickies, micro-stickies, dissolved and colloidal substances, pulp, white water, paper machine, deinking, wet-strength agent

PREFACE

The following project is the Master thesis for the degree of Master of Science in Chemical Engineering performed in cooperation with SCA Edet Mill and the Division of Forest Products and Chemical Engineering at Chalmers University of Technology.

The project was initiated by engineers at SCA Edet Mill with the aim to “control the stickies-problem at the mill”. It was invented to get an idea about why the problems arise and how they can be prevented.

First of all we want to thank our great supervisor Jonas Pihlström who has been a very important support during the project, without his skills and commitment the project would not have been possible to carry out. Mikael Pettersson and Lars Widmark, process engineers at paper machine 8 and 5 respectively, have also contributed with valuable information and ideas.

We would like to thank Jan-Erik Eriksson and other staff at the pulping department for helping us with the process mapping of the deinking lines and valuable inputs during meetings.

Great part of this project has been elaborated in collaboration with different chemical suppliers. We would like to express our gratitude to Björn Hettefeldt and staff from Clariant, Patrik Ljungdal and Kristian Forsell representing Banmkark oy AB, Helena Wassenius and team from BIM Kemi AB, they have all contributed with valuable knowledge and helped us with laboratory work.

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LIST OF DEFINITIONS AND ABBREVIATIONS

Stickies - Tacky substances found in pulp and process water system, which tend to deposit on paper machine clothing, cylinders and rolls and enter the process with recycled fiber.

MOW- Mixed office waste

ONP- Old newsprint

White water - Water that is reused within the paper making process.

CCT- Colloidal collection test

DCM- Dichloromethane extraction

DisCo- Dissolved and colloidal substances

HT-CTMP- High temperature chemothermomechanical pulp

PSA- Pressure sensitive adhesives

UV-VIS- Ultra violet- visible spectroscopy (micro-/colloidal- stickies)

PROCESS RELATED TERMS:

DIP 1- Deinking line 1, raw material: Mixed office waste (MOW)

DIP2- Deinking line 2, raw material: News Print

T1- Tower 1- Internal pulp from DIP1 (Brightness +78)

T2- Tower 2- Internal pulp from DIP1 (Brightness +75)

T3- Tower 3- Pulp from DIP2 (Brightness 52)

C4- Chest 4- Virgin short fiber pulp

C5- Chest 5- Virgin HT-CTMP pulp

C6- Chest 6- Virgin long fiber pulp

C7- Chest 7- Internal broke line

C12- Chest 12- Internal wet strong broke line

White Water - Dilution water in pulp tower 1 and 2 (Blekt BV)

White water (virgin) – Dilution water for the virgin pulp lines

MC pump – mixing pump in which pulp from screw press 2 is diluted to medium consistency before feeding it to the Tower 1 and 2 (DIP1)

DAF- Dissolved air flotation

Krofta- Dissolved air flotation equipment from Krofta Engineering. Used for removal of dissolved and colloidal substances from the process water.

PM- Paper machine

1 INTRODUCTION

In Chapter 1 the background, aim and limitations of this report are presented. In the latter part a short presentation of SCA and Edet Mill is given together with a general description of the process parts treated in the project.

1.1 BACKGROUND

Increasingly growing environmental awareness as well as competition in the paper industry has promoted the use of recycled fiber for paper manufacturing during several decades.

The use of recycled fibers has been growing rapidly. In Sweden the amount of recycled paper increases each year and during 2011, 94% of all newsprints, catalogues, magazines and paper advertisements have been collected for recycling [2]. One of the important issues that has contributed to this is the decrease in fiber supply together with rising prices on virgin pulps. Moreover, producing paper from recycled fiber is more sustainable not only because of the reduced deforestation, but the process itself is also more energy efficient (energy requirement is 70% less than for the virgin pulp) and consumes less water and chemicals[3].

Even though this opportunity is attractive from both economic and environmental standpoint, it has also several drawbacks. The major challenge for industries utilizing the recycled fiber as raw material is formation and accumulation of detrimental substances in different locations of the paper machine system resulting in quality and process-related problems [4].

These problems are mainly caused by tacky materials referred to as “stickies” that may arise from various sources e.g. hot glues used in magazines, envelopes, post-it notes, coating residues from certain sorts of paper etc. [5].

The wire section in the paper machine provides formation and dewatering of the paper web and the performance of this step is determinative for the final product properties. Utilization of recycled fibers may often lead to the deposit formation on PM clothing, which as mentioned results in quality- and process-related problems. The former includes holes’ and specks’ formation in the paper, which results in higher amount of paper reject and complaints from the customers [5]. This, in turn, leads to economic losses. The latter include runnability issues in the paper machine leading to for example breaks in the paper web. Plugging of wires and felts is one of the largest problems at SCA Edet mill and cleaning of the wires is time-consuming and has a substantial influence from the economic standpoint due to decreased production.

The pulp production process from recycled fibers can be divided into three major steps; Re-pulping where the paper is disintegrated into separate fibers or clusters of fibers, removal of contaminants e.g. ink, plastic materials, metal objects etc. and bleaching of fibers, if needed. The second step which includes several separation stages such as screening, cleaning and flotation to reduce the amount of impurities is crucial to obtain high performance of the process and desired end-product quality. The efficiency of the removal of contaminants is one of the most important factors when preventing formation of stickies [6].

1.2 AIM

The aim of the following project was to obtain information about sticky-deposits, their origin, how to detect them and possible ways of preventing them. Further the purpose was to study, describe and analyze the tissue manufacturing process at SCA Lilla Edet paper mill with focus on formation of deposits i.e. stickies in the clothing of the paper machine.

1.3 PROJECT DESCRIPTION

In order to obtain an overview of the tissue manufacturing process used at SCA Lilla Edet Paper mill a general process mapping was performed as the first step. The literature study was divided into different parts including production of pulp from recycled paper, general study regarding tissue production process and overview of deposit problems. In the latter part composition and origin of the stickies as well as their impact on the papermaking process was examined. The literature study was performed by using Chalmers library electronically data basis, relevant books, information available on internet and data provided by SCA regarding particular processes used at the mill. Information regarding stickies measurement methods was also provided by Clariant, Banmark Oy AB, Ashland and BIM Kemi AB.

After performing literature study and getting familiar with the process at SCA, the next step was analysis of the historical data. Methods currently used at SCA Edet for quantification of the sticky deposits are rather questionable in terms of their usefulness in monitoring stickies in the process. Therefore, with the intention of finding correlations to cleaning sessions, chosen data has been observed.

After analyzing historical data, the experimental part started with Process Survey 1. The aim of this was to perform preliminary mapping of the tissue manufacturing process at SCA Lilla Edet, obtain information of the variance in process parameters and correlation between different parameters as well as their impact on cleaning occasions due to stickies formation. Further, the project was focused on finding suitable methods for stickies determination and getting an overview of the stickies content in the process. To achieve this, additional three larger process surveys were performed.

1.4 LIMITATIONS

Due to time restrictions following parts are not included:

- Analysis of raw material i.e. recycled paper entering the mill
- Extensive analyses on PM5 and PM7
- Extensive surveys of the deinking line
- Detailed qualitative analysis of deposits;
- Extensive examination of methods used
- Laboratory work regarding passivation analysis (no design of laboratory or pilot equipment)
- Detailed description of the papermaking process
- Extensive work regarding the Krofta on PM8

- No analysis of stickies content in final paper have been performed

Due to the fact that external companies were involved and that many process locations were chosen, as well as the use of time consuming analysis were used, very few duplicates were performed which would be preferable.

1.5 DISPOSITION OF THE REPORT

In Chapter 1, the reader is introduced to the project and the process at SCA Edet Mill. In Chapter 2, theory regarding stickies, analysis methods etc. are presented. The methods used in the project are described in Chapter 3. Then the project is divided into sub-parts where the different process surveys, mixing experiments and method examinations are presented. All parts include an introduction to the area, method, results, discussion and conclusions. Then final discussion and final conclusions are presented in Chapter 11 and 12 respectively. Finally, future perspectives are discussed in Chapter 13.

1.6 SCA and Edet Mill

SCA Lilla Edet mill is a tissue manufacturing site located North of Gothenburg. The mill manufactures tissue and wet-strength grades on three different paper machines; PM5, PM7 and PM8. On PM5 mainly low budget grey paper, i.e. toilet paper, household towel and wipers are produced. On PM7 premium toilet paper and household towels are manufactured. Finally, on PM8 Away-From-Home toilet paper and wipers are produced. Depending on the grade and quality of desired end product, the furnish is made up of different amounts of recovered and virgin fibers. Commonly 95% recovered fiber is used in the process.

SCA is a global company. During 2011 the annual sales reached SEK 106bn and the company had about 44.000 employees. The company develops and produces products within personal care products, tissue, forest products and packaging solutions [7].

1.6.1 PROCESS DESCRIPTION

SCA Edet mill is a paper mill i.e. does not have any pulp production on the site. Though the pulps used in the process must be treated before they enter the paper machine. For virgin pulps this process is very simple. The pulp-sheets are fed into the pulper together with dilution water before they are fed into the pulp chests. Before the recycled fiber reaches the machines it has to be deinked and separated from other detrimental substances. This is done in the deinking plant. The mill has two deinking lines; DIP 1 for mixed office waste and DIP2 for newsprint. From DIP 1, pulp with brightness higher than 78 is fed to bulk Tower 1 and the rest with lower quality to Tower 2. Pulp from DIP2 is fed to Tower 3 and is unbleached, i.e. has a lower brightness, approximately 50. See more detailed description for the individual deinking lines in Appendix A Figure 1 and 2. How the different parts work in the deinking lines is generally described in Chapter 2.2.2. In the project, pulp from different sources will be handled and they are presented in Table 1-1.

Table 1-1. Pulp sources used in the process, their origin and properties.

Process name	Pulp origin	Pulp properties
Chest 4	Virgin short fiber pulp: Hardwood (Eucalyptus) Kraft/Sulfate process	Short flexible fibers for surface and bulk softness
Chest 5	Virgin pulp Softwood HT CTMP	Stiffer fibers, more open structure for adsorption
Chest 6	Virgin long fiber: Softwood (pine, spruce) Kraft/Sulfate process	Soft wood fibers for wet-strength
Chest 7	Pulp from the internal broke line	Mixed Fibers
Chest 12	Pulp from the wet-strong broke line	Mixed Fibers
Tower 1	Pulp from DIP 1: Recycled mixed office waste	Mixed Fibers Medium qualities (sulfate, softwood, hardwood) Brightness +78
Tower 2	Pulp from DIP 1: Recycled mixed office waste	Mixed Fibers Medium qualities (sulfate, softwood, hardwood) Brightness +75
Tower 3	Pulp from DIP2: Recycled Newsprint	Bad quality but low cost Brightness 52

The pulps from the pulp towers (from pulp chests for virgin pulps and broke line pulps) are refined and then fed into the machine chest. The refining is performed in order to beat the pulp to make it more flexible and to generate a fraction of fines all to improve inter-fiber bonding (strength properties) [8].

On PM8 the pulps are fed into the paper machine in different layers from 3 machine chests: bottom layer called Yankee layer, middle layer and top layer called Hood layer (see flow-sheet in Figure 1-1). After the machine chests, the pulp is diluted in several steps passing mixing pumps and machine screens before it finally reaches the headbox which distributes the pulp suspension on the wires. Along the paper machine the pulp is dewatered and the paper-web is formed [6]. Water that leaves the paper-web enters the wire pit and parts of it are cleaned in the white water system and the rest is recycled in the short circulation. On PM8 the white water is treated in the Krofta by using dissolved air flotation, DAF, which is briefly described in the theory section 2.2.2. The clear filtrate from the Krofta is used as dilution and spray water on PM8.

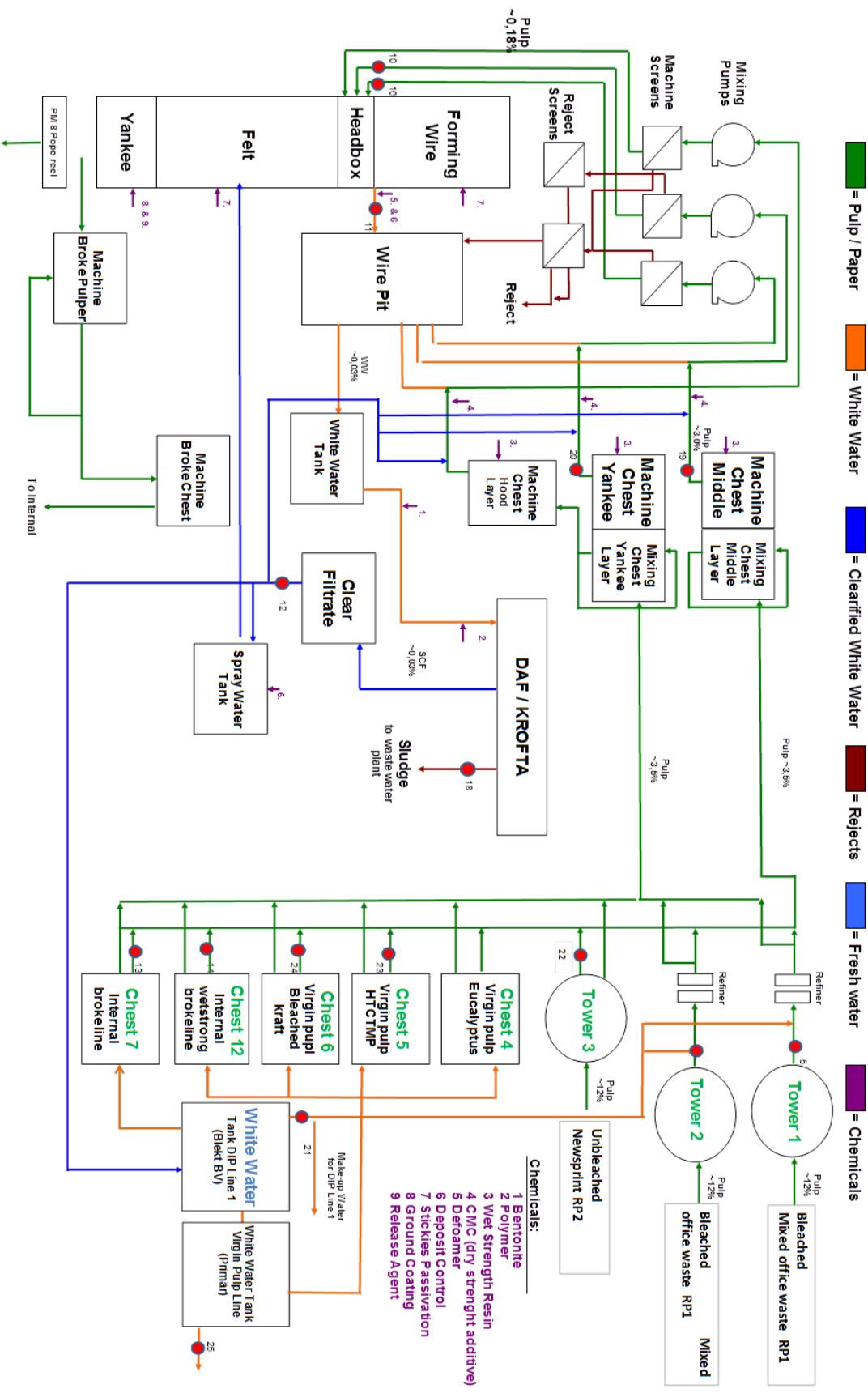


Figure 1-1 Flow-sheet for the PM8 system. Sampling points are marked with red dots.

2. LITERATURE REVIEW

In Chapter 2, background to stickies, unit operations used in the paper recycling process and theory regarding methods used in the project is presented.

2.1 STICKIES

In this part the term stickies is described more in depth, classification, deposition mechanisms etc. are discussed. Further, classification of stickies determination methods is presented.

2.1.1 INTRODUCTION

As mentioned, the problems caused in the papermaking process when utilizing recycled fibers arise due to so-called stickies. Stickies are defined as tacky constituents which originate from the raw material (waste paper) used in the paper recycling process [5].

Stickies can be located in pulp and process water as well as in the final paper product [9]. It must be noted that the term "stickies" does not presume any particular chemical composition, it is rather a mix of organic substances coming into the process with the raw material. Properties they have in common are hydrophobicity, tackiness, low surface energy, softness and deformability [10]. Stickies melt and flow at 90-95°C and 0,8 bar in a heated press[11].

The tackiness can be defined as "the tendency of a material to adhere" as a result of collision with the surface. In order for the particle to develop tacky properties its glass transition temperature (T_g) must be exceeded. The majority of organic deposits exhibit T_g values in a range from ~30° to 60 °C which is a usual temperature range for papermaking operations[12].

Major source of stickies are pressure sensitive adhesives (PSA) due to their permanent thermoplastic state which ensures lasting tack. These substances are contained in post-it notes, labels used for marking prices on different products etc. Another group of substances that are considered as stickies are hot melt adhesives which are solid at room temperature and soften at temperatures between 65°C and 120°C, but it can also occur at room temperature which is mainly predetermined by their chemical composition. Stickies of this type tend to deposit mainly on stationary parts of the equipment such as pipes and chest walls. However, they influence movable parts as well, predominantly wires, felts and rolls. Materials classified as hot melts are mainly plastics, such as polyethylene and polypropylene [9].

Virgin pulps may contain stickies in form of wood pitch. Wood pitch is a mixture of resin acids, fatty acids, natural oily materials and they are of hydrophobic nature. Moreover, they tend to be sticky and therefore are prone to contribute to the deposit formation in the paper machine [13].

Chemical analysis of sticky-deposits at SCA Edet Mill have indicated component such as, talc, calcium carbonate, latex, silicone, tri glycerides, poly-phenols and amides [14].

2.1.2 CLASSIFICATION

There are two major ways of classifying stickies [9]:

- According to their origin:
 - Primary stickies;
 - Secondary stickies.
- According to their physical or chemical properties:
 - Micro stickies;
 - Macro stickies.

Primary stickies are tacky substances that enter the process with the raw material. These substances are adhesives coming from hot melt glues, binders present in book-backs, envelopes, sticky notes, magazine coatings etc. They mainly consist of organic materials, e.g. styrene-butadiene, styrene acrylic latex binders, rubber, vinyl acrylates, polyisoprene, polybutadiene etc. [15].

Secondary stickies are formed as a result of physical and chemical interactions occurring during the manufacturing process [5]. A potential reason for formation of the secondary stickies is shock-type alteration in critical process parameters, such as temperature, pH and charge which promote colloidal destabilization and agglomeration of dissolved and colloidal substances [9].

Classification into micro- and macro- stickies is performed by the size of the tacky substances. This is determined by laboratory screening where macro-stickies are the particles retained on 100 μm or 150 μm laboratory screens and micro-stickies refer to the tacky particles that pass through these screens but are larger than 1-5 μm [5]. According to Doshi, micro-stickies can be further classified into suspended stickies (20-100 μm), dispersed stickies (1-20 μm), colloidal stickies (5-0,01 μm) and dissolved stickies (<0,01 μm) [16].

The classification according to the size is necessary due to different removal approaches for micro- and macro-stickies, specific methods used for their quantification and different strategies for minimizing their impact on papermaking [11]. Moreover, size and concentration of stickies have a great influence on the paper quality and the runnability of the paper machine [17].

2.1.3 DEPOSITION MECHANISMS

In order to tackle the formation of stickies in the process it is important to understand their formation and accumulation mechanisms. The first step is to understand what kind of stickies that are involved (micro-, macro-, primary, secondary). There are two main mechanisms due to which deposition may occur: impact deposition due to the tacky nature of stickies and flow deposition due to destabilization of colloidal material [18].

Figure 2-1 schematically describes possible interactions between recovered paper, virgin pulp and chemical additives. Pitch present in virgin pulp may create a synergic effect with micro-stickies present in deinked pulp and destabilize each other [18]. Chemicals used in the process may destabilize the detrimental substances present in the process and cause formation of secondary stickies. As it was mentioned before, stickies are rather complex substances and might incorporate both organic and inorganic matter.

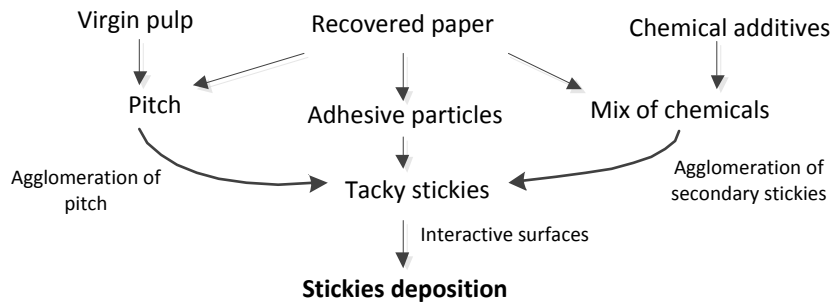


Figure 2-1 Interactions contributing to deposition of stickies [9].

2.1.4 DESTABILIZATION OF DISSOLVED AND COLLOIDAL MATERIAL

Due to environmental awareness and economic issues the use of water in a paper manufacturing process has been drastically decreased compared to conventional processes. This has been achieved by process closure and maximal reuse of process water with several treatment steps. Even though this trend has been evaluated as a great success, process closure leads to several problems which are rather hard to tackle. One of the most common problems is accumulation of the dissolved and colloidal substances (DisCo) in the white water loops [19].

DisCo are introduced into the process with the raw material (recycled or virgin fibers), process chemicals and fresh water [18]. Particular attention here is devoted to DisCo since it is the only fraction of the contaminants that cannot be removed by conventional processes and therefore are problematic [18].

DisCo are anionic substances, also termed as “anionic trash” due to their negative charge. They are potential deposit formers which may form stickies when destabilized by physiochemical shock such as electrostatic shock, temperature shock and evaporative destabilization [20]. The mechanisms of stickies deposition due to colloidal destabilization are schematically described in Figure 2-2.

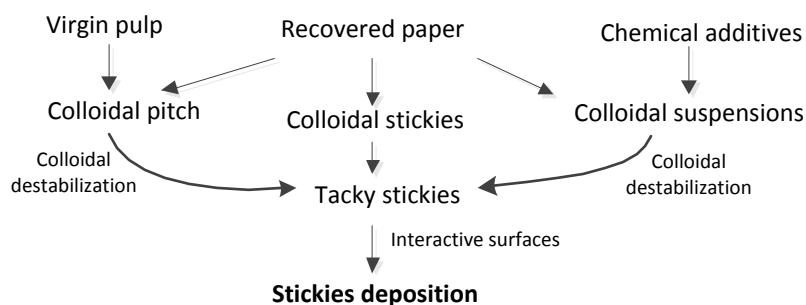


Figure 2-2 Interactions contributing to formation of secondary stickies [9].

Electrostatic destabilization occurs from cationic polyelectrolytes such as retention, dewatering or wet-strength additives. These process chemicals tend to react with anionic colloids and dissolved materials present in the process water and as a result the precipitates are formed. Another cause for the electrostatic destabilization may be a conductivity or hardness shock [21].

Hardness shock occurs due to pH variations in the processes where calcium carbonate (CaCO_3) is used as filler. Solubility of CaCO_3 increases with decreasing pH. At a pH below 8 the dissolution of CaCO_3 starts to be substantial and as a consequence, concentration of free calcium ions is increased [22]. Calcium ions react with anionic trash and reduce the cationic demand, which leads to the lower efficiency of cationic papermaking additives, for example wet-strength agent. This, in turn, results in accumulation of detrimental substances in the papermaking system. Dissolved calcium ions can also destabilize and agglomerate colloidal particles, leading to deposits on the paper machine clothing and spots in the finished paper furnish [23]. Another aspect of CaCO_3 is its decreasing solubility at increasing temperatures. As a result, deposition and scaling may occur on heat-exchanging surfaces in the process. The problems related to dissolved calcium ions are more severe, though [24].

Evaporative destabilization may occur in the drying section where the water evaporates and the concentration of the detrimental substances increases leading to formation of precipitates. During the first part of the drying, when the free water still is present between the fibers, water flow towards the paper surface takes the concentrated DisCo with it. The temperature in the drying section is above the softening temperature for a large part of the synthetic polymers causing their aggregation and formation of sticky deposits. These deposits remain in paper or stick to dryers or felts [20].

2.1.5 STICKIES DETERMINATION METHODS

A large number of stickies measuring methods can be found in the literature. There are numerous ways to classify the existing methods [9] for example:

1. By the size of stickies which are determined:
 - Macro
 - Micro
2. By the mechanisms used:
 - Screening
 - Adsorption
 - Deposition
 - Precipitation
 - Extraction

2.2 UNIT OPERATIONS IN PAPER RECYCLING PROCESS

There are two major ways for handling the stickies in a paper manufacturing process, i.e. removal and prevention of remaining stickies from deposition. The first priority is to remove as much stickies as possible [5]. This is performed in unit operation steps described in following chapter.

2.2.1 INTRODUCTION

As mentioned, the process for handling of recycled fibers is more complex than the one for processing of virgin fibers. This is, firstly, due to fact that waste paper consists of mixture of different fiber types or paper grades. The other reason is the presence of contaminants and detrimental substances which can contribute to stickies formation [25].

In order to meet quality requirements, contaminants must be removed. If contaminants cannot be removed their effects must be eliminated. This is achieved by choosing appropriate processing steps that are suitable for the particular process and quality requirements [25]. The term “cleanliness” is normally used with regard to optical, chemical, colloidal, microbiological and processing aspects, e.g. sand content and stickies [25].

Production of recycled paper most often consists of the following operation steps [6]:

Pulping→ Cleaning→ Screening→ Washing→ Flotation→ Dispersion → Bleaching

The sequence of separation steps is constructed in a way that contaminants and detrimental substances are eliminated step by step using different separation criteria, such as particle size, shape and deformability, density and surface properties. This sequence of unit operations mainly depends on properties of the raw material and desired properties of the end product [6].

2.2.2 SUB- STAGES IN RECYCLING PROCESS

PULPING

A paper recycling process starts with collection and handling of the recycled paper. Next step is sorting and feeding. In order to utilize the recycled paper as much as possible the recycled paper is sorted into several categories [6]. In this stage also unwanted contaminants that may demolish the equipment and accumulate in the system are removed. This is done manually or by fully- or half-automatic sorting (introduced in Sweden and Germany).

After the sorting system the recycled paper is further fed into a conventional high or low consistency pulper or alternatively into a pulping drum. This step serves for liberation of the fibers by dissolving the raw material as well as removal of ink and coating from the paper surface. Residence time is in average 20 minutes [6]. The performance of the pulping operation is an important unit operation since it affects efficiency of the sub-processes, pulp properties at different stages as well as the final pulp [26].

SCREENING

Further separation of the contaminants (polymers used in catalogue backs, coatings, glues and other adhesives, metal clips) is obtained in a screening operation where the separation is achieved based on the size, shape and deformability of the detrimental particles [25]. In order to achieve desired degree of purity several screening steps can be introduced: course screening, pre-screening and fine screening [6].

The screening efficiency is mainly influenced by operating parameters such as stock consistency, rotor speed, pulp throughput, reject ratio as well as pulp characteristics such as freeness, fiber length distribution, ash content and size distribution of contaminants [25]. Removal of macro-stickies by screening is claimed to range from 40% to 90% (depending on the stickies quantification method) [9].

A group of researchers from Spain found that amount of micro-stickies coming out from the screening at medium consistency is higher than the one coming into the screen. According to them, this could be due to shock of calcium which results in sticky calcium soap micro-stickies [10].

Fine screens increase the amount of micro- and secondary stickies, which can be explained in two ways. Firstly, shear forces can break down parts of the larger sticky particles that are removed and thus increasing the amount of smaller stickies. The other reason could be dilution of the pulp with white water from the paper machine which might contain high amounts of micro-stickies [10].

The screening step, especially fine screening is the most effective way of removing macro-stickies. However, some part of the macro-stickies can still squeeze through the screens due to their elasticity. A sequence of course and fine screens is normally introduced in order to avoid the plugging of the screens, to reduce the loss of fibers and to capture as large fraction of stickies and other contaminants as possible [27].

One should note that the data describing the removal of stickies, to a large extent, depend on the method used for quantification of the sticky particles. Since completely reliable methods have not been developed so far, this data only gives a rough picture in order to be able to evaluate efficiency of different separation steps in the deinking line [25].

CLEANING

Cleaning is referred to as a process unit operation where contaminants are removed by means of centrifugal forces [25]. The cleaning step is performed by hydrocyclones also called centrifugal cleaners where contaminants and detrimental substances are separated from the pulp fibers by differences in densities and hydrodynamic characteristics. Heavy weight cleaners remove particles that have high density such as sand, metal, glass and part of shives (also referred to as coarse cleaning in Figure 2-3). Light-weight cleaners on the other hand remove contaminants with lower density than the water such as low density plastic materials [6].

The benefit of the cleaning step compared to the screening is the ability to remove very small and soft particles which might not be removed by screens due to their size and deformability. These particles have considerably lower density than the water and wetted fibers and will therefore be removed in this step [25]. However, cleaning does not remove dissolved and colloidal material [10].

Since sticky particles cover a wide range of substances, they can have higher, same or lower density than water. Removal of stickies in this stage is claimed to be from 20 to 40% depending on the type of cleaner (forward-, reverse-, through-flow or rotating). However, removal of adherent particles in this step is rather low due to the fact that cleaning is inefficient for removal of particles with density around 1 g/cm³. Therefore, in order to achieve desired level of stickies removal, several cleaning steps must be introduced. This is also done in order to minimize the loss of fibers [25].

DISPERSING AND KNEADING

Dispersing is achieved in dispergers which make residual specks and stickies smaller or floatable, detaches ink still adhering to the fibers, mixes in bleaching agents and conditions fibers technically [25]. It has been observed that an increase of 60% in micro-stickies is obtained after this process stage [28].

FLOTATION

In the flotation step a large range of particles are separated based on surface chemistry [6]. Flotation provides a high fiber yield but poor physical strength of the stock due to a high recovery of filler and fines [29].

During flotation, air is introduced to low consistency pulp (0,8-1,5%) and hydrophobic particles will rise to the surface when attaching to the air bubbles. The ink containing froth formed in the process is removed mechanically at the top of the flotation cell, by overflow or by applying vacuum extraction [30]. As mentioned, flotation is based on surface chemistry and requires hydrophobic and larger particles compared to washing which is favored by hydrophilic particles.

Several surface chemical sub-processes take place in a flotation deinking mill and the main steps are detachment of ink from fibers, ink agglomeration, flotation and froth formation. Redeposition of ink onto the fiber is unwanted but will occur and is particularly a problem for water-based inks. Agglomeration of ink particles is required to achieve flotation, since small particles will not attach to the bubbles due to hydrodynamic forces. The momentum of the small particles is too low and they will follow the stream line around the bubble while larger particles will collide with it and under favorable conditions interact and attach to it. To achieve agglomeration and further improve the chemical interactions between ink particles and air bubbles, collectors, often fatty acid calcium soaps, are used [6].

Theoretically, flotation should contribute to effective removal of micro-stickies due to their hydrophobicity which facilitates attachment of the sticky particles to the air bubbles in a similar way as ink particles. Moreover, detrimental particles entering the flotation step are in the size range which is favorable for this operation (10-100 μm) due to pre-screening where most of the large stickies are removed [6]. Researchers from Paper Technology Centre (CTP) in Grenoble found that 81% of micro-stickies are removed in pre-flotation stage. They also observed that post-flotation stage is able to remove stickies in both macro- and micro- range [28, 31].

Most of the micro-stickies, introduced in the process in the dilution step after the dispersion, are removed in post-flotation step [28]. However, particular conditions during flotation may result in decrease of the hydrophobicity of the sticky particles leading to lower separation efficiency. This can be due to that surface tension of adherent particles is time-dependent resulting in decrease in surface tension of the stickies after addition of deinking chemicals. The other reason can be adsorption of surfactants on the surface of stickies [25]. Poor removal of the stickies in favorable size range can also occur due to disk-shaped structure of adherent particles which are not removed by air bubbles [25].

WASHING

During washing, which in principle is a filtration process, particles smaller than 30 μm are removed from the pulp suspension. At the same time, dissolved and colloidal contaminants end up in a filtrate and are removed. Materials that are removed in the washing stage include fillers and coating particles, fines, micro-stickies and ink particles [25]. No information can be found about efficiency of the washing stage in terms of stickies removal due to lack of reliable methods for measuring stickies in this size range. Therefore, most often ash content is normally used as an indicator for the efficiency of this stage [9].

DISSOLVED AIR FLOTATION (DAF)

A deinking line can contain up to four DAF units (1st loop, 2nd loop, paper machine loop, effluent, sludge) which is determined by the quality demands of the pulp [5]. DAF is used for removal of ink, stickies, pitch and other suspended solids such as fiber fines and clay from the process water and effluents. In DAF anionic trash and fines agglomerate by using polymeric flocculants in order to be able to remove them by flotation [32].

Contrary to flotation operation, DAF is not selective. This is a result of a pressure drop which is created when the dissolved air is discharged into the water creating very small bubbles. Micro

bubbles tend to collect all dissolved matter and bring it up to the froth. DAF is however found to be effective in removal of micro-stickies and wood pitch [5].

2.2.3 SCA EDET MILL

The raw materials used for production of tissue at SCA Edet mill are as mentioned bleached office waste, newsprint and virgin fiber. Since these fibers are of different quality there are two deinking lines – DIP1 and DIP2 (see Figure 2-3a and 2-3b). More detailed flow-sheets of DIP1 and DIP2 are given in Appendix A.

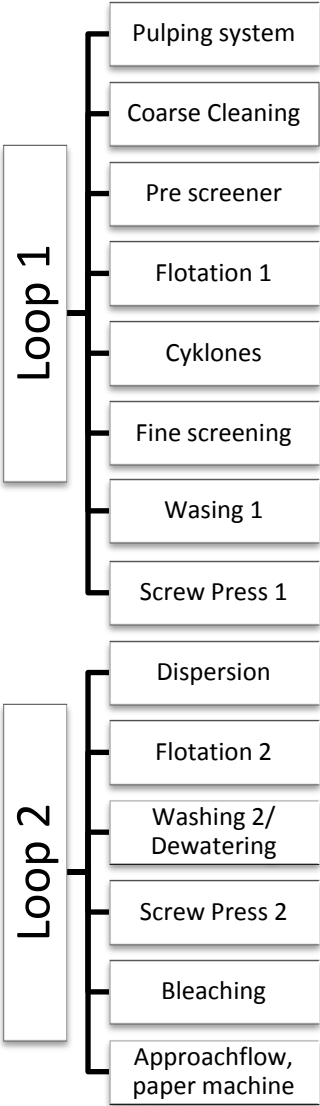


Figure 2-3a Outline deinking line 1 (DIP1). The raw material is mixed office waste. Loop 1, water circuit from pulping system until the first pressing to high consistency. Loop 2, water circuit from disperser to press 2 and approach to paper machine.

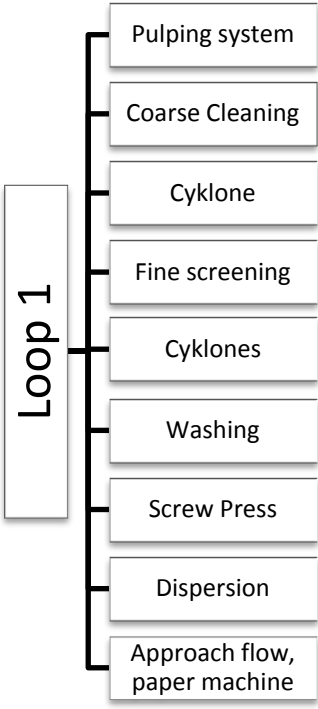


Figure 2-3b Outline deinking line 2 (RP2) for tissue paper grades at SCA Lilla Edet. The raw material is newsprint. Loop 1, water circuit for RP2 from pulping system until approach flow to paper machine.

2.3 CONTROL OF STICKIES

As mentioned, stickies can be tackled by removal and prevention from deposition. The detrimental substances that are not removed in different unit operation steps must be treated to avoid deposition. It can be achieved by different actions. First of all, equipment materials must be selected with great care since it may significantly influence the deposition. Another option is to use chemicals for protection of the equipment. Then, the deposit tendency of stickies can be reduced by using wet-end additives. Finally, it is important to maintain a stable process to avoid pH, temperature, conductivity or charge shocks [5].

2.3.1 CHEMICAL TECHNOLOGY FOR CONTROL OF STICKIES

There are five main technologies developed for the chemical control of stickies: dispersion and fixation with cationic polymers, detackification, passivation and use of cleaning solvents [5, 33].

DISPERSION AND FIXATION

Dispersion of stickies is performed in order to improve the retention by breaking down stickies into small, discrete particles. As a result of increased retention accumulation of stickies in the white water is prevented since they end up in the final paper product and are thus removed from the process. Dispersion chemistry also acts in a way to prevent reagglomeration of stickies by stabilizing them. Chemicals are normally added in the pulper to improve the sheet quality, avoid deposition of stickies in dry-end of paper machine and solve converting problems [33].

When thinking globally, the advantage of this method is lessened since stickies may re-enter the process with the recycled paper [5].

Fixation, using low molecular weight and high charge density cationic polymers, is used to improve the attachment of the dispersed stickies to the anionic fibers. Patented cationic polymers used for stickies control are Poly-DADMAC together with acrylic acid or acrylamide. It is also stated that fixatives may have a positive influence on increasing the interactions between mechanical pulp and DIP [5].

DETACKIFICATION

Detackification is “the elimination of the contact adhesion properties” of stickies surfaces. It is also performed to avoid agglomeration of stickies and formation of large particles. This strategy is based on adsorption of inorganic detackifying agent on the surface of sticky particle and thus reducing its tackiness [33]. The most common chemicals used for this purpose are talc, bentonite, ground calcium carbonate (GCC), precipitated calcium carbonate (PCC) and aluminum chemicals [5].

The mechanisms of the detackification depend on the chemical used. For example, one of the most popular detackifiers, talc, is hydrated magnesium silicate with a layered structure. Due to its hydrophobic nature (on the surface of the platelets) talc interacts with stickies, adsorbing on their surface thus shielding the tacky surface of stickies [5, 33].

WIRE TREATMENT (STICKIES PASSIVATION)

Sticky deposition can be prevented by creating a soluble film that keeps the surface of the paper machine fabrics or rolls free from stickies. Neutralization of the surfaces is obtained by stabilization of contact surfaces. The chemicals used for this activity are usually cationic, low molecular weight and high charge density polymers, e.g. water soluble polymers containing acrylamide and vinyl acetate [5, 33].

Wire treatment is performed to prevent stickies-deposition but it does not remove the stickies already present in the process [33].

USE OF CLEANING SOLVENTS

Solvents are used for cleaning the wires and felts from sticky deposits. In this activity stickies are made soluble in order to remove them from the fabrics. Cleaning should be performed as efficient as possible and solvents should not be returned to the white water since it may cause aggregation when solubilized stickies come in contact with stock [33].

2.4 WASTE PAPER CATEGORIES

The quality of the raw material entering a process will determine the properties of the end product to a great extent. The raw materials normally used in manufacturing of recycled paper is mixed office waste (MOW), old newsprint (ONP), old magazine (OMG) and old corrugated containers (OCC) [6]. Only the two former categories will be described more in detail since they are used at SCA.

ONP mainly consists of mechanical, groundwood or thermomechanical pulp. However, admixture of chemical fibers in these furnishes may reach up to 30 weight percent. Besides the fibers, ONP contains the papermaking additives used for production of the newsprint, such as starch, inorganic fillers and dyes [34]. The amount of these substances in ONP is rather low, i.e. ash content is around 3-15% and therefore it exhibits high fiber yield [6]. Finally, ONP contains 1-2 weight percent of printing ink [34].

MOW is bleached chemical pulp from printing and writing papers. MOW has rather high filler content (15-35%) and it is relatively hard to deink. It is seen as a high quality raw material compared to old news print [6].

2.5 ULTRAVIOLET- VISIBLE SPECTROSCOPY, UV-VIS

The UV-VIS method, Ultraviolet-visible spectroscopy is an absorption spectroscopy in the ultraviolet-visible region (190nm-800nm) [35]. The UV-VIS method is most commonly used to determine the concentration of light absorbing species in a solution by using Beer-Lambert law [36].

The principle of the method is that radiation passes through the sample and the intensity of the outcoming light (residual radiation) from the sample is detected. The residual radiation will pass a prism and yield an adsorption spectrum [35].

During measurements, electromagnetic radiation is absorbed and atoms or molecules will undergo energy transitions. When a molecule absorbs energy electrons will be excited from their (lower) ground state to a higher energy level. The electrons pass from an occupied molecular orbital to a high potential energy unoccupied orbital. The energy difference between the levels is equal to the energy absorbed in the sample, see equation (1).

$$\Delta E = E_{excited} - E_{ground\ state} \quad (1)$$

Depending on the sample composition the electrons will undergo different transmissions yielding different absorption spectra. The extent of light absorption is dependent on number of present molecules that are capable to absorb at the certain wavelength and the efficiency of the absorption. The absorbance can be calculated by using Beer-Lambert Law see Equation (2) [35]:

$$A = \log\left(\frac{I_0}{I}\right) = \epsilon cl \quad (2)$$

Where A is the absorbance, I_0 is intensity of the incoming light to the sample, I is the intensity of light leaving the sample, c is the concentration of solute and ϵ is the molar absorptivity.

From a stickies point of view, organic compounds are interesting and these absorb light in the UV or visible region of the electromagnetic spectrum. Many systems only absorb light in the UV-region and are therefore colorless to the human eye. By using the UV-VIS method a quantification of the light absorption in the ultraviolet to visible region can be done forming a base for the entire field of photochemistry [36].

The measurements provide an absorbance value which is proportional to the number of absorbing molecules (at a certain wavelength) in sample. The light intensity out from the sample is reduced by both absorption (when light passing dissolved and colloidal particles) and reflection on surface of larger particles.

When using UV-VIS for monitoring stickies the absorbance is measured at 200 and 400nm. This is in order to distinguish unsaturated groups which are present in pitch, stickies and coating binders around 200 nm. At this wave length loss of light intensity occurs due to reflection and absorption. At 400 nm no resonance with chemical groups takes place and therefore loss of light is only due to reflection. The value obtained from the UV-VIS spectrophotometer is the difference between the absorbance values at 200 nm and 400 nm. This is performed in order to avoid inclusion of undesired substances in the result [36].

The UV-VIS method, supplied by Ashland, is quite easy to handle and provides a quick result. It though has to be taken in to consideration that the method requires experience and does not work in all systems [36].

2.6 CATIONIC DEMAND, COLLOIDAL CHARGE MEASUREMENTS

Process waters in papermaking industry tend to exhibit high amount of dissolved and colloidal material due to high process closure [13]. These materials are mainly anionically charged and may react with cationic additives in a papermaking process. Since this mainly is an undesired effect these substances are often termed as “anionic trash” [37].

In order to determine the amount of anionic material titration with cationic polymer as a titrant is used. The obtained result is regarded as “Cationic Demand” since it describes how much cationic polymer is needed to neutralize the anionic material. The cationic Demand value is considered as empirical since it is dependent on the titrant used. It is normally expressed in $\mu\text{eq/L}$ and is calculated from (3) [37].

$$X = \frac{c \times V_{\text{titrant}}}{V_{\text{sample}}} \times 1000 \quad (3)$$

Where X is the cationic demand, in micro equivalents per liter; c is the charge concentration of the poly-DADMAC solution, in milliequivalents per liter; V_{titrant} is the consumed volume of the poly-DADMAC solution, in milliliters; V_{sample} is the volume of the test portion, in milliliters. The result is expressed in micro equivalents per liter. Most often the charge concentration of the poly-DADMAC solution is 1 meq/l and therefore the cationic demand can be calculated as:

$$X = V_{\text{titrant}} \times 100 \quad (4)$$

2.6.1 DIFFUSE DOUBLE LAYER THEORY

Colloidal and dissolved substances form an ion cloud corresponding to the double layer model [38]. The diffuse double layer theory assumes that a particle, when dissolved in water, acquires a charge on its surface. This charged surface attracts oppositely charged ions and forms a Stern Plane which is bound to the surface of the particle. Similarly, counter ions are attracted to Sterns Plane and form a so-called diffuse layer which spreads out in bulk solution. A diffuse layer is not bound to the surface of the particle (see Figure 2-4) [39].

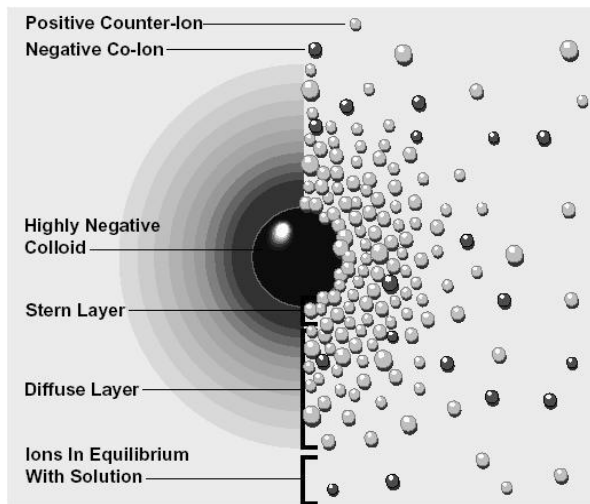


Figure 2-4 Two ways of displaying the double layer: on the left-hand side the change in charge density around the colloid is shown. On the right-hand side distribution of positive and negative ions around the charge colloid is displayed[40].

2.6.2. STREAMING CURRENT (SC) METHOD

Typical SC device is presented in Figure 2-5. The sample is inserted into the cylindrical vessel which is made of non-conducting plastic material, most often from PTFE. The cylinder is narrow in the bottom and wider on the top. The PTFE piston, which fits closely into the narrow section of the cylinder, is moving up and down in a sinusoidal manner within a dead-ended cylinder. As a result, liquid is squeezed out from the bottom part and then drawn back in providing a high flow rate between the piston and the wall of the vessel.

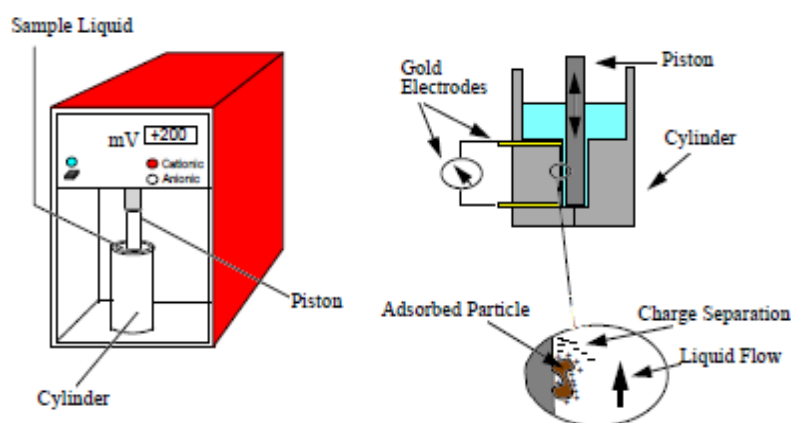


Figure 2-5 Streaming current device [41].

A streaming current is created as the flow separates the material attached to the wall or the piston from its counter-ions. This current is received by two electrodes which are located in the

sight of the cylindrical vessel. Then the current is converted to the corresponding potential and displayed on the instrument [41].

This device is used for determination of the endpoint of a titration. A solution of cationic or anionic polymers is gradually added to the sample and when the SC instrument displays "zero" it is assumed that sufficient amount of polymer solution has been added to the mixture to neutralize any excess of electrical charge [42].

2.6.3 APPLICATION IN PAPER INDUSTRY

Cationic demand indicates the existence of detrimental substances in process water [18]. Even though stickies are mainly anionic [43], cationic demand measurements must be complemented with other methods, for example deposition tests, in order to monitor their content in the system. This is due to a fact that not all stickies are anionic charged and not all anionic charged particles are sticky [20].

The monitoring of cationic demand in the system is important for achieving the optimal performance of polyelectrolytes such as retention aids, drainage aids, sizing agents, strength additives etc. [44].

3. METHODS

In Chapter 3 the methods used in the project and the aim of using them is presented.

3.2 DETERMINATION OF CONCENTRATION IN PULP SUSPENSION

The aim is to determine the concentration in pulp suspension which is further needed for analysis of macro-stickies and ash content measurements. The determination is performed according to TAPPI standard T240.

3.3 DETERMINATION OF ASH CONTENT IN PULP SUSPENSIONS

The aim is to determine ash content in pulp or paper. The ash in pulp consists of several different components. These can be mineral substances from the wood raw material, metallic residues from pipes and machinery parts of the process or residues from chemicals used in the pulping process. Often the ash content is related to the filler content in pulp. (e.g. calcium carbonate, clay). Ash content measurements on pulps coming out from the deinking line can to some extent indicate the efficiency of the line. Determination is performed according to TAPPI standard T413.

3.4 ANALYSIS OF MACRO-STICKIES IN PULP (HAND-SHEET METHOD)

The aim is to determine the amount of macro-Stickies in pulp. This is done in order to get an idea about the stickies-content in the pulp fed to the paper machines. Analysis is performed according to SCA standard ST0082 which is described below.

Equipment needed: Vacuum suction pump, filter flask, Buchner funnel, filter paper Munk tell 5 (11 cm), test paper Munk tell (22cm), heating plates and scales, Sommerville screen, 500 ml beaker, Buchner funnel (size larger), filter paper (15cm), bucket and a microscope.

Initially determine the pulp concentration according to TAPPI standard T240. The second step is to weigh up 10.0 grams of fully dried pulp (1000/concentration wet pulp in percent) in a 500 ml beaker. Next step is to prepare the Sommerville Screen equipment; put the sieve on the packing in the Sommerville screen and secure the side cover. Fasten the water hose to the spray nozzle and start the water flow.

Fill water until the nozzle is covered, pour in the sample and turn on the apparatus when the water level has reached the overflow. Sieve the pulp for 10 minutes. Turn off the apparatus and close the water inlet, pour out the water and rinse the wall surfaces so that all stickies are located on the sieve. Rinse the sieve and collect the rising water in a bucket. The next step is to filter the rinsing water by using a filter paper with 15 cm diameter. Stamp the filter paper with sticky side facing a test paper. Dry the paper sheet in the oven for 5 min at 105°C.

Finally count the amount of stickies and grade them by using a microscope. The Microscope should be set to 0.8 giving an enlargement of 7-10 times. Grade the stickies according to their size as follows: 0.1-0.3 mm = 1 point, 0.4-0,8mm = 4 points and >0.8= 9 points.

3.4.1 CALCULATION

Amount of macro-stickies is expressed in number of stickies per 100g of pulp (No/100g) and in points per 100g. The differentiation is made in order to obtain information about both number and size of stickies.

$$\text{Stickies (No/100g)} = \text{number of stickies} \times 10 \quad (5)$$

$$\text{Stickies(points/100g)} = \text{number of points} \times 10 \quad (6)$$

3.5 Determination of Cationic Demand (Charge measurements)

The aim is to determine the cationic demand in pulp and process water. This is performed in order to get an indication regarding the existence of detrimental substances in the process. The determination is performed according to SCAN-W 12:04.

3.6 UV-VIS MEASUREMENTS

The aim of the measurements is to determine the UV-VIS absorbance in the sample at 200 and 400nm wavelengths. This is done in order to distinguish unsaturated groups which are present in pitch, stickies and coating binders. See theory chapter 2.3.

Reagents and chemicals used are deionized water or distilled water and ethanol.

Equipment needed: Filtration equipment consisting of Buchner funnel, Munktell glass micro fiber disc (47mm), UV-VIS spectrophotometer, cuvette, 50 ml HDPE bottle and pipette.

First of all sample preparation is performed by filtration of the pulp or process water through the Munktell glass fiber filter (Ø 47 mm). Filtrate is collected in 50 ml HDPE bottle. Then instrument preparation is performed including cleaning the cuvette (both inside and outside) with ethanol (95%) by using cotton stick. Further cleaning is performed by rinsing the cuvette with distilled water.

5 ml of filtrate is pipetted into a graduated glass cylinder and diluted 3 times (the dilution is adjusted if needed). Then, cuvette is placed in the UV-VIS spectrophotometer (Spectroquant®Pharo 3000 M). When absorbance value appears on the display of the apparatus measurement is completed. After measuring the cuvette is rinsed with distilled water before using it again.

3.7 ADSORPTION AND DYEING OF STICKIES

The aim is to get an indication about the stickies content in the pulp and water system along the process by adsorption followed by subsequent dyeing. The method primarily detects micro-stickies.

Red dye is used for dyeing and equipment needed is: heating plate, stirring equipment consisting of Lab ball and impeller; 600 mL glass beaker and glass beaker for dyeing; 125 mL HDPE bottle.

First of all, install the equipment including heating plates and stirring system. Attach a lid of a 125mL bottle to the stirrer. The lid is marked with a black selection to facilitate the stirring adjustment. Assemble the stirrer into the Lab ball and mount the Lab ball to the stand. Then carefully cut off the bottom part of the 125 mL HDPE bottle and mount the bottle to the lid on the stirring equipment. Do not touch the surface of the HDPE bottle to avoid contaminations.

Next step is sample preparation. Add 400 mL of sample, process water or pulp into the 600 mL glass beaker. In case of high consistency pulps let the sample pass through a Büchner funnel and then adjust the consistency. After sample preparation, place the glass beaker onto the heating plate and lower the stirring equipment until half of the plastic bottle is immersed into the sample and start the stirring. Adjust the stirring speed to obtain appropriate mixing and an even level in the glass beaker. Heat the sample and then let it stir for 5 minutes at this temperature level. After 5 minutes lift up the stirring equipment and remove the HDPE bottle. Carefully rinse both the inside and outside of the HDPE bottle in cold water for approximately 1 minute to remove non sticky contaminants and fibers from surface of the bottle. After rinsing, the bottle is dried in oven for 5-6 minutes at 50°C to enable dyeing. During drying prepare the dye. Use a glass beaker suitable for the 125 mL HDPE bottle and fill it up to appropriate level to cover the bottle surface. When the bottle is dried immerse it into the dye during 15 seconds. Carefully rinse off excess dye with cold water. Finally, the bottle is dried a second time for 5-6 minutes at 50°C. To enable visual evaluation, place the bottle on a white surface and ensure appropriate lighting.

3.8. METHODS PERFORMED BY EXTERNAL COMPANIES

Dichloromethane (DCM) extractions are performed by Clariant and the method is claimed to determine the total stickies content. The DCM extraction method is based on TAPPI standard T 204. Colloidal collection tests are performed by BIM Kemi AB in order to determine amount of colloidal particles i.e. micro-stickies in the system. Description of this method is given below.

3.8.1 COLLOIDAL COLLECTION TEST (CCT)

The aim is to estimate micro-stickies in filtrates from pulp and process waters by measuring number of colloidal particles.

Equipment needed: filter (DFS): 40/0,22, two 1000 mL glass beakers which are used with the filter, three 150 mL glass beakers for heating the pulp suspension, heating plate with stirrer, counting chamber (hemocytometer) and cover glass, pipette to insert the filtrate into counting chamber.

The performance can be divided into two parts; sample preparation and image analysis. First of all determine the pulp concentration. Then dilute the pulp to 0,5 % and 2 L and place it in the disintegrator. When the pulp is disintegrated heat 100 ml to 40°C. While heating prepare the hemocytometer; clean the chamber and cover glass and wipe it off with lens paper. Wet the cover glass with water and place it on the hemocytometer and slide the cover glass over the counting chamber. After heating to 40°C filter the sample through the DFS-filter. Then 10 seconds after the filtrate has passed the filter, tilt the beaker 45° and pipette 20 µL from the middle of the beaker. Add the filtrate at the edge of the cover glass and let it be sucked into the counting chamber. Pipette another 20 µL to the second counting chamber. Then place the counting chamber in a microscope and analyze it by using Image Pro Plus. The particles are divided into four different classes and number of particles are counted and presented in a data collector. Class 1-3 includes colloidal particles and in class 4 fibers and larger (1,6-20 µm) particles can be found.

4 ANALYSIS OF HISTORICAL DATA

4.1 INTRODUCTION

Due to lack of time very few follow-ups of historical data regarding the stickies-related measurements performed at the laboratory are made by process engineers. Therefore and in order to get an introduction to SCA, the process and methods used for stickies measurements; analysis of historical data was performed. It was aimed for finding critical parameters affecting depositions. By doing this it was expected to obtain relevant information basis for drawing conclusions regarding the origin of the deposits and correlation to cleaning occasions.

4.2 METHOD

Analysis of the historical data has been performed based on the data available from the SCA database QIS, Plain Rapport (detailed stops – washing of wires and felts due to sticky deposits) and history of online measurements available from the process control software ABB.

There is a wide range of tissue products manufactured at SCA Edet Mill. Responsible engineers on the paper machines suspected that the cleaning sessions occurred more often while producing toilet paper compared to wet-strength grades. With the intention of finding correlations to cleaning sessions following data were observed in relevant periods of time:

- Stickies-related measurements performed at the SCA Edet laboratory; macro-stickies counting (hand-sheet method) and UV-VIS measurements (colloidal stickies).
- Detailed stop reports which provide information about cleaning occasions caused by sticky deposits on wires and felts.
- Addition of wet-strength chemical.

It has been reported in literature that primary stickies originate from the raw material [9], therefore analysis of washing occasions in correlation with the particular pulp dosage was performed for the period from January to December 2011 for PM7 and PM8. During this period the mill was experiencing problems with sticky deposits and also performed test runs with passivation chemicals supplied by two competitor companies. To see if the pulp dosage (recipe) had any influence on number of cleaning occasions pulp dosage, wet-strength dosage and cleaning occasions were plotted and observed month by month.

4.3 RESULTS

4.3.1 MEAN VALUES DAILY MEASUREMENTS

Available data of daily macro-stickies and UV-VIS measurements performed at SCA Edet laboratory has been acquired and mean values as well as maximum and minimum values have been calculated and summed-up in Table 4-1.

Table 4-1 Mean values from macro-stickies measurements during 2011. Mean values for UV-VIS measurements for C7 and C12 Jan 2012 and Jan-Feb 2012 for T1, T2, and T3.

Sampling point	Macro-stickies (No/100g)	Macro-stickies (Points/100g)	UV-VIS (Abs)
Pulp Tower 1	139,7 (10-670)	153,5 (10-670)	6,4 (3-8)
Pulp Tower 2	190 (10-870)	224 (10-980)	6,4 (3-8)
Pulp Tower 3	96 (30-250)	111,2 (10-470)	6,3 (3-8)
Pulp Chest 12			4,5 (3,4-5,3)
Pulp Chest 7			4,9 (3,8-6,8)

It can be seen from the table that amount of macro-stickies can vary rather much and that Tower 2 tends to exhibit highest amount of macro-stickies whilst Tower 3 have the lowest value. The UV-VIS values are in the same level for all towers whilst they are slightly lower for Chest 7 and 12.

4.3.2 MACRO-STICKIES VS CLEANING OCCASIONS

It was not possible to observe any clear relationships between macro-stickies measurements and production stops on the machine due to cleaning of wires and felts (see Figure 1-8 in Appendix B). However, the macro-stickies measurements can indicate if there are stickies in the system or not.

4.3.3 MICRO- STICKIES VS CLEANING OCCASIONS

No clear relationships between micro-stickies (UV-VIS) measurements and cleaning occasions (production stops) could be observed (see Appendix B Figure 9). Due to technical problems during the initial month of measuring, the reliability of the results during this period is questionable (the cuvette was damaged etc.).

4.3.4 RELATIONSHIP MACRO-MICRO

No clear relationships neither between macro-stickies measurements and UV-VIS measurements on pulp towers nor macro-stickies measurements on the pulp towers and UV-VIS measurements on dilution water (white water to towers) could be observed.

4.3.5 OCCURRENCE OF CLEANING OCCASIONS: TOILET VS WET-STRONG PAPER

As mentioned the influence of wet-strength additives was observed. On PM7 mostly toilet paper is produced and therefore results for this paper machine will not be presented.

In Figure 4-1 and 4-2 historical data from PM8 is presented. After treating historical data for PM8 it could be seen that most of the cleaning occasions occurred during production of toilet paper; 74% during 2010 and 81% during 2011. It could also be seen in the figures that the total number of cleanings increased from 43 during 2010 to 95 during 2011.

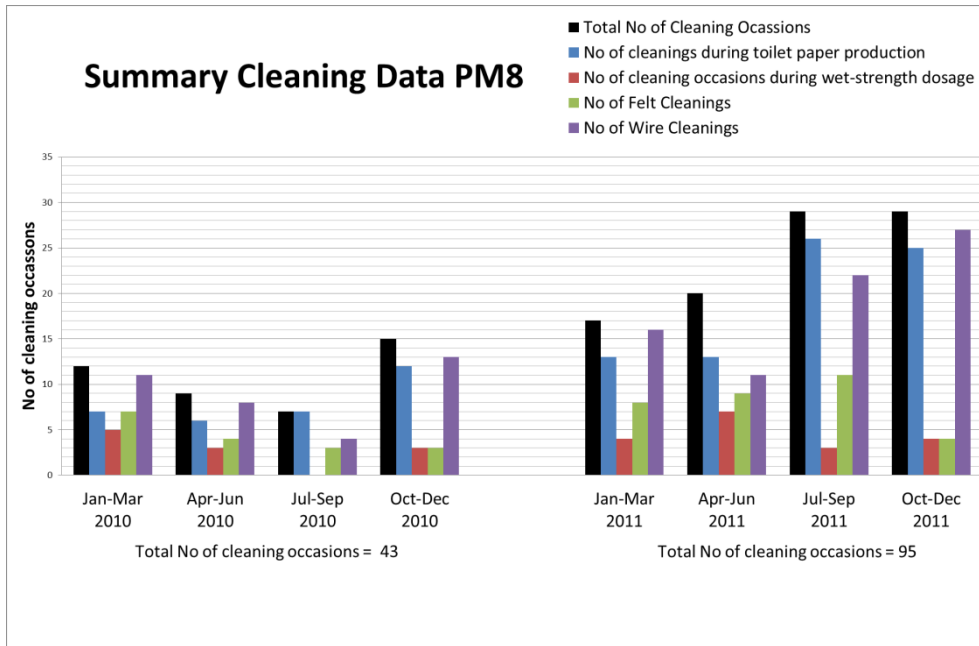


Figure 4-1 Summary of cleaning data from PM8. The diagram shows total number of cleaning occasions, number of cleaning occasions during toilet paper production, number of cleaning occasions during production of wet-strength grades, number of felt cleanings and number of wire cleanings.

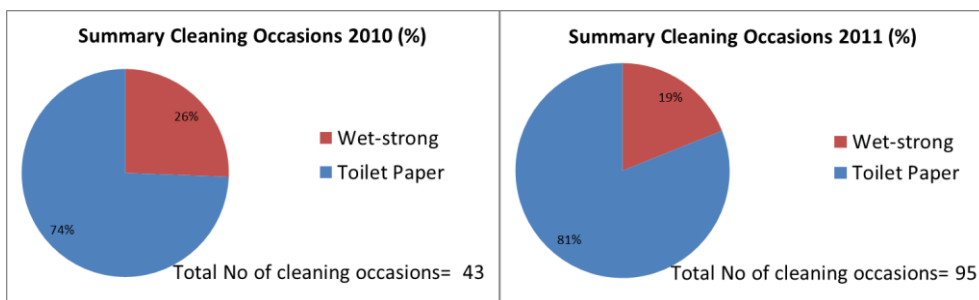


Figure 4-2 Summary of cleaning occasions in % on PM8 2010 and 2011. During 2010 74% and during 2011 81% of the cleaning occasions occurred during wet-strong paper production.

4.3.6 INFLUENCE OF PULP DOSAGE

When observing the influence of pulp dosage (%) on paper machine 8, PM8 (see Appendix C) the cleaning occasions and wet-strength dosage were plotted over the pulp dosage month by month. In order to examine how the individual pulp affected the washing occasions a summary of the pulp dosage is presented. Analysis of the influence of pulp dosage on the frequency of cleaning occasions for PM8 is given in Tables 4-2, 4-3 and Figures 4-3, 4-4. Similarly the results for paper machine 7 (PM7) are presented in Tables 4-4, 4-5 and Figures 4-5, 4-6.

SUMMARY PM8

First of all total number of rolls on respective machine was calculated, see last row in Table 4-2 and 4-3 for PM8. Then it was calculated in how many of these rolls respective pulp was included. For example Chest 5 was included in 5197 rolls on PM8 Jan-Jun 2011, see Table 4-2.

Table 4-2 Summary of results from PM8 January-June 2011.

January-June 2011 PM8	Chest 5	Chest 6	Tower 1	Tower 2	Tower 3	Chest 7	Chest 12
No of rolls/pulp	5197	2804	5869	3867	2526	2238	4508
No of cleanings/pulp	13	9	18	25	20	17	13
No of cleanings per pulp/No of rolls per pulp	0,25%	0,32%	0,31%	0,65%	0,79%	0,76%	0,29%
No of rolls per pulp/Total No of rolls	61%	33%	69%	46%	30%	26%	53%
No of cleanings per pulp/Total No of cleanings	35%	24%	49%	68%	54%	46%	35%
Total No of cleanings=	37						
Total No of rolls=	8468						

To get an overview of how many percent of the total rolls each pulp was included in, number of rolls per pulp was divided by total number of rolls, see top left pie diagram in Figure 4-3. It can be seen from the diagram in the top left, January-June 2011 PM8, is that pulp from Tower 1 is included in 69% of the total No rolls, Chest 5 61%, chest 12 53%, Tower 2 46%, Chest 6 33%, Tower 3 30%, and Chest 7 26%.

In the top right pie diagram number of cleanings per pulp is divided by total number of cleanings to get an overview in how many percent of the total cleanings each individual pulp is involved in. It can be seen that pulp from Tower 2 was included in 68% of the cleaning occasions, Tower 3 54%, Tower 1 49%, Chest 7 46%, Chest 12 35%, Chest 5 35% and Chest 6 24%.

In the bottom pie diagram number of cleanings per pulp was divided by number of rolls per pulp to see how many percent of the rolls of each pulp that contributes to a cleaning occasion. It can be seen that for all rolls from Tower 3 0,79% of them contributed to a cleaning occasions. For pulp Chest 7 0,76%, Tower 2 0,65%, Tower 1 0,31%, Chest 6 0,32%, Chest 12 0,29% and Chest 5 0,25%.

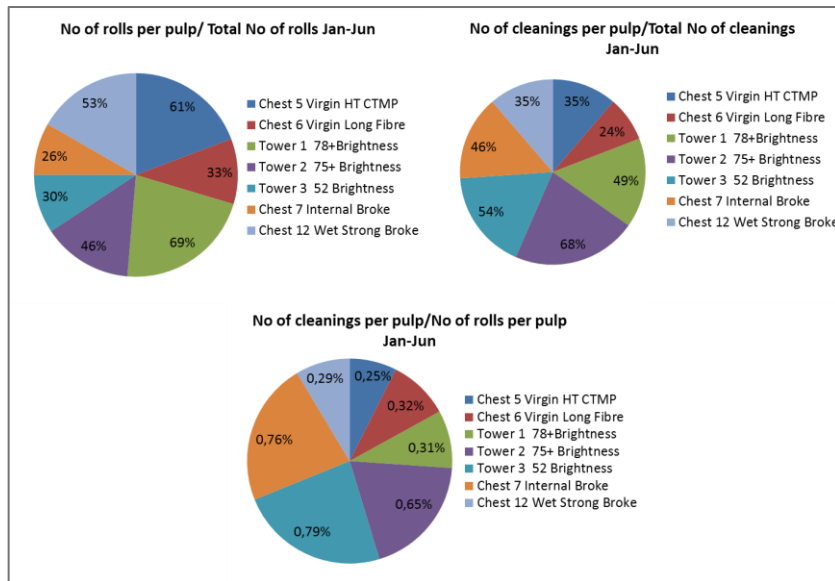


Figure 4-3 Pie Diagram showing a summary of the results from PM8 January-June 2011.

The same procedure was used for PM8 July-December. What can be seen in Table 4-3 is that total number of rolls was almost the same whilst total number of cleanings increased from 37 to 58. Even though total number of rolls increased by 5% for Pulp Chest 5 and 12 number of cleaning occasions decreased by 3 and 5 respectively. Number of rolls including Pulp Chest 6 decreased by one percent and also number of cleanings decreased by 2. Number or rolls including Pulp Chest 7 decreased substantially by 22% and also number of cleaning occasions decreased by 10. Total number of rolls for pulp Tower 1 and 3 increased by 5% and 3% respectively whilst Tower 2 decreased by 1 %. Remarkably, number of cleanings increased by 15, 23, and 20 respectively.

Further it can be seen in Figure 4-3 top right that the pulp from Tower 1,2 and 3 (from DIP1 and DIP2) have a large influence on number of cleaning occasions. Tower 1 was included in 33 cleaning occasions but it was also included in a remarkably high number rolls. When considering number of cleanings for each pulp with respect to number of rolls per pulp, Tower 2, Tower 3 and Pulp Chest 7 have the largest influence (see bottom diagram in Figure 4-4)

Table 4-3 Summary of results from PM8 July - December 2011.

July-December 2011 PM8	Chest 5	Chest 6	Tower 1	Tower 2	Tower 3	Chest 7	Chest 12
No of rolls/pulp	5673	2476	6286	3834	2824	369	4999
No of cleanings/pulp	10	7	33	45	43	7	8
No of cleanings per pulp/No of rolls per pulp	0,18%	0,28%	0,52%	1,17%	1,52%	1,90%	0,16%
No of rolls per pulp/Total No of rolls	67%	29%	74%	45%	33%	4%	59%
No of cleanings per pulp/Total No of cleanings	17%	12%	57%	78%	74%	12%	14%
Total No of cleanings=	58						
Total No of rolls=	8469						

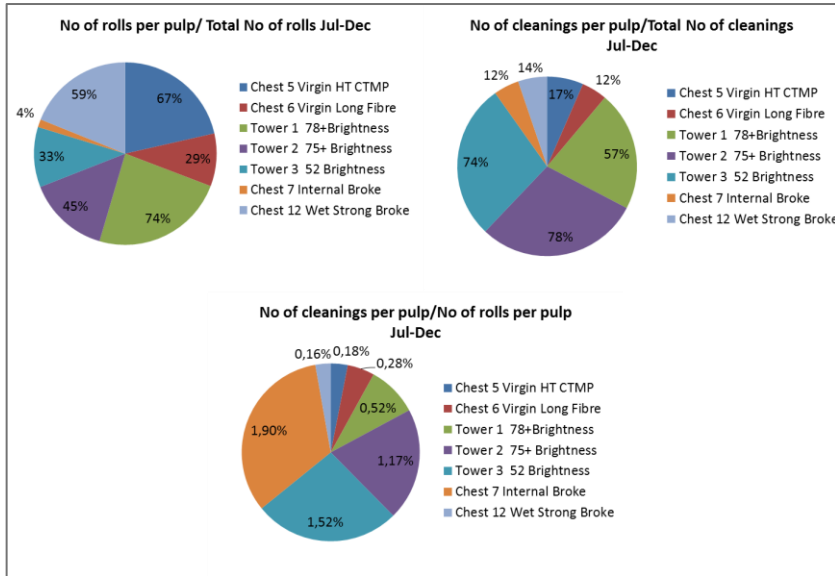


Figure 4-4 Pie diagram showing the summary of the results from PM8 July - December 2011.

SUMMARY PM7

As mentioned the same procedure was performed for PM7, Jan-Jun and Jul-Dec. What can be seen for PM 7 is that even though number of rolls decreased from 7834 during Jan-Jun to 7530 during Jul-Dec number of cleanings increased from 33 to 49. It can be observed for both periods in top right pie diagram in Fig 4-5 and 4-6 that pulp Tower 1,2,3 and Pulp Chest 7 are again included in a large part of the cleaning occasions. When observing the pie diagram at the bottom (no of cleanings per pulp/no of rolls per pulp) it can be seen that the distributions between the different sections are larger than for PM8, especially regarding period Jul-Dec.

Table 4-4 Summary of results from PM7 January-June 2011.

January-June 2011 PM7	Chest 4	Chest 6	Tower 1	Tower 2	Tower 3	Chest 7
No of rolls/pulp	2121	2045	2449	4876	3483	2450
No of cleanings/pulp	7	6	21	27	15	13
No of cleanings per pulp/No of rolls per pulp	0,33%	0,29%	0,86%	0,55%	0,43%	0,53%
No of rolls per pulp/Total No of rolls	27%	26%	31%	62%	44%	31%
No of cleanings per pulp/Total No of cleanings	21%	18%	64%	82%	45%	39%
Total No of cleanings=	33					
Total No of rolls=	7834					

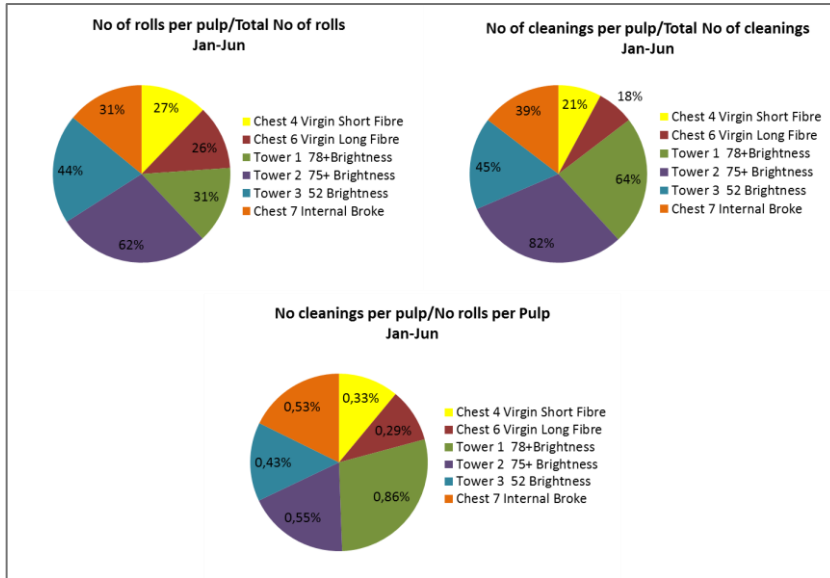


Figure 4-5 Pie diagram showing the summary of the results from PM7 January - June 2011.

Table 4-5 2011 Summary of results from PM7 July - December 2011.

July-December 2011 PM7	Chest 4	Chest 6	Tower 1	Tower 2	Tower 3	Chest 7
No of rolls/pulp	1815	1711	2836	4872	4016	2148
No of cleanings/pulp	7	8	23	31	28	12
No of cleanings per pulp/No of rolls per pulp	0,39%	0,47%	0,81%	0,64%	0,70%	0,56%
No of rolls per pulp/Total No of rolls	24%	23%	38%	65%	53%	29%
No of cleanings per pulp/Total No of cleanings	14%	16%	47%	63%	57%	24%
Total No of cleanings=	49					
Total No of rolls=	7530					

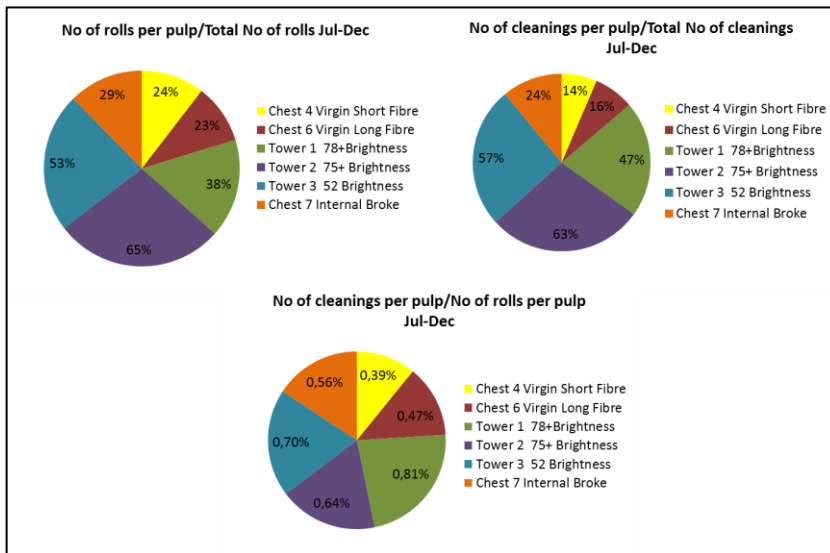


Figure 4-6 Pie diagram showing the summary of the results from PM7 July - December 2011.

More cleanings were performed on PM7 when running virgin fiber in relation to number of rolls including virgin fiber compared to PM8. It is however hard to conclude which pulp has the largest influence on PM7 with regard to cleaning occasions.

DISCUSSION/CONCLUSION PULP DOSAGE

Overall it can be seen that pulp Tower 1,2,3 and pulp Chest 7 influence the number of cleaning occasions on both machines. This is not surprising since they are all internal pulps which originate from recycled fibers. Chest 4, 5, and 6 are all virgin pulps and should be clean and further not contribute to cleaning occasions. Results obtained from PM8 indicated that Tower 2, Tower 3 and pulp Chest 7 have the largest effect on occurrence of cleaning occasions. As mentioned in previous chapters Tower 2 is coming from same deinking line as the pulp in Tower 1 (DIP1) but has in general lower quality, Tower 3 comes from DIP2 and has much lower quality and brightness compared to pulps from DIP1. Tower 3 originates from old newsprint which should include less stickies than mixed office waste due to lower amount of contaminations such as glues. However, since DIP2 consists of fewer separation steps than DIP1 the pulp has a lower quality.

The results obtained from PM7 indicated that more cleaning occasions occurred while using virgin fiber in comparison to PM8. These can perhaps be explained by the fact that there are high quality demands on certain qualities including for example Chest 4 (virgin short fiber) and Chest 6 (virgin long fiber) and due to this machine operators clean preventively. This gives a broader distribution in the bottom pie diagram than on PM8 which makes it hard to conclude anything about the pulp influence but still the top right pie diagram indicates that the internal pulps are causing the main problems.

4.5 CONCLUSION HISTORICAL DATA

Main conclusions from the analysis of historical data are that most of the cleaning occasions occur during production of toilet paper and less during production of wet-strong paper grades. This can be due to improved retention which is achieved as a result of addition of wet-strong agent. The other reason could be that the quality of the pulp mixture when running wet-strong product is higher (primary pulp from Pulp Chests 5, 6 and bleached office waste from Pulp Towers 1,2) compared to when running the toilet paper which often includes internal broke from Pulp Chest 7 and unbleached newsprint from Pulp Tower 3.

The connection between pulp dosage and cleaning occasions could be seen more clearly for paper machine 8 than for paper machine 7.

It was hard to observe clear trends between cleaning occasions and stickies measurements performed at the laboratory at SCA. The assumption that can be made so far is that cleaning occasions most probably depend both on the pulp mixture fed to the paper machines and if wet-strength additives are used.

5 PROCESS SURVEY 1: VARIATION IN PROCESS PARAMETERS

5.1 INTRODUCTION

The aim of Process Survey 1 was to perform mapping of the tissue manufacturing process at SCA Edet mill, obtain information of the variance in process parameters and correlations between different parameters as well as their impact on cleaning occasions due to formation of sticky deposits on wires and felts.

5.2 DESCRIPTION (METHOD)

During the first set of experiments measurements of pH, temperature, cationic demand and UV-VIS absorbance were performed for 3 weeks. Samples were taken at different process locations 5-8 times a day with one hour interval.

Table 5-1 Sampling points during process survey 1

Sampling Points
Pulp Tower 1
Pulp Tower 2
Pulp Tower 3
Wire Pit PM7
Wire Pit PM8
White Water (Blekt BV)

These particular sampling points were chosen based on information mentioned in literature, where the white water, used for dilution in different process stages, is one of the potential sources of micro and secondary stickies [31]. Samples from pulp towers, in turn, provide information about the pulp quality which is fed into the paper machine.

In order to obtain a robust picture of the process, it would be favorable to add more sampling points before and after different steps in deinking plants. However, due to time restrictions number of sampling points has been limited to the most important ones (process water and pulp from the three pulp towers).

5.3 RESULTS

By plotting the obtained data in Microsoft Excel and the statistical software Minitab it has been observed that there is rather significant variation in process parameters, especially temperature of the white water, UV-VIS absorption and cationic demand values. Figures from Minitab (variables charts for individuals) can be found in Appendix D.

Pulp dosage, addition of wet-strength additive and CMC plotted in the same diagram as measured parameters for Wire Pit PM 7 and PM8 are presented in Figure 5-1 and 5-2.

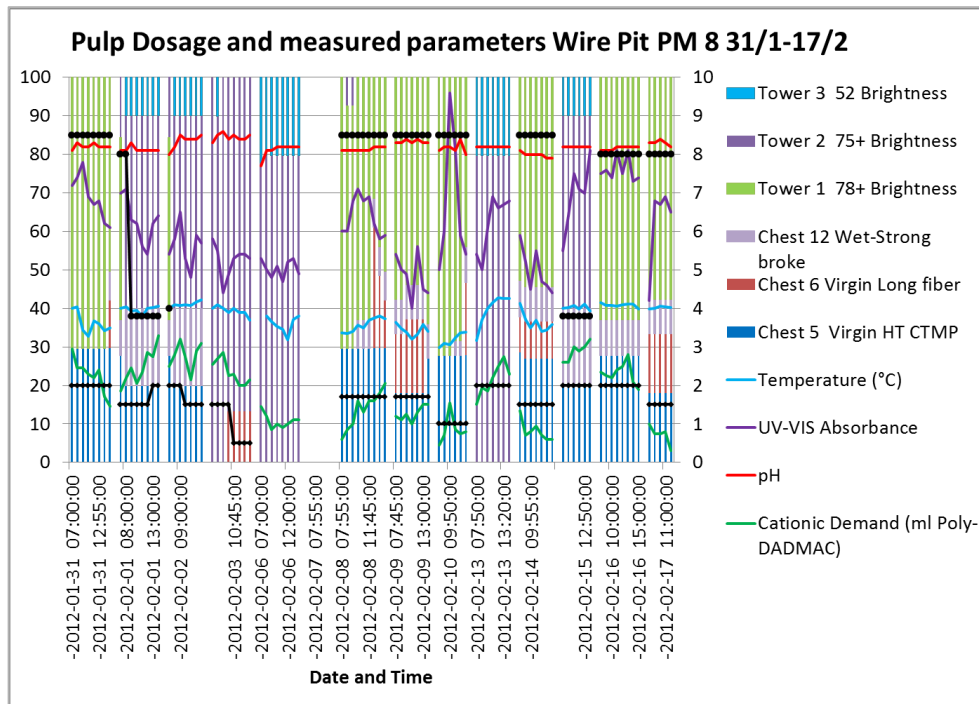


Figure 5-1 Summary of the pulp dosage, dosage of wet-strength additive, CMC and measured parameters. On the left-hand axis pulp dosage of individual pulps expressed in percentage of total pulp dosage and temperature are plotted. On the right-hand axis pH, UV-VIS absorbance, cationic demand, dosage of wet-strength additive and dry-strength additive (CMC) are plotted. Note that the cationic demand is expressed in mL of poly-DADMAC instead of $\mu\text{eq/L}$.

In figure 5-1 it can be seen that cationic demand follows the CMC dosage which is due to anionic charge of CMC. In general cationic demand is lower in Wire Pit on PM7 compared to PM8 which can be explained by the fact that CMC is added only on PM8. In Fig 5-2 it can be seen that the UV-VIS values are lower while running virgin pulps (Chest 4 and 6) on PM7.

An overall trend is also that cationic demand follows UV-VIS absorbance values to some extent. Most probably this trend can be seen since both methods measure small particles in the same size range (they are both measured on filtrate from glass fiber filters). It has also been observed that the wet-strength additive contributes to lower cationic demand since it is positively charged.

Thus, it can be seen that cationic demand is influenced by addition of ionic polymers and therefore cannot be treated as a direct indicator for overall cleanliness of the process.

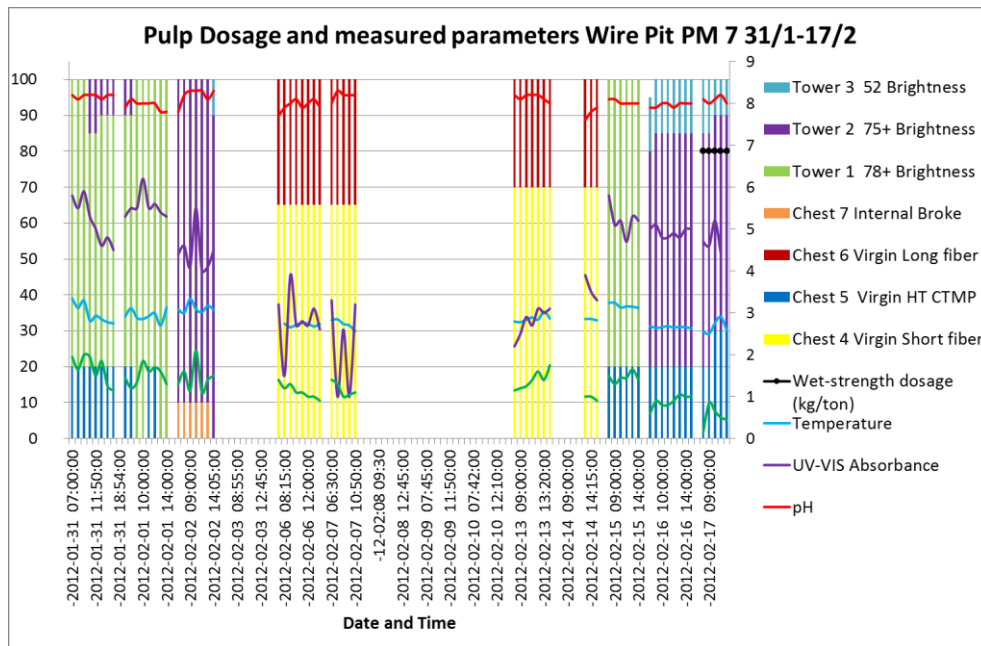


Figure 5-2 Summary of the pulp dosage, dosage of wet-strength additive, CMC and measured parameters. On the left-hand axis pulp dosage of individual pulps expressed in percentage of total pulp dosage and temperature is plotted. On the right-hand axis pH, UV-VIS absorbance, cationic demand and dosage of wet-strength additive are plotted. Note that the cationic demand is expressed in mL of poly-DADMAC instead of $\mu\text{eq/L}$.

5.3.1 WHITE WATER (DILUTION WATER FOR PULP TOWERS)

Observed temperature interval for the white water during the measurement period has been rather wide – from 8,7°C up to 39,3°(see Fig 1 in Appendix D). UV-VIS values varied from 1,4 to 5,9 and cationic demand values were in range from -25 $\mu\text{eq/L}$ to -200 $\mu\text{eq/L}$. pH values were rather stable within one day, i.e. they varied by 0,1-0,3 units. However, more significant pH variations were noticed between days, i.e. from 7,3 to 8,1.

5.3.2 PULP TOWER 1

Temperature variance in Pulp Tower 1 was also rather substantial, i.e. from 24,8°C to 44°C. UV-VIS values varied from 3,3 to 8,9 and cationic demand values – from -25 $\mu\text{eq/L}$ to -300 $\mu\text{eq/L}$. Within one day pH varied by 0-0,4 units, but difference between days was slightly higher, i.e. pH interval was from 6,9 to 7,5 (see Figure4 in Appendix D).

5.3.3 PULP TOWER 2

Observed temperature interval was from 17,7°C to 43°C, UV-VIS absorbance values varied from 4 to 9 and cationic demand – from -10 $\mu\text{eq/L}$ to -250 $\mu\text{eq/L}$. pH variations during the measurement period were substantial, i.e. interval was from 6,9 to 8,3. The pH deviations were

significant even within one day, i.e. difference was from 0,1 up to 1,3 units (see Figure 5 in Appendix D).

5.3.4 PULP TOWER 3

Observed temperature interval was 25,7°C – 39,5°C, UV-VIS values varied from 2,7 to 9,1. It was observed that the cationic demand values during the three-week measurement period have been rather stable – they varied from -20 µeq/L to -80 µeq/L, while most of the values were around -30 µeq/L. pH values within one day were mostly stable, however during some days they could vary by 0,4-0,8. pH interval during the whole measurement period was from 7,3 to 8,1 (see Figure 6 in Appendix D).

5.3.5 WIRE PIT PM7

Temperature interval was from 29,2°C to 39°C, UV-VIS values varied from 1 to 6,2 and cationic demand values – from -15 µeq/L to -210 µeq/L. It was observed that pH varied by 0,1 to 0,5 units within one day and interval during the three-week measurement period was from 7,6 to 8,3 (see Figure 2 in Appendix D).

5.3.6 WIRE PIT PM8

The observed temperature interval was from 29,8 to 42,8°C, UV-VIS values varied from 1 to 9,6 and cationic demand values – from -30 µeq/L to -320 µeq/L. pH interval during the measurement period was from 7,7 to 8,6, whereas within one day differed by 0,1-0,5 units.

5.4 DISCUSSION

Based on the variables charts for individuals obtained using statistical software Minitab it can be seen that there are large variations in the measured parameters. It is visualized in the charts as a rather high amount of values below and above the lower and upper control limit respectively. Unfortunately these variations cannot be linked to the cleaning occasions since during the three-week period of measuring there was only one cleaning performed for each paper machine. This, in turn, does not provide necessary information to draw any objective conclusions.

The temperature variance in the white water can be explained by addition of fresh water which also influences the values of UV-VIS absorbance and cationic demand. Similarly, temperature in the pulp towers varies according to addition of the fresh water in the white water tank since this water is used for dilution of the pulp in these towers.

It has been observed that Tower 1, 2 and 3 exhibited similar average pH values (7,2-7,3), however Tower 2 showed significantly higher pH variations both within one day as well as during the whole measurement period. Another trend is that pH was higher in Wire Pit PM7 and PM8 compared to pulp towers and white water. In general, it can be seen that the process pH varies rather substantially both within one day as well as between the days. It is however hard to conclude whether these variations are significant in terms of deposit formation. This is due to lack of information regarding the critical pH range which, in turn, also is mill-specific [23].

It can be noted that different information sources state opinions that often are not in agreement with each other, also the chemical companies involved in the project have shared different opinions. However it is agreed that pH is an important parameter that should be controlled. This is for example due to pH effect on dissolution of calcium carbonate discussed in Chapter 2.1.4. Therefore, it can be of great interest for SCA Edet Mill to properly examine this phenomenon, for example initiate a new Master thesis with the main focus on this area.

As mentioned above, on one hand high variation of UV-VIS values during the measurements performed the same day can be explained by the variations in the amount of fresh water added to the system. At the moment of performing the survey UV-VIS absorbance method was one of the daily measurements performed at SCA Edet laboratory for the monitoring of the colloidal stickies. Since the variation in UV-VIS absorbance values obtained within one day is rather high the single value is perhaps not representative for the whole day.

Highest UV-VIS absorbance and cationic demand values have been obtained from the Wire Pit PM8. Cationic demand and UV-VIS values lay rather high at Wire Pit PM7, Pulp Tower 1 and 2 as well. It should be noted that the cationic demand for the Pulp Tower 3 lays rather low ($\bar{x}=0,34$) and stable while the UV-VIS absorbance is rather high ($\bar{x}=7,21$).

As mentioned earlier Tower 1 and 2 originate from DIP1 and include bleached office waste whilst Tower 3 originates from DIP2 and includes unbleached newsprint. The difference between the deinking lines can be seen in Appendix A Fig 1-2. Whether the origin of pulp and the design of the deinking system affect the cationic demand and how is not known at the moment and further investigations are needed.

These differences between pulp Tower 1, 2 and Tower 3 may cause the disturbances in colloidal equilibrium when these pulps are mixed together which in turn may result in formation of secondary stickies and consequentially – deposition.

Since both UV-VIS and cationic demand determine the colloidal and dissolved substances in the samples, it was suspected that results obtained by these two methods should correlate with each other.

Cationic demand of the system depends on the dosage of CMC and wet-strength additive due to their anionic and cationic charge respectively. However, data obtained during this survey does not provide any information about synergy of papermaking additives on contamination of the system.

5.5 CONCLUSIONS

Methods used for the determination of stickies in the process did not provide valuable information regarding stickies in the process. Since no cleanings were performed on the machines during the period no correlations could be observed between process variations and cleaning occasions.

Main conclusion obtained by this process survey was that there are substantial variations in process parameters during the measurements performed at the same day. This in turn, may promote the destabilization of the colloidal balance leading to formation of secondary stickies. The reliability of the UV-VIS absorbance measurements as a daily method has been questioned due to high variability of the values obtained during the same day.

Process parameters are dependent on the pulp quality fed into the paper machine which is in correlation with results obtained from the analysis of historical data. In contrary to pulp Tower 1 and 2, pulp Tower 3 exhibited rather low and stable cationic demand which may result in formation of secondary stickies when pulp from Tower 1 or 2 is mixed with the one from Tower 3.

In order to obtain more information about stickies in the process other methods than the ones used in Process Survey 1 must be employed.

6 PROCESS SURVEY 2: STICKIES ADSORPTION WITH SUBSEQUENT DYEING

6.1 INTRODUCTION

During the analysis of historical data no correlations between available stickies measurements and cleaning occasions could be seen, therefore new possible methods were examined. One of the stickies determination methods mentioned in literature is adsorption of the sticky particles on HDPE bottle and subsequent dyeing for the visual evaluation [9]. The conditions of the method are however not clearly described in literature.

During 29th of February and 1st of March, representatives from Finnish company Banmark Oy visited SCA and performed stickies analysis using the adsorption and dyeing method which has been elaborated at this company. Simultaneously with the stickies trial cationic demand measurements were performed. The aim of the process survey was to test a new method and to get an idea about the stickies content in the process.

6.2 METHOD

The principle of the method is agitation of the pulp sample at elevated temperature. At this temperature tack of the stickies increases thus promoting their adhesion to the surface of the plastic bottle. Sticky particles are brought into contact with the plastic bottle by turbulence. It is assumed that the method determines sticky particles in micro range (however larger than colloidal particles). Plastic bottle is then immersed into the ink which selectively dyes sticky particles and the color of the bottle is not changed. The amount of stickies is then evaluated visually. Thus, this method does not provide any quantitative value of the stickies content in the samples, it only indicates if stickies are present or not (more detailed description of the method is given in Chapter 3.6). In order to get an overview of stickies content in the process sixteen sample points were chosen in the deinking line (DIP1) and papermaking process as follows (see flow-sheets Appendix A):

Table 6-1 Sampling points process survey 2

Sampling Points	
In flotation	Headbox PM8 Middle
Out Washing	Headbox PM8 Yankee
Out Screw press 2	Headbox PM8 Hood
Water loop 2	Wire Pit PM8
Pulp Tower 1	Clear Filtrate PM8
Pulp Tower 2	Pulp Chest 7
Headbox PM7	Pulp Chest 12
Wire Pit PM7	Clear Filtrate RP1
Clear Filtrate PM7	

6.3 RESULTS AND DISCUSSION

Results from the first experimental round are summarized in Table 6-2. Observations in the last column of the table indicate the level of stickies in samples at different process locations.

Table 6-2 Results obtained Day 1.

No	Sampling points Day 1	Temp (°C)	pH	Cationic demand (µeq/l)	Adsorption and Dyeing (Observations)
1	In flotation 1	50,4	7,5	-549	No stickies
2	Out from cleaning stage	39,3	7,4	-487	High
3	Out from screw press 2	35	8,1	-529	High
4	Water from loop 2	64,1	7,9	-679	Low
5	Tower 1	42,1	6,6	-230	Low
13	Pulp Chest 7	*	*	*	*
14	Pulp Chest 12	38,9	6,9	-252	High
7	Head box PM7	39,1	7,4	-146	No stickies
8	Wire pit PM7	34,3	7,6	-143	Very low
9	Clear filtrate PM 7	37,8	7,4	-133	No stickies
10	Head box PM8 Middle layer	39,6	7,4	-169	High
11	Wire pit PM8	41,1	7,6	-162	High
12	Clear filtrate PM 8	39,3	7,8	-293	Very low
15	Head box PM8 Hood layer	40,3	7,3	-156	Low
16	Head box PM8 Yankee layer	40,3	7,2	-161	Medium

Deinking line
 Pulps
 PM7
 PM8

In Table 6-2 and Figure 6-1 it can be observed that temperature at different process locations varied from 34,3°C to 64,1°C. pH along the process varied between 6,6-7,9 (pH in the screw-press sample is probably slightly higher than actual value due to dilution with tap-water). Variation of the cationic demand was rather substantial i.e. from -161 µeq/L in Headbox PM8 Yankee layer down to -679 µeq/L in the water from loop 2.

Further, in Table 6-2 it can be seen that samples 2-3, 10-11 and 14 exhibited high amount of stickies, whereas in samples 4-5, 12 and 15 it has been evaluated as low. Sample 8, 12 and 16 showed very low content of stickies. No stickies have been observed in samples 1, 7 and 9 which correspond to sampling points In flotation 1, Head box PM7 and Head box PM8 Middle layer respectively. Even though the pulp out from screw press 2 exhibited high amount of stickies, surprisingly almost no stickies could be determined in Tower 1.

It was unexpected that sample 1 In flotation 1, which should be the most contaminated pulp in the mill, did not indicate any sticky content whilst the pulp from sample 3 out from screw press 2 did. Since screw press 2 is the last stage in DIP1 it should be the pulp with the lowest level of contaminants. Though, it exhibited rather high stickies content and the dilution water was suspected to be a source of stickies and an additional sample was taken during day 2 from the clear filtrate on DIP1.

Results obtained during Day 2 (see Figure 6-3) indicated that sample 3 out from screw press 2, 10 Head box Middle layer PM8, 11 Wire pit PM8 still exhibited quite high amount of stickies. Sample 2 out from the cleaning stage and sample 14 from Chest 12 still contained stickies but a lower level compared to first round of trials. The additional sample from the dilution water on DIP1 (Clear filtrate DIP1) only indicated traces of stickies and therefore it is probably not the

reason for the high amount of stickies in the pulp coming out from washing during Day 1 and out of screw press 2 during Day 1 and 2.

Table 6-3. Results obtained Day 2.

No	Sampling points Day 2	Temp (°C)	pH	Cationic demand (µeq/l)	UV-VIS (Abs)	Macro-stickies (No/100g)	Macro-stickies (points/100g)	Adsorption and Dyeing (Observations)
1	In flotation 1	51,3	7,3	-478	12,8	*	*	Very low
2	Out from cleaning stage	48,5	7,3	-404	*	*	*	Very low
3	Out from screw press 2	56	8	-466	4,1	*	*	High
4	Water from loop 2	64,2	7,6	-626	*	*	*	No stickies
5	Tower 1	48,2	6,7	-136	10,9	290	390	Low/Medium
13	Pulp Chest 7	41,9	6,5	-99	*	150	350	Low
14	Pulp Chest 12	42	6,7	-115	6,5	*	*	Medium
7	Headbox PM7	35,8	7,6	-182	*	*	*	No stickies
8	Wire pit PM7	32,6	7,6	-183	*	*	*	No stickies
9	Clear filtrate PM 7	34,7	7,6	-176	*	*	*	No stickies
10	Headbox PM8 Middle	42,5	7,6	-146	*	*	*	Low
11	Wire pit PM8	43,4	7,7	-136	9,9	*	*	High
12	Clear filtrate PM 8	41,2	7,8	-321	8,5	*	*	No stickies
15	Headbox PM8 Hood	40,3	7,3	-156	*	*	*	Low
16	Headbox PM8 Yankee	40,3	7,2	-161	9,5	*	*	Medium
17	Clear filtrate RP1	*	7,2	-394	*	*	*	Very low

Deinking line
 Pulps
 PM7
 PM8
 White Water

One explanation to the lower sticky content in the pulp in to flotation 1 could be higher filler content in the beginning of the deinking line. The fillers may passivate the sticky particles and therefore the adsorption and dyeing method shows a low sticky content for sample 1. Later on in the process the fillers are removed and perhaps do not contribute to sticky passivation any longer. In order to check this theory ash content has been determined for sample 1 and 2 (see Table 6-4).

Table 6-4 Additional ash content results from two different sample points in deinking line 1. Two different samples from the pulp in to flotation 1 and out from the first washing step.

Sampling point	Ash content 1 (%)	Ash content 2 (%)
In flotation	20,2	19,1
Out wash	5,3	5,7

It can be seen that ash content before the first flotation step was considerably higher compared to the pulp from the cleaning stage which might serve as a confirmation for the theory mentioned above. In the report Functional Promoters for Stickies (Tissue world magazine 2010) they discuss the influence of ash content on stickies. Process charge and ash content can play a role in the degree of stickies deposition throughout the machine. The ash can act as nuclei onto which the hydrophobic micro-stickies particles tend to adsorb, increasing in size and eventually forming a sticky mass [45]. Which in this case contradicts the theory mentioned above.

A second possible explanation could be that the stickies fed into the system with raw material are mainly in a macro range. While screening in a fine screen, largest part of the macro-stickies are left in rejects, however part of the macro-stickies might split into smaller particles thus contributing to the high amount of micro-stickies. Since particles removed in washing step are smaller than 30 μm and micro-stickies are defined as particles smaller than 150 or 100 μm , micro-stickies larger than 30 μm are retained and thus could contribute to the high amount of stickies out washing step determined by adsorption and dyeing method.

Finally contaminants before the first flotation step might be potential stickies which are not tacky before they experience some kind of shock in process parameters. Therefore, they do not tack to the plastic bottle and thus are not determined by adsorption and dyeing method.

Further temperature, pH and cationic demand during both days are presented in Figure 6-1. It can be seen that water from loop 2 exhibited highest temperature, pH and cationic demand. Cationic demand was lowest in Tower 1, Chest 7 and Chest 12. Measured parameters were rather stable in PM7 and PM8 system.

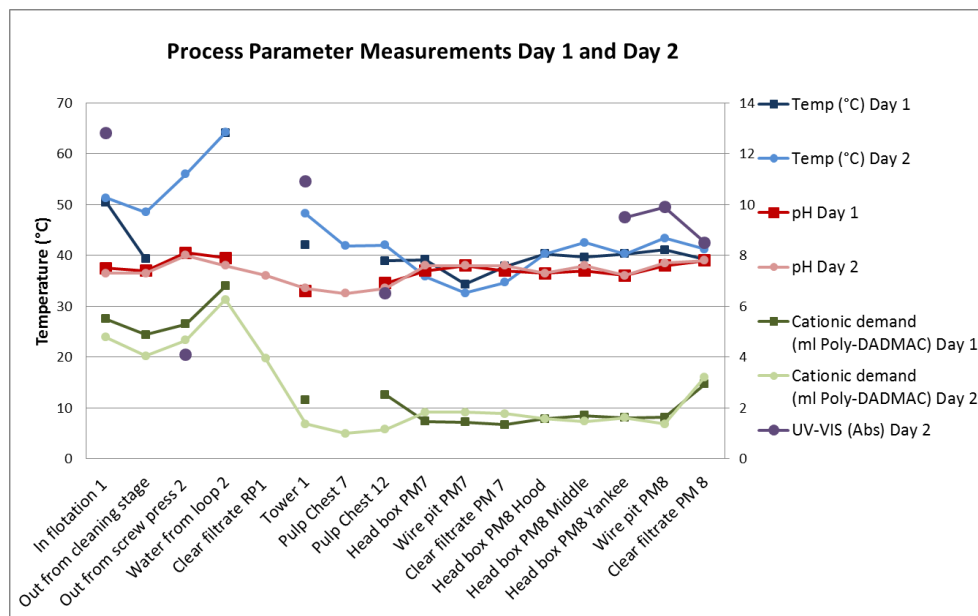


Figure 6-1 Diagram showing variation in process parameters during day 1 and 2. Temperature is presented on the left hand axis whilst pH, cationic demand and UV-VIS absorbance values are presented on the right hand axis

By comparing results obtained during two days of trials it could be seen that temperature, pH and cationic demand did not vary substantially from Day 1 to Day 2. Slightly higher temperature was observed during Day 2. Cationic demand in DIP1 was higher during Day 1 whereas PM7 system exhibited higher values during Day 2. No significant differences in pH could be observed during Day 2 compared to Day 1.

After performing second round of trials seven samples were chosen for UV-VIS measurements. The aim was to see whether correlations between adsorption and dyeing method and UV-VIS measurements could be observed. Therefore, the samples that showed highest, lowest and medium level were chosen (see Table 6-2). It has been observed that UV-VIS values decrease

along the deinking line¹. However, value in Tower 1 and PM8 system was relatively high. However, no correlations could be seen between the two methods.

Daily measurements of macro-stickies using hand sheet method which are performed by the laboratory staff at SCA Edet have been compared to the results obtained from the experiments using adsorption and dyeing method and UV-VIS absorbance.

All three methods were used on Tower 1 and the results showed a high UV-VIS value and high points of macro-stickies whilst the adsorption and dyeing method showed almost no sticky content.

During the second round of experiments overall lower stickies content has been observed in the process (see 6-2 and 6-3). It could be interpreted as an indication for the cleaner system.

6.5 CONCLUSIONS

No correlations between adsorption and dyeing method and UV-VIS could be observed. On one hand, this could be due to different size range of particles that are determined by these two methods. On the other hand, this could also indicate unreliability of these methods.

Even though the adsorption and dyeing method does not provide quantitative evaluation of stickies, it determines particles which are tacky. This is not necessarily the case for UV-VIS and cationic demand measurements.

7 PROCESS SURVEY 3: STICKIES SURVEY MARCH 2012

7.1 INTRODUCTION AND METHOD

During the Process Survey 1 (week 8-10) it was found that there are substantial variations in process parameters (such as temperature, pH and cationic demand) along the process, which might have an impact on colloidal and chemical balances in the system. This may, in turn, favor formation of secondary stickies and agglomeration of micro-stickies. Results obtained in Process Survey 1, however, did not provide appropriate information basis for drawing conclusions about stickies deposition mechanisms and accumulation locations.

In order to be able to understand the stickies formation and accumulation mechanisms in the system, information about the size distribution (micro-, macro-, dissolved and colloidal stickies) and origin of stickies (primary or secondary) is crucial. Since most of the existing stickies determination methods are applicable within a certain particle size range, a combination of available methods has been used to obtain a more robust picture. In addition to available methods at the mill, the adsorption and dyeing method, which was introduced and tested in Process Survey 2, as well as a DCM-extraction method was used. A summary of stickies determination methods used in this survey is presented in Table 7-1. In addition, temperature and pH has been measured in order to observe the variations along the process.

Table 7-1 Stickies determination methods used in Stickies Survey 3.

Method	Type of stickies measured	Performed by
DCM extraction	Total amount of stickies	Clariant
Handsheet method	Macro stickies	SCA Edet Laboratory
Adsorption of stickies	Micro stickies	Banmark Oy AB
UV-VIS absorbance	Dissolved and colloidal particles	Chalmers Masters students

Based on the study of historical data, it has been suspected that cleaning occasions due to sticky deposits on wires and felts occur more frequently when running toilet paper in comparison with the wet-strong quality. Therefore, the aim of this survey was to observe the differences in process parameters and amount of sticky deposits in the system while running these two qualities. The survey was performed during two days and it was planned so that on the first day paper machine 8 was running wet-strong tissue product and the second day – toilet paper.

Sampling points along the process for Day 1 and Day 2 are presented in Table 7-2.

Table 7-2 Sampling points Day 1-2 during stickies survey march 2012.

Day 1 (Wet strong)	Day 2 (Toilet Paper)
In Flotation	In flotation
Out Washing	Out Washing
Out Screw press 2	Our Screw press 2
Water Loop 2	Water Loop 1
Pulp Tower 1	Pulp Tower2
Pulp Chest 12	Pulp Tower 3
Pulp Chest 5	Pulp Chest 7
Machine Chest PM8 Middle	Machine Chest PM8 Middle
Machine Chest PM8 Yankee	Machine Chest PM8 Yankee
Headbox PM8 Middle	Headbox PM8 Middle
Headbox PM8 Yankee	Headbox PM8 Yankee
Wire Pit PM8	Wire Pit PM8
Clear Filtrate μ PM8	Clear Filtrate Krofta PM8
Sludge Krofta PM8	Sludge Krofta PM8
White Water (Blekt BV)	White Water (Blekt BV)

As mentioned in the previous chapter, adsorption and dyeing method performed by Banmark is based on visual evaluation of the dyed stickies which have been attached to the plastic bottle. Therefore, quantification of the obtained results is complicated and no grading system is provided by Banmark Oy. However, in order to be able to compare obtained results, a rough evaluation system has been developed where amount of stickies has been graded on a scale from 0 to 5 (see Table 7-3).

Table 7-3 Grading used to evaluate the results obtained by adsorption and dyeing method

Grade	Amount of stickies
5	Very high
4	High
3	Medium
2	Low
1	Very Low
0	None

Thus, it must be kept in mind that values provided for adsorption and dyeing method are only useful for comparison of obtained results in the particular survey and should not be perceived as a generally representative value.

7.2 RESULTS AND DISCUSSION

Results obtained from the Stickies Survey 3 for Day 1 and Day 2 are presented in Table 7-4 and Table 7-5 respectively and further in diagrams which can be found in Appendix E. Note that

amount of macro-stickies, concentration, ash content and amount of DCM extractives have not been determined for the process waters.

Table 7-4 Results Day 1 (Wet-strong quality)

No	Sampling points	Day 1 Wet-Strong Paper								
		Temp (°C)	UV-VIS (Abs)	Conc (%)	Macro Stickies		Ash content (%)	pH	Adsorption and Dyeing	DCM (%)
					No/100g	points/100g				
1	In Flotation	45	8,9	1,48	540	870	21,8	7,6	5	0,567
2	Out Washing	43,8	9,9	4,90	60	170	7,1	7,5	4	0,281
3	Out Screwpress	50,1	1,3	*	40	70	1,2	8,6	3	0,100
4	Water Loop 2	60	11,2	*	*	*	*	8,2	1	*
5	Tower 1	38,1	6,8	3,42	130	220	1,2	6,9	0	0,034
6	Chest 12	42,3	4,2	3,05	60	70	0,49	7,3	1	0,067
7	Chest 5	37,6	6,1	3,23	30	90	0,74	7,2	3	0,135
8	Chest 6	38,6	5,2	3,23	10	20	0,28	7,3	1	0,023
9	Machine Chest PM8 Middle	38,6	8,4	3,07	20	40	0,85	7	3	0,149
10	Machine Chest PM8 Yankee	41	6	3,70	70	130	0,62	7	3	0,072
11	Headbox PM8 Middle	38,6	6	*	*	*	*	7,4	2	*
12	Headbox PM8 Yankee	39,3	6	*	*	*	*	7,5	3	*
13	Wire Pit PM8	39	6,3	*	*	*	*	7,6	3	*
14	Clear Filtrate Krofta PM8	38,9	5,8	*	*	*	*	7,6	1	*
15	Sludge Krofta PM8	38	4,9	3,25	110	290	14,4	7,4	2	0,037
16	White Water (Blekt BV)	27,5	3,1	*	*	*	*	6,8	0	*

Deinking Line 1 Pulp PM8 System White Water

Table 7-5 Results Day 2 (Toilet paper)

No	Sampling points	Day 2 Toilet Paper								
		Temp (°C)	UV-vis (Abs)	Conc (%)	Macro Stickies		Ash content (%)	pH	Adsorption and Dyeing	DCM (%)
					No/100g	points/100g				
1	In Flotation	48,6	9,3	1,28	220	1030	22,6	7,3	5	0,377
2	Out Washing	47,6	12,2	3,61	190	460	8,1	7,3	3	0,227
3	Out Screw press 2	52	1,5	*	50	110	1,2	8,4	2	0,224
4	Water Loop 2	61	13,7	*	*	*	*	8,1	2	*
8	MC Pump to Tower 1 and 2	59,1	16,4	9,48	110	280	1,5	6,8	2	0,057
5	Tower 2	46,2	7,9	3,44	80	140	1,2	6,6	1	0,051
6	Tower 3	46,2	10,6	2,00	140	200	6,9	6,9	4	0,163
7	Chest 7	45,9	5,7	3,47	130	220	1,3	6,6	3	0,137
9	Machine Chest PM8 Middle	47,2	9,3	3,14	160	280	2,6	6,6	3	0,210
10	Machine Chest PM8 Yankee	47,7	8,1	3,42	70	160	2,1	6,6	2	0,070
11	Headbox PM8 Middle	45,2	7	*	*	*	*	7,6	3	*
12	Headbox PM8 Yankee	45,7	6,5	*	*	*	*	7,7	3	*
13	Wire Pit PM8	46	6,9	*	*	*	*	7,8	4	*
14	Clear Filtrate Krofta PM8	43,7	7,2	*	*	*	*	7,7	2	*
15	Sludge Krofta PM8	42,1	6,8	2,20	310	440	41,3	7,4	4	1,063
16	White Water (Blekt BV)	42,1	6,2	*	*	*	*	7,1	1	*
17	White Water (Virgin)	44,1	7,2	*	*	*	*	7,5	0	*

Deinking Line 1 Pulp PM8 System White Water

Temperature profile along the process during both days is presented in Figure 7-1. It can be seen that the temperature in different process locations was lower during Day 1 while running wet-strong paper compared to Day 2. It can be explained by addition of the fresh water to the system during Day 1 since temperature of white water was significantly lower during Day 1 compared to Day 2.

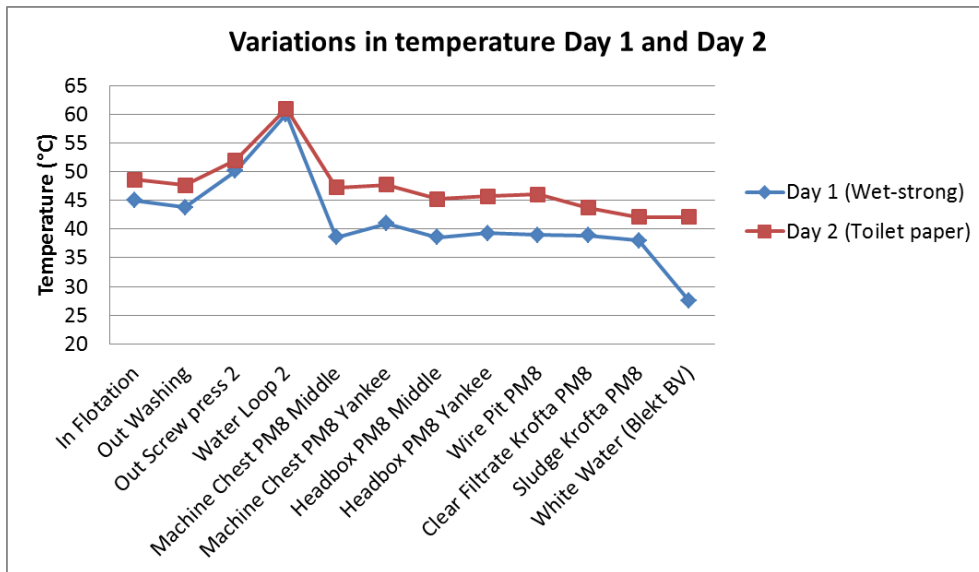


Figure 7-1 Comparison of variations in temperature during Day 1 (wet-strong paper) and Day 2 (toilet paper)

It can be also seen that temperature varies rather much along the deinking line but it is relatively stable in PM8 system.

pH variations along the system during Day 1 and Day 2 are presented in Figure 7-2.

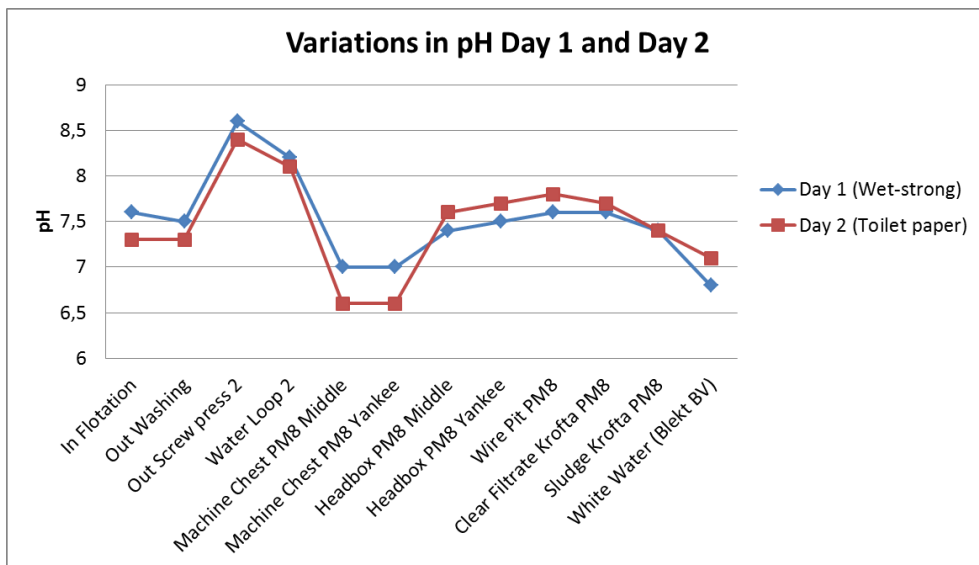


Figure 7-2 Comparison of variations in pH during Day 1 (wet-strong paper) and Day 2 (toilet paper)

It was observed that pH was slightly lower in deinking line during Day 2, but the opposite trend was noticed in Headbox PM8. It is notable that pH varies rather much along the process, difference in pH between Machine Chest and Headbox was more than 1 unit during Day 2, which might contribute to stickies-related problems in the paper machine. However, pH difference in Machine Chest and PM8 system during Day 1 was not as significant as during Day 2. As it was discussed in Process Survey 1, the critical pH range is not known, but it is clear that the variations in this parameter can be crucial to deposit formation.

7.2.1 DEINKING LINE 1

The samples taken before the first flotation step serve as a reference for the quality of raw material entering the process. The results obtained from samples in to flotation indicate that during Day 1 the raw material entering the process contained more detrimental substances in terms of amount of macro-stickies and content of DCM extractives compared to Day 2.

Reduction in amount of macro-stickies as well as DCM extractives along the deinking line has been observed during both days. Nevertheless, the efficiency of the deinking line 1 was higher during Day 1, i.e. removal of macro-stickies was 92,6% and reduction of the DCM extractives along the deinking line was 82,36 % compared to 77,3% and 40% respectively during Day 2.

Results obtained by using UV-VIS spectrophotometer deviated from the general trend, i.e. slight increase in absorbance values between sample into the first flotation step and after the washing stage has been observed during both days. Most probably the source for the detrimental substances is water from the Screw-press 1 which is fed back into the system before the cyclones. Due to their size colloidal and dissolved substances are rather hard to eliminate from the process and pre-flotation does not provide efficient removal of these substances. According to the literature, the most effective stage for the removal of the micro-stickies is post-flotation [46].

The UV-VIS value for the pulp coming out from the screw-press 2 was significantly lower compared to the sample "Out washing". The explanation might be that colloidal and dissolved particles are removed together with the water in dewatering stage, resulting in a rather high UV-VIS value in water loop 2 during Day 2 (see Table 7-5). However, it must be noted that the pulp from the Screw press 2 was diluted with tap water which might influence the obtained result.

Extra sample taken Day 2 (MC pump) exhibited unexpected values (Table 7-5). Substantial increase in UV-VIS values can be seen for sample taken from the mixing pump compared to the one out from screw-press 2. This is also far higher than for the other following pulps. This indicates rise in amount of colloidal and dissolved substances which could be caused by the white water used for dilution. However, the UV-VIS value for the white water was low during both days and therefore there is no objective explanation for this phenomenon and the credibility of this value has been questioned. Moreover, after performing the Process Survey 1 it was concluded that UV-VIS values vary substantially within one day which means that single value is perhaps not representative for the whole day.

However, similar trend has been observed using hand-sheet method, i.e. amount of macro-stickies was higher in the pulp coming out from the mixing pump compared to the one coming out from the Screw-press 2. It has been observed that temperatures in the Screw press 2 and mixing pump are rather high compared to other sampling points (~60°C). The white water which is used for dilution of pulp coming out from the Screw-press 2 is also heated-up to 60°C before entering the mixing pump. At this temperature tackiness of the sticky particles increase which may lead to agglomeration of micro-stickies and formation of macro-stickies. However, available information and methods do not provide any clear evidence for this theory.

The adsorption and dyeing method did not exhibit substantial differences in stickies content in the pulp coming out from Screw press 2 and the one coming out from the mixing pump. The DCM extraction value was much lower in the mixing pump compared to the sample out from Screw press 2. The pulp in Tower 2 also had a low DCM value which is reasonable since the pulp from the mixing pump is fed to Tower 1 and 2.

7.2.2 PULP AND WHITE WATER

When comparing the pulp sources it must be kept in mind that different qualities have been run during Day 1 and Day 2 which means that different pulp sources have been involved.

It has been observed that during Day 1 the pulps contained less detrimental substances determined by hand-sheet method, adsorption and dyeing method as well as DCM extraction method. The ash-content values were also lower on Day 1.

It can be seen that during Day 1, Pulp Chest 5 stood out in comparison with the other pulp sources in terms of results obtained by DCM extraction as well as adsorption and dyeing method. Taking into account that Pulp Chest 5 contains virgin pulp – HTCTMP it is rather surprising. The occurrence of stickies in virgin pulp can perhaps be justified by the presence of wood pitch.

The results obtained from Pulp Chest 12 showed low values for all methods. Since Pulp Chest 12 contains pulp from the internal wet-strong broke line its quality can differ rather much. Traces of stickies were also observed in Chest 6 (Bleached Kraft) by adsorption and dyeing method even though this pulp was not supposed to contain any stickies. The possible reason could be that virgin Pulp chests have a common dilution water source in which colloidal and dissolved particles can accumulate, contributing to micro-stickies in the pulp. Another explanation could be that Kraft pulp might also contain wood pitch.

When comparing Tower 1 and Tower 2 both of them showed similar UV-VIS and ash content values, amount of macro-stickies was, however, lower in Tower 2 whilst the adsorption and dyeing as well as DCM extraction method showed a somewhat higher value for Tower 2.

The sample from Pulp Tower 3 showed highest values determined by all methods even though the values from the hand-sheet method were only slightly higher than in Tower 1 and almost equal to the ones in Chest 7 (internal broke). This might indicate high amount of micro-stickies in Tower 3 which may be justified by the fact that pulp in Tower 3 originates from DIP line 2 which consists of less purification stages compared to DIP1. However, the most significant is absence of post-flotation in DIP2, since this unit operation is claimed to be the most efficient in terms of removal of micro-stickies. Moreover, DIP2 contains only one water loop which facilitates the accumulation of dissolved and colloidal substances.

Nevertheless, the measurements performed on the pulp from Pulp Chest 7 also showed high values determined by all methods and this can be explained by the fact that it is pulp from the internal broke line in which, as mentioned earlier, the quality differs rather much.

The samples taken from the two White water tanks showed UV-VIS values in the same levels as Tower 2 and Chest 7. Traces of stickies could be observed in the White water used as dilution water in the pulp towers, which can contribute to stickies. However, low values for the dilution water indicate that the high values observed in Tower 3 are more dependent on the lower cleaning efficiency of the DIP line 2 and the fact the raw material has a lower quality. On the other hand, it has been noticed in previous “trials” that the quality of the dilution water can differ quite much.

The sample from the second White water tank (Virgin), used as dilution water in the virgin pulp lines, was collected Day 2 in order to see if it could be a probable source of the stickies in Chest 5 (Day 1). But, since the adsorption and dyeing method did not indicate any stickies in this water during Day 2, wood pitch is still the most probable source of the stickies in Chest 5.

7.2.3 PAPER MACHINE 8

Differences in amount of detrimental substances in Middle and Yankee layer of the Machine Chest have been observed during both days. This is mainly due to different pulps which are fed into the respective layers (see Table 7-6 for Day 1 and Table 7-7 for Day 2).

Table 7-6 Pulp distribution in Machine Chest Middle, Yankee and Hood layer during Day 1 when running wet-strong paper. The total pulp dosage fed to the paper machine is presented to the right.

Day 1 (Wet-strong)				
Pulp Source	Middle	Yankee	Hood	Tot. Pulp Dosage
Tower 1	0%	70%	70%	42%
Chest 5	74,3%	0%	0%	29,7%
Chest 6	0%	30%	30%	18%
Chest 12	25,7%	0%	0%	10,3%

Table 7-7 Pulp distribution in Machine Chest Middle, Yankee and Hood layer during Day 2 when running toilet paper. The total pulp dosage fed to the paper machine is presented to the right.

Day 2 (Toilet)				
Pulp Source	Middle	Yankee	Hood	Tot. Pulp Dosage
Tower 2	50%	96%	96%	74,1%
Tower 3	50%	0%	0%	23,3%
Chest 7	0%	4,0%	4,0%	2,3%

During Day 1, Middle layer exhibited higher content of DCM extractives, ash content and UV-VIS absorbance values compared to the Yankee layer while adsorption and dyeing method indicated equal level of stickies in both layers. Macro-stickies measurements indicated higher values for the Yankee layer compared to the Middle layer, which is due to a 70% admixture of Tower 1 in Yankee layer.

However, during Day 2 the Middle layer exhibited higher stickies content in terms of macro-stickies, DCM extractives, UV-VIS absorbance as well as adsorption and dyeing method. In case of the Middle layer the high values were expected since it contained 50% pulp from Tower 2 and 50% pulp from Tower 3 which showed high values for all methods. The Yankee layer also showed anticipated values, i.e. they were slightly higher than the ones obtained from Tower 2 (96% of this pulp was contained in Yankee layer) and this is most probably due to the admixture of Chest 7.

During Day 1 traces of stickies were indicated in Clear filtrate by using adsorption and dyeing method. Stickies content in the Wire Pit was evaluated as medium using the same method. During Day 2 adsorption and dyeing method indicated rather high amount of stickies in Wire Pit and surprisingly high value for the Clear filtrate from the Krofta. Since the system was more contaminated during Day 2, probably the efficiency of Krofta is also affected. One more indication of the contamination level in the system is the Krofta sludge which exhibited high values obtained by all methods. Possible interpretation of this phenomenon could be that the

pulps run on PM8 during Day 2 contribute to a more contaminated system and thus heavier load on the Krofta.

High amount of macro-stickies was observed in the Krofta sludge during both days of the survey. As mentioned in theory part anionic trash and fines agglomerate using flocculants in order to be able to remove them by flotation in the Krofta [32]. Thus, the amount of macro-stickies in the DAF sludge might indirectly indicate the amount of micro-stickies and/or secondary/potential stickies in the white water.

After performing Stickies survey 3 and taking into account the results obtained from the first process survey, the usefulness of the UV-VIS method in terms of stickies determination has been questioned. This is mainly due to high variation of the UV-VIS absorbance values within one day as it was observed during the process survey 1. This raises a doubt whether a single value is representative for the whole day. Moreover, there are no clear correlations between UV-VIS values and results obtained by other methods.

Results indicated an overall dirtier system during Day 2. It should be mentioned that the fresh water was added to the system during Day 1 (which can be seen from the lower temperature in system). Therefore, it is hard to conclude whether the cleaner system is due to addition of wet-strength agent, pulp dosage or addition of the fresh water.

7.3 CONCLUSIONS STICKIES SURVEY MARCH 2012

The system was less contaminated while running wet-strong quality (Day 1) compared to the toilet paper (Day 2). Most probably the main reason is the inferior quality of the pulp when running the toilet paper which contains higher amount of recycled pulp with relatively low quality and addition of fresh water.

It is rather hard to judge whether wet-strength additive has an impact on stickies content in the system but it is quite clear that the pulp fed into the paper machine has a direct influence on the content of detrimental substances in the system.

One possible source of stickies still might be dilution water which carries around the colloidal particles. These substances might contribute to the formation of secondary stickies under certain conditions (pH, temperature, colloidal charge shock).

Tower 3 is suspected to contribute to the stickies-related problems since it exhibited the largest amount of stickies determined by all methods.

Amount of macro-stickies in the Krofta sludge might indirectly indicate the amount of micro-stickies in the white water. Obtained results showed higher amount of detrimental substances in sludge during the Day 2 which correlates with general observations, i.e. more contaminated PM8 system during Day 2.

The UV-VIS method has been questioned as a method used for daily measurements.

8 MIXING EXPERIMENTS

8.1 INTRODUCTION

Based on results obtained from analysis of historical data and Process Survey 3, the pulp mix of Tower 2 and Tower 3 was suspected to contribute to stickies-related problems in the paper machine. Therefore, mixing experiments were performed at the laboratory. The aim of the mixing experiments was to observe differences in macro-stickies measurements between the single pulps and the mixed sample. Pulp was also stored during the night (in a 40°C water bath) to see if there were any differences in the results from macro-stickies measurements. This was performed in order to simulate the pulp storage in towers since the pulp residence time in the towers sometimes can be rather long, e.g. when having stops due to process problems etc.

8.2 METHOD

Pulp samples from Tower 2 and Tower 3 were collected in 2 liter plastic bottles, two samples for each pulp. One sample from Tower 2 and one from Tower 3 have been stored in a 40°C water bath during 24 hours.

The pulp mix was prepared by mixing 30% of pulp from Tower 3 and 70% from Tower 2. After preparation it was diluted with distilled water to 1,5% concentration (appropriate concentration to achieve good mixing in the mixing equipment). Determination of concentration in pulp suspension has been performed according to TAPPI standard T240. 750g of the mixed sample (concentration ~1,5%) was placed in 1L glass beaker, heated to 40°C and stirred during 10 minutes. At the same time, identical procedure was performed for single samples from Tower 2 and Tower 3 in order to be able to compare amount of macro-stickies in individual pulps and mixture.

After stirring, concentration of pulp samples has been repeatedly determined for macro-stickies measurements. Finally, amount of macro-stickies was determined for all samples according to SCA standard ST0082 se method chapter 3.4.

8.3 RESULTS AND DISCUSSION

The results of the mixing experiments are summarized in Table 8-1. Macro-stickies values both in No/100g and points/100g are presented. In column 8 and 9 the difference between Day 1 and Day 2 is presented to see how the samples are affected by storing. The last column presents the difference between the no of stickies per 100g in the mix and expected number of stickies per 100g when mixing Tower 2 and Tower 3 with values seen in column 4 (see Table 8-1).

Difference in amount of stickies is calculated by using Equation (5) and (6):

$$N_c = (n_1 \times x_1) + (n_2 \times x_2) \quad (5)$$

$$Diff = N_m - N_c \quad (6)$$

Where

N_c = Calculated Value

N_m = Values obtained from mixing experiment

n_x = number of stickies in respective tower

x_x = percent dosage of each pulp in the mix

Table 8-1 Results obtained from the mixing experiments. Each experiment (see sample No) includes 3 samples; Tower 2, Tower 3 and a mix of these two pulps. Difference between value obtained from mixing experiments and the calculated value is presented in the last column to the right.

No	Sample Point	Macro Stickies (No/100g)	Macro Stickies (points / 100g)	Sample 2 Macro Stickies (No/ 100 g)	Sample 2 Macro Stickies (points/ 100g)	Diff Day 2- Day 1 (No/ 100g)	Diff Day 2- Day 1 (points/ 100g)	Value mix- Calculated Value mix (No/100g)	
1	Tower 2	220	400	80	80	-140	-320		
	Tower3	150	240	180	180	30	-60		
	Mix	110	290	160	160	50	-130		-89
2*	Tower 2	80	80	*	*	*	*		
	Tower3	180	180	*	*	*	*		
	Mix	160	160						50
3	Tower 2	90	120	80	80	-10	-40		
	Tower3	80	110	10	10	-70	-100		
	Mix	120	360	90	90	-30	-270		33
4	Tower 2	50	50	130	180	80	130		
	Tower3	120	120	110	220	-10	100		
	Mix	70	100	90	120	20	20		-1
	Mix 2	30	30						
5*	Tower 2	80	80	*	*	*	*		
	Tower3	10	10	*	*	*	*		
	Mix	90	90						31
6*	Tower 2	130	180	*	*	*	*		
	Tower3	110	220	*	*	*	*		
	Mix	90	120						-34

Mix = Tower 2 and Tower 3

2* Sample 1 stored during the night

5* Sample 3 stored during the night

6* Sample 4 stored during the night

It can be seen in Table 8-2 that samples collected during Day 1 (sample 1) showed lower amount of macro-stickies in pulp mix compared to single pulps from Tower 2 and Tower 3.

Sample 2, which is the pulp collected same day as sample 1 and stored during the night, showed lower macro-stickies values for Tower 2 compared to sample 1 while Tower 3 showed higher values in terms of number of macro-stickies/100g. The pulp mix exhibited similar amount of macro-stickies as sample 1, however it must be noted that it was higher than the calculated

value which takes into account the amount of macro-stickies in each individual pulp and their percentage in mixture. This is opposite to the results obtained during Day 1.

It can be seen that for samples 1, 4 and 6 the values obtained from the mixed sample are lower than the calculated value from the two components separately whilst for sample 2, 3 and 5 it is higher.

No clear trend could be seen from the storing experiments either. Some samples show higher values during Day 2 while some show lower. Due to the inconsistent results and since the reliability results of macro-stickies measurements are sometimes questioned a repeatability test was performed. A 5 liters sample was collected from Tower 3 and macro-stickies measurements were performed. All samples were analyzed by two persons. Results obtained from these measurements are presented in Table 8-2. It must be noted that these samples served only for obtaining information about repeatability of the method, i.e. to what extent values can vary for the same sample.

Table 8-2 Results obtained from repeatability test for macro-stickies measurements performed on samples from Tower 3

Sample No	Macro-stickies (No/100g)	Macro-stickies (points/100g)
1	90	90
2	80	80
3	100	130
4	160	160
5	100	130
Standard Deviation	31,3	32,7

It can be seen that obtained values varied from 80 to 160 both in terms of no/100g and points/100g resulting in standard deviation of 30 which is acceptable value.

8.4 CONCLUSIONS

Based on obtained results no clear effect of the mixing of pulps from Tower 2 and Tower 3 on amount of macro-stickies could be observed nor for the storing experiments. The standard deviation of macro-stickies measurements is at an acceptable level.

9 METHOD INVESTIGATION

9.1 INTRODUCTION

One of the main factors hindering the solving of the problem with adherent deposits is lack of reliable methods for their determination. Therefore, further investigation of adsorption and dyeing method used in Stickies Surveys 2 and 3 was performed.

9.2 ADSORPTION AND DYEING EXPERIMENT

The adsorption and dyeing method elaborated by Banmark is, as mentioned, based on adhesion of stickies on the surface of plastic bottles and further dyeing. The main drawback of this method is lack of opportunity to quantify the stickies content which makes it rather hard to determine the critical concentration of micro-stickies leading to deposition. One of the methods mentioned in literature is rather similar to Banmark's method, the only difference is that the plastic bottle is not dyed but weighted before and after immersing it into the sample, simultaneous mixing and heating [9]. In this way the weight of the stickies could be obtained. Blanks have been performed in order to determine the bottle weight loss during experiment and trials have been performed by using pulp before the first flotation step in DIP1. Experiments have been performed using 250mL and 125 mL bottles.

9.2.1 RESULTS

The results from the weighing experiments are presented in Appendix E. In Table 1 results from the blank samples and in Table 2 from the pulp samples are presented.

Blank experiments performed using 250 mL and 125 mL bottles both exhibited high variations in weight loss. Taking into account that the weight of stickies adsorbing on the plastic bottle is very low, obtained variation is unacceptable since it in some cases was even larger than the weight of stickies. This makes the method unreliable and no further trials were made.

9.2.3 CONCLUSIONS

Weighing of adsorbed stickies cannot be used for the further work due to high weight-loss of the plastic bottles and variance in obtained results.

9.3 FILM METHOD EXPERIMENTS

9.3.1 INTRODUCTION

Since adsorption and weighing of stickies was not eligible due to high weight-loss of the plastic bottles, evaluation of another method provided by Clariant has been performed. It is based on

adsorption of stickies on a plastic film and dyeing followed by image analysis which provides opportunity to quantify amount of stickies.

9.3.2 METHOD

In order to test the film method provided by Clariant, three pulp samples are collected: in flotation, pulp Tower 2 and pulp Tower 3.

740 g of the sample (mass of the samples required for 10 g (o.d) pulp used in experiments) before the first flotation step in DIP1 is weight up and pulped in laboratory pulp disintegrator for 2 minutes. Then dilute it with hot tap water (~60°C) until 1000 mL (in 1 l glass measuring cylinder).

The solution is mixed and separated into 200 mL samples. Then the sample is poured into the 400 mL beaker where the plastic film is placed. The pulp is agitated at 200 rpm for 15 minutes. During the mixing 0,4 mL of the dye is added. After stirring, the film is removed, carefully rinsed with cold tap water to remove the fibers and contaminants and dried in oven at 105°C for 10 minutes. After drying visual evaluation of the film is performed.

9.3.3 RESULTS

After experiment no stickies were visible on the film. The film was observed in microscope and only 1 sticky particle could be detected. Numerous small black dots could also be observed in microscope which could be potential stickies.

The same procedure has been repeated at higher temperature (up to 80°C), however large stickies still could not be observed. The amount of dye has been increased until 1 mL but it did not give any positive result.

9.3.4 ELIGIBILITY TEST

Since almost no stickies adsorbed on the film when running the sample "In flotation", the test was performed by adding "artificial" sticky material (envelopes and post-it notes) and run as mentioned previously. As a result rather large stickies could be detected by the "naked eye". Though the methods so far only has been used for laboratory use, to prove the effect of stickies passivation chemicals added to a mixture of pulp with admixture of stickies from envelope and post-it notes.

9.3.5 IMPROVEMENT OF THE FILM METHOD

Since results from the previous experiment performed following the instructions provided by Clariant were not successful, attempts to adopt the method to industrial use were made.

Micro-stickies are mainly present in the water phase, therefore one of the possible reasons why stickies were not detected in previous experiments is that the pulp concentration was probably too high and fibers are preventing the stickies from reaching the surface of the film. In order to check if this has an influence on the result; one sample was taken from the Wire Pit on paper machine 8. In previous experimental rounds it has been observed that stickies content at this point is rather high.

During the experiment small dyed particles have been observed on the film, mostly on the side which is in contact with the glass beaker. When the film was removed, dyed stickies were observed only where the film was overlapping which might mean that the stickies have been removed while taking out the film from the beaker.

In order to check whether the dye is coloring the stickies or not; two experiments were run with plastic bottles (similarly as in case of adsorption and dyeing method performed by Banmark) – one with sample from wire pit and one with sample “In flotation”. Small amounts of dyed particles were observed. Since the wire pit sometimes might contain small amounts of stickies, one more experiment was run with the sample in flotation, which normally contains rather high amount of stickies. However, only negligible amount of dyed stickies was observed.

In order to test the adherence of the stickies to the wire, one experiment has been run by using the material taken from an old wire (from the paper machine). The material was cut into 27,5 cm × 5 cm stripes and cleaned with acetone in order to remove the stickies which have been formed on the wire during its exploitation in the process. The stripe of the wire material was placed into a beaker and 400 mL of undiluted pulp from the sample point “In flotation” was added. The pulp was then heated until 60°C and then 0,4 mL of the dye was added. The pulp suspension was stirred for 30 min at 200 rpm. Then, the wire stripe was carefully removed from the beaker, washed with cold tap water and dried in the oven at 105°C for 10 minutes. The wire stripe was observed in microscope but no dyed stickies could be seen.

9.3.4 CONCLUSION

After 2 days of experimental work it has been concluded that the plastic film method provided by Clariant cannot be applied as the method for stickies determination in industrial pulp and process water.

10 PROCESS SURVEY 4: STICKIES SURVEY MAY 2012

10.1 INTRODUCTION

A final survey was performed based on results from Historical data and previous surveys. Process Survey 4 was performed in order to evaluate the impact of Tower 3 as well as addition of wet strength agent. The effect of Tower 2 was also studied but was not the main purpose of the investigation.

To achieve this, pulps and process water were analyzed from sampling points according to Table 10-1. Effect of Tower 1 and Tower 2 is studied when comparing Day 1 (Trial 1) with Day 2 AM (Trial 2), effect of Tower 3 when comparing Day 2 PM (Trial 3) and Day 2 AM and effect of wet-strength agent when comparing Day 3 (Trial 4) and Day 2 PM.

Table 10-1 Quality, pulp and sample points for Trial 1-4 (Day 1-3).

Trial 1: Day 1 Toilet (T2,T3)	Trial 2 Day 2 AM Toilet (T1,T3)
In Flotation DIP1	In flotation DIP1
Pulp Tower 2	In RP2
Pulp Tower 3	Pulp Tower 1
Machine Chest PM8 Middle	Pulp Tower 3
Machine Chest PM8 Yankee	Machine Chest PM8 Middle
Wire Pit PM8	Machine Chest PM8 Yankee
Clear Filtrate Krofta PM8	Wire Pit PM8
Sludge Krofta PM8	Clear Filtrate Krofta PM8
White Water (Blekt BV)	Sludge Krofta PM8
	White Water (Blekt BV)
Trial 3: Day 2 PM Toilet (T1)	Trial 2: Day 2 AM Toilet T1,T3)
Pulp Tower 1	In RP2
Machine Chest PM8 Middle	Pulp Tower 1
Machine Chest PM8 Yankee	Pulp Chest 5
Wire Pit PM8	Pulp Chest 12
Clear Filtrate Krofta PM8	Machine Chest PM8 Middle
Sludge Krofta PM8	Machine Chest PM8 Yankee
White Water (Blekt BV)	Wire Pit PM8
	Clear Filtrate Krofta PM8
	Sludge Krofta PM8
	White Water (Blekt BV)

This Stickies survey has been performed in collaboration with the companies Clariant and BIM Kemi AB. Methods used in the survey are summarized in Table 10-2. In addition to the methods used in process survey 3, CCT (Colloidal collection test) has been used for determination of micro-stickies. Temperature, pH and cationic demand have also been measured in order to observe the variations along the process.

Table 10-2 Stickies determination methods during survey May 2012.

Method	Measured type of stickies	Performed by
DCM extraction	Total amount of stickies	Clariant
Handsheet method	Macro stickies	Chalmers Masters Students
Adsorption of stickies	Micro stickies	Chalmers Masters Students
UV-VIS absorbance	Dissolved and colloidal particles	Chalmers Masters students
Cationic Demand	Dissolved and colloidal particles	Chalmers Master students
CCT	Micro-stickies	BIM Kemi AB

10.2 RESULTS AND DISCUSSION

10.2.1 TEMPERATURE, PH AND ASH CONTENT

In Figure 10-1 and 10-2 the process parameter measurements from Trial 1-4 are presented and variations in the system can be studied in the same way as during Process Survey 3.

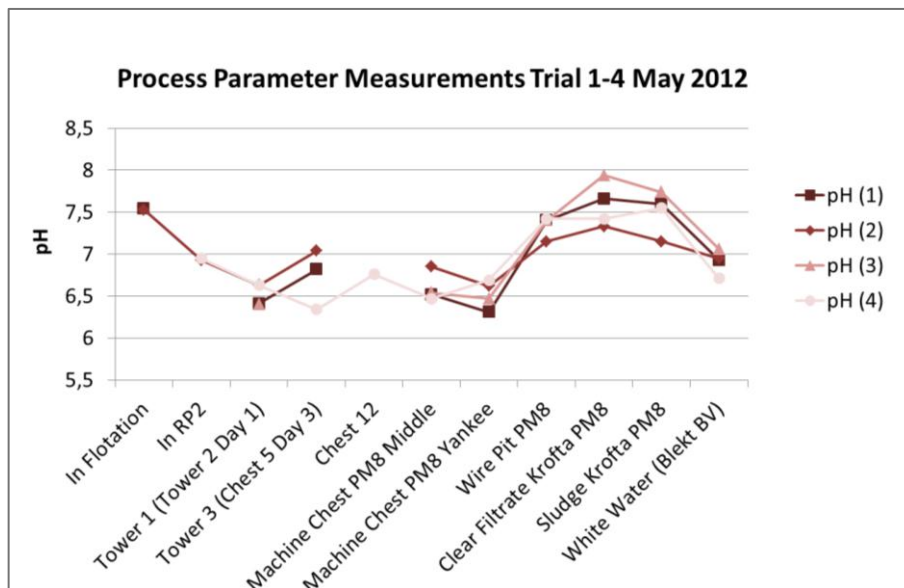


Figure 10-1 Results from pH measurements during stickies survey May 2012, Trial 1-4.

Similarly as in Process Survey 3, it can be seen from Figure 10-1 that the pH varies along the process, in this case between 6,3-7,9.

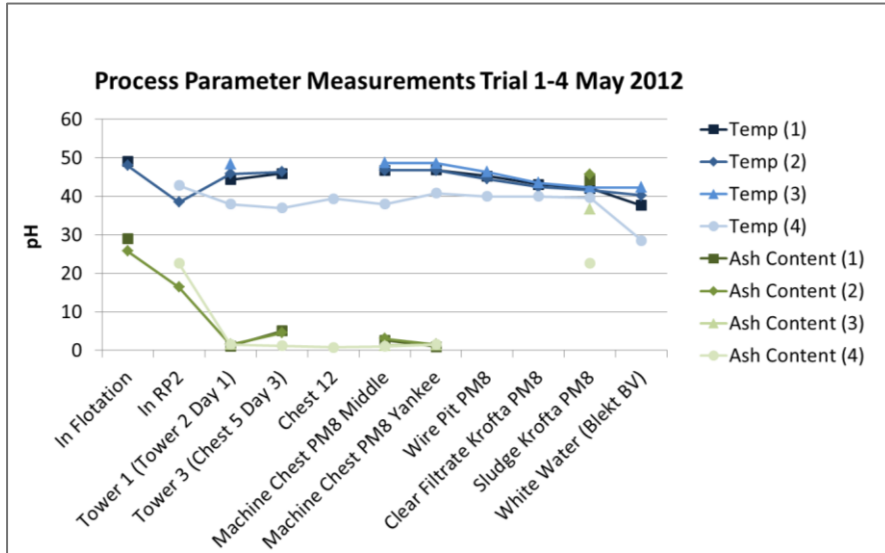


Figure 10-2 Results from temperature and ash content measurements during stickies survey May 2012, Trial 1-4.

In Figure 10-2 it can be seen that the system temperature is lower during trial 4. Further it can be seen that the ash content is higher in the overall system especially in the Krofta during Trial 1 and Trial 2. This is probably due to the higher ash content in the pulp from Tower 3.

All results obtained from the stickies survey for Trials 1-4 are presented in Table 10-3. During Process Survey 1 it has been observed that CMC dosage influences the cationic demand in the system. However, during process survey 4 CMC dosage was constant and therefore should not contribute to variations in cationic demand.

Table 10-3 Results from trial 1- 4 are presented; also quality and pulps run on the machine are included.

Trial 1: Day 1 Toilet (T2, T3)												
No	Sampling points	Temp (°C)	UV-vis (Abs)	Conc (%)	Macro Stickies		Ash content (%)	pH	Adsorption and Dyeing	DCM (%)	Cationic Demand (µekv/l)	CCT (No)
					No/100g	points/100g						
1	In Flotation DIP 1	49,1	9,8	1,22	140	230	28,9	7,54	5	0,52	-352	*
2	Tower 2	44,3	6,92	3,74	320	560	1,1	6,41	2	0,08	-135	*
3	Tower 3	45,9	11,38	3,11	140	140	5	6,82	4	0,45	-69	*
6	Machine Chest PM8 Middle	46,7	9,13	3,19	250	370	2,6	6,52	3	0,23	-126	*
5	Machine Chest PM8 Yankee	46,8	7,37	3,44	300	360	0,98	6,31	2	0,07	-166	*
8	Wire Pit PM8	45,2	7,64	*	*	*	*	7,4	3,5	*	-254	2048
9	Clear Filtrate Krofta PM8	43,1	7,35	*	*	*	*	7,66	2	*	-350	46
7	Sludge Krofta PM8	42,1	6,79	3,06	1750	2440	44,2	7,6	2	0,82	-243	*
4	White Water (Blekt BV)	37,6	4,48	*	*	*	*	6,93	3	*	-113	245
Trial 2: Day 2 AM Toilet (T1, T3)												
No	Sampling points	Temp (°C)	UV-vis (Abs)	Conc (%)	Macro Stickies		Ash content (%)	pH	Adsorption and Dyeing	DCM (%)	Cationic Demand (µekv/l)	CCT (No)
					No/100g	points/100g						
2.1	In Flotation DIP1	48	11,03	1,18	80	290	25,8	7,53	5	0,47	-417	*
2.10	In DIP 2	38,5	18,51	3,03	20	20	16,4	6,93	*	1,36	-115	*
2.2	Tower 1	45,8	6,9	3,35	110	170	1,5	6,63	4	0,08	-143	*
2.3	Tower 3	46,3	9,73	3,36	90	120	4,5	7,04	4	0,8	-71	*
2.6	Machine Chest PM8 Middle	46,7	8,94	3,26	70	70	3,04	6,85	2	0,32	-127	*
2.5	Machine Chest PM8 Yankee	46,7	7,02	3,50	110	140	1,5	6,62	3	0,1	-150	*
2.8	Wire Pit PM8	44,5	7,13	*	*	*	*	7,15	3,5	*	-281	2319
2.9	Clear Filtrate Krofta PM8	42,4	7,51	*	*	*	*	7,33	3	*	-287	64
2.7	Sludge Krofta PM8	41,6	6,67	3,18	410	590	45,7	7,15	3	2,32	-232	*
2.4	White Water (Blekt BV)	40,2	4,46	*	*	*	*	6,95	2	*	-149	191
Trial 3: Day 2 PM Toilet (T1)												
No	Sampling points	Temp (°C)	UV-vis (Abs)	Conc (%)	Macro Stickies		Ash content (%)	pH	Adsorption and Dyeing	DCM (%)	Cationic Demand (µekv/l)	CCT (No)
					No/100g	points/100g						
3.2	Tower 1	48,4	8,28	3,85	70	100	1,5	6,4	2,5	0,08	-146	*
3.6	Machine Chest PM8 Middle	48,7	7,7	3,53	130	160	1,3	6,54	2	0,06	-224	*
3.5	Machine Chest PM8 Yankee	48,6	6,72	3,40	320	530	1,4	6,47	3	0,07	-232	*
3.8	Wire Pit PM8	46,3	7,8	*	*	*	*	7,4	3,5	*	-383	731
3.9	Clear Filtrate Krofta PM8	43,5	9,46	*	*	*	*	7,94	2	*	-395	40
3.7	Sludge Krofta PM8	42,3	9,01	3,86	930	1340	36,6	7,74	2	1,34	-314	*
3.4	White Water (Blekt BV)	42,3	5	*	*	*	*	7,06	2	*	-191	116
Trial 4: Day 2 PM Wet-strong (T1, C5, C12)												
No	Sampling points	Temp (°C)	UV-vis (Abs)	Conc (%)	Macro Stickies		Ash content (%)	pH	Adsorption and Dyeing	DCM (%)	Cationic Demand (µekv/l)	CCT (No)
					No/100g	points/100g						
4.10	In DIP 2	42,8	7,25	2,9	10	90	22,6	6,95	5	1,09	-108	*
4.1	Tower 1	38	6,76	3,93	480	630	1,5	6,63	2	0,11	-100	*
4.6	Chest 5	37	3,44	3,55	20	50	1,2	6,34	1	0,09	-261	*
4.7	Chest 12	39,4	4,32	2,93	120	120	0,8	6,76	1	0,1	-145	*
4.2	Machine Chest PM8 Middle	38	8,06	3,2	70	70	1	6,47	2	0,13	-228	*
4.4	Machine Chest PM8 Yankee	40,8	6,73	3,37	320	410	1,5	6,69	2	0,11	-124	*
4.5	Wire Pit PM8	40	6,21	*	*	*	*	7,42	3,5	*	-149	43
4.9	Clear Filtrate Krofta PM8	40	5,75	*	*	*	*	7,42	2	*	-136	14
4.3	Sludge Krofta PM8	39,6	5,69	3,07	730	1150	22,6	7,55	1	0,56	-155	*
4.8	White Water (Blekt BV)	28,5	3,35	*	*	*	*	6,71	1	*	-85	24

Deinking Line
 Pulp Sources
 PM8 System
 White Water

10.2.2 TOILET PAPER QUALITIES (TRIAL 1-3)

Effect of Tower 2 and Tower 3 has been examined while running toilet paper with different pulp dosage. Notable is that there were cleaning occasions both before and after samples during Day 1 were analyzed which indicates that these pulp compositions/process parameters were not optimal.

DIP1 AND DIP2

It can be seen that the raw material entering DIP1 (In flotation) was more contaminated in terms of macro-stickies, DCM extractives and ash content during Trial 1 compared to Trial 2. UV-VIS absorbance was however slightly higher for Trial 2.

Raw material entering deinking line 2 (Trial 2) exhibited very low amount of macro-stickies whereas DCM extraction and UV-VIS values were considerably higher compared to the ones for DIP1 (Trial 1 and 2). DIP2 exhibited significantly lower cationic demand compared to the DIP1.

PULP AND WHITE WATER

It has been observed that Tower 2 exhibited considerably higher amount of macro-stickies compared to Tower 3 (Trial 1 and 2) and Tower 1 (Trial 3). Tower 1 contained slightly lower content of macro-stickies compared to Tower 3. The fact that Tower 2 showed the highest amount of macro-stickies is in correlation with mean values obtained from macro-stickies measurements from the three towers (Tower 2 =190 st/100g, Tower 1=139/st 100g and Tower 3=90 st/100g). Again, even though the pulp from Tower 1 and Tower 2 both originate from DIP1 (bleached office waste) Tower 2 has generally lower quality and therefore higher amount of macro-stickies in Tower 2 compared to Tower 1 is not surprising. Though it must be kept in mind that the quality from DIP1 can vary a lot but if the system works evenly for a longer time the pulps in Tower 1 and Tower 2 respectively can have almost the same quality).

Results obtained by using DCM-extraction exhibited a different trend. Tower 1 and Tower 2 showed considerably lower values compared to Tower 3. Same is the case for values obtained by UV-VIS absorption but for this method the difference was not as significant. Adsorption and dyeing method indicated a higher amount of stickies in Tower 3 compared to Tower 2 during Trial 1 but during Trial 2 it was evaluated as equal to the one in Tower 1.

This could mean that Tower 3 contains large amount of micro-stickies and rather low amount of macro-stickies. However, it must be kept in mind that the quality of the pulp from different towers varies with time.

Correlations between content of detrimental substances in raw material entering deinking line 2 (DIP2) and in Tower 3 have been noticed. Relatively low amount of macro-stickies and high amount of DCM extractives describes both raw material entering DIP2 and Tower 3. Raw material entering DIP2 exhibited low cationic demand compared to the raw material entering DIP1. Similarly Tower 3 showed lower cationic demand compared to the pulp from DIP1 (Tower 1 and Tower 2).

Notably is also that the ash content in Tower 3 is higher than in Tower 1 and 2 (see Figure 10-1) even though the ash content is higher in the pulp coming into DIP1 than into DIP2. The high ash content and DCM values could be explained by the fact that less cleaning stages are used in DIP2,

particularly absence of post-flotation which is claimed to be the most efficient unit operation for removal of micro-stickies.

During Trial 1, 2 and 3 the temperature of white water was almost equal which means that no fresh water has been added. Amount of detrimental substances in the white water determined by adsorption and dyeing method as well as CCT method was lowest during Trial 3 compared to Trial 1 and 2. The cationic demand of the white water exhibited a different trend. The highest cationic demand has been observed during Trial 3 which can be explained by absence of Tower 3 that normally exhibits relatively low values.

PAPER MACHINE 8

Differences in amount of stickies in Middle and Yankee layer of the Machine Chest have been observed during both days. This is mainly due to different pulps which are fed into the respective layers (see Table 10-4, 10-5 and 10-6).

Amount of macro-stickies in Middle and Yankee layer of the Machine Chest was lower during Trial 2 compared to Trial 1 but the amount of DCM extractives was higher during Trial 2 which can be correlated to the high DCM value for Tower 3 during Trial 2. This correlates with the pulp dosage (see Table 10-4), i.e. during Day 1 high amount of macro-stickies in Yankee layer was determined by 100% of Tower 2. Slightly lower macro-stickies content in Middle layer was due to 54,6 % admixture of Tower 3 which exhibited lower values compared to Tower 2.

Table 10-4 Pulp distribution in Machine Chest Middle, Yankee and Hood layer during Trial 1 when running Toilet paper including Tower 2 and 3. The total pulp dosage fed to the paper machine is presented to the right.

Trial 1: Day 1 Toilet (T2,T3)				
Pulp Source	Middle	Yankee	Hood	Tot. Pulp Dosage
Tower 2	45,5%	100%	100%	81,6%
Tower 3	54,6%	0%	0%	18,4%

Similarly during Trial 2 lower macro-stickies content in the Middle layer can be justified by 60% admixture of the Tower 3 which exhibited lower values than Tower 1. Yankee layer exhibited same macro-stickies value as Tower 1 since it was the only pulp which was run in this layer.

Table 10-5 Pulp distribution in Machine Chest Middle, Yankee and Hood layer during Trial 2 when running Toilet paper including Tower 1 and 3. The total pulp dosage fed to the machine is presented to the right.

Trial 2: Day 2 AM Toilet (T1,T3)				
Pulp Source	Middle	Yankee	Hood	Tot. Pulp Dosage
Tower 1	40,0%	100%	100%	79,8%
Tower 3	60,0%	0%	0%	20,2%

Thus, in this case it can be seen that no effect on macro-stickies content can be observed when mixing different pulps in Yankee and Middle layer, since these values correspond to the ones for

the individual pulps taking into account their percentage in respective layer. Same is the case for UV-VIS and DCM extraction.

However, during Trial 3 the amount of macro-stickies in the Yankee layer was surprisingly high compared to the values obtained from Tower 1 which was the only pulp run on the paper machine (see Table 10-6). Why this increase is obtained cannot really be explained. Reasonably the Middle and Yankee layer should exhibit the same amount of macro-stickies. The DCM results correlated in Machine chest and Tower 1, though. Same is the case for results obtained by adsorption and dyeing method as well as ash content and UV-VIS.

Table 10-6 Pulp distribution in Machine Chest Middle, Yankee and Hood layer during Trial 3 when running Toilet paper including Tower 1. The total pulp dosage fed to the paper machine is presented to the right.

Trial 3 Day 2 PM Toilet (T1)				
Pulp Source	Middle	Yankee	Hood	Tot. Pulp Dosage
Tower 1	100%	100%	100%	79,8%

Only slight differences between Trial 1 and Trial 2 could be observed for the Wire pit PM8. The CCT method showed slightly higher values during Trial 2 compared to Trial 1 whereas adsorption and dyeing method indicated equal values during both days. UV-VIS value was somewhat lower during Trial 2. Considerably lower CCT values have been observed during Trial 3 compared to Trial 1 and 2 but adsorption and dyeing method as well as UV-VIS absorbance did not indicate significant dissimilarities. Cationic demand was however higher during Trial 3 compared to Trial 1 and 2 which can be explained by absence of the Tower 3 that normally exhibits low values.

Based on results obtained from CCT measurements the amount of detrimental substances in Clear filtrate from Krofta was lowest during Trial 3 and highest during Trial 2 which possibly could be explained by the high DCM extraction value from Tower 3. Unfortunately there are no samples that are analyzed by both the CCT and DCM method but previous surveys performed by BIM Kemi have showed that Tower 3 exhibits far higher CCT values than Tower 2.

Adsorption and dyeing method indicated equal amount of stickies during Trial 1 and Trial 3 and slightly higher level during Trial 2. UV-VIS values during Trial 1 and Trial 2 were similar but slightly higher during Trial 3.

The Krofta sludge contained significantly lower amount of macro-stickies during Trial 2 compared to Trial 1. Opposite trend was observed by DCM extraction method, i.e. amount of DCM extractives in Krofta sludge during Trial 2 was considerably higher. Same is the case for the results obtained using adsorption and dyeing method; however the difference is not as sharp as in the case of DCM extraction. Probably the high DCM values in the Krofta during Trial 3 even in this case can be explained by the high values in Tower 3 run previously during the day.

The macro-stickies content in the sludge was higher during Trial 3 compared to Trial 2 but still lower than Trial 1. Adsorption and dyeing method indicated equal level during Trial 1 and Trial 3 which was evaluated as slightly lower than during Trial 2.

During Stickies survey 3 it was mentioned that amount of macro-stickies in Krofta sludge could be a potential indicator of micro-stickies in the system and it can be seen that the CCT values for the wire pit Trial 1-3 are very high and so are the macro-stickies values for the Krofta sludge.

Notable is the high cationic demand obtained from sampling points after the paper machine, especially the clear filtrate out from the Krofta. This is probably due to addition of CMC after the Machine chest. The even higher value in the clear filtrate could be a result of using bentonite for water treatment, since “bentonite contains primarily smectite clay mineral, characterized by its colloidal size, lamellar crystal shape and high negative charge density” [47].

10.2.3 WET-STRONG QUALITY (DAY 3)

In Table 10-3 the effect of running wet-strength agent (or the effect of running Tower1 combined with Chest 5 and 12 instead of only Tower 1) can be observed.

DIP 1 AND DIP2

During Trial 4 DIP 1 was not in operation and therefore it can be assumed that properties of raw material coming into DIP1 were similar to the ones observed during Day 2.

Even though no pulp from DIP2 was run on the paper machine during Trial 4 raw material samples entering this deinking line were taken in order to examine its properties. It was done since surprisingly low values of macro-stickies were obtained during Trial 2. However, similar trend was observed also during Trial 4, i.e. very low amount of macro-stickies but high amount of stickies indicated by DCM extraction as well as adsorption and dyeing method.

PULP AND WHITE WATER

What can be seen is that during Trial 4 the macro-stickies measurements on Tower 1 showed far higher values than during Trial 3. Chest 12 showed somewhat higher values whilst Chest 5 showed somewhat lower values than Tower 1 during Trial 3.

The amount of stickies in Tower 1 determined by adsorption and dyeing method was slightly lower compared to Trial 3 and considerably lower compared to Trial 2. DCM extraction method exhibited slightly higher values.

Chest 5 contained low amount of macro-stickies and ash content. UV-VIS values were also low and only traces of stickies were observed by adsorption and dyeing method. The amount of DCM extractives was slightly lower than in Tower 1. Since Chest 5 is a virgin pulp and since Tower 1 has showed quite low DCM extraction values in previous measurements these were expected results. Chest 12 contained higher amount of macro-stickies than Chest 5 but lower than Tower 1. (Notable is that Chest 12 exhibited higher value than during Process Survey 3 which shows that the quality in the broke-lines can differ quite much). DCM extraction values were similar to the ones for Tower 1 and adsorption and dyeing method indicated only traces of stickies. UV-VIS values were slightly higher than in Chest 5 but lower than in Tower 1.

During Trial 4 the white water exhibited considerably lower stickies content determined by all methods compared to the first trials. This can be explained by the addition of fresh water since the temperature of white water was much lower during Trial 4 compared to previous trials. The lower value could also be due to better retention caused by the wet-strength agent.

PAPER MACHINE 8

When observing the macro-stickies results from the PM8 system it can be seen that the Machine chest Middle layer and Krofta sludge showed lower values during Trial 4 whilst Machine chest Yankee layer exhibited almost equal level as during Trial 3. While running toilet quality with

Tower 1 and wet-strong quality with Tower 1, Chest 5 and Chest 12 in both cases Yankee layer contained only Tower 1 (see Table 10-8 and Table 10-10). Taking into account that Tower 1 showed significantly higher values during Trial 4 compared to Trial 3, Yankee layer should perhaps contain more macro-stickies during Trial 4. However, it depends on the time delay, i.e. when the pulp actually reaches the paper machine.

The amount of detrimental substances in Wire pit and Clear filtrate determined by CCT and UV-VIS was considerably lower during Trial 4 compared to previous trials. Also the cationic demand values were lower. This can perhaps be due to that better retention is obtained in the system due to the wet-strength agent or again addition of fresh water to the white water system.

The adsorption and dyeing measurements showed similar values in the PM8 system. Slight decreases could be observed in the system compared to Trial 3.

Table 10-7 Pulp distribution in Machine Chest Middle, Yankee and Hood layer during Day 3 when running wet-strong paper including Tower 1, Chest 5 and 7. The total pulp dosage fed to the paper machine is presented to the right.

Trial 4: Day 3 Wet-strong paper (T1, Chest 5, Chest 12)				
Pulp Source	Middle	Yankee	Hood	Tot. Pulp Dosage
Tower 1	0%	100%	100%	64,8%
Chest 5	76,4%	0%	0%	26,9%
Chest 12	23,6%	0%	0%	8,3%

During the survey in may no clear correlations between cationic demand and amount of detrimental substances could be observed. From one point of view a high cationic demand indicates high amount of disturbing substances in form of anionic trash. The system has a lower cationic demand while running wet-strength agent which most often contributes to a cleaner system. Though in both cases of Process Survey 3 and 4 the white water temperature has been lower when running wet-strength agent which indicates addition of fresh water. Further it is again rather problematic to state if it depends on the addition of wet-strength agent or the addition of fresh water. Though there are indications that the system is a bit cleaner due to better retention when running wet-strength agent but it cannot be proved at the moment and needs to be further investigated.

From another point of view the system could be cleaner when cationic demand is higher, since the system seems more contaminated while running tower 3 which give a lower cationic demand in the system. Notable is that the cationic demand is also lower in the beginning of DIP2 compared to DIP1 which means that the phenomenon is most probably caused by the raw material or the procedures performed at the initial processing stages in deinking line which also need further investigation.

10.3 CONCLUSIONS

The pulp coming into DIP2 has very high amount of DCM-extractives whilst the amount of macro-stickies is very low compared to DIP 1. The same trend can be seen in pulp coming out from the deinking lines; Tower 1 and Tower 2 in general exhibit high amount of macro-stickies

but low amount of DCM extractives whilst Tower 3 has a lower macro-stickies content but high DCM values.

Amount of macro-stickies is higher in Tower 2 compared to Tower 1 though no clear effect of Tower 2 on the overall cleanness in the system obtained by the other methods has been observed when comparing with Tower 1.

Tower 3 seems to contribute to higher amount of detrimental substances in the system mainly in terms of micro-stickies. It can be seen that Tower 3 has lower cationic demand than Tower 1 and 2 which contributes to an overall lower cationic demand in the system when Tower 3 is run on paper machine.

The cationic demand value from the Krofta sludge was lowest during Trial 4 which is probably due to addition of cationic wet-strength agent. Whether the lower cationic demand in the system in this case contributes to stickies formation is not known.

There are still questions regarding if the lower values obtained from the samples when running wet-strength agent is due to better retention achieved by using wet-strength agent, pulp of higher quality used for production of wet-strong tissue or addition of the fresh water to the white water system. Thus, further studies are required.

11 FINAL DISCUSSION

The main challenge while studying the stickies-related issue was a lack of appropriate methods for monitoring of stickies. Large part of the methods used does not take into account the tack of stickies and therefore cannot be defined as direct stickies measurement methods. The only method that is well-elaborated and is known to detect tacky particles is hand-sheet inspection method in which the stickies are observed in microscope. However, the major drawback of this method is inability to determine amount of stickies in process water.

The UV-VIS absorbance method to some extent correlates with other methods, however no clear trends have been observed. In fact, the UV-VIS absorbance method requires experience to be able to interpret the results correctly and judge the eligibility of this method for determination of stickies. However, this method is simple and relatively fast to perform which is of great importance for industrial use.

Adsorption and dyeing method provides an indication of whether sticky particles are present in the system. The main drawback of this method is lack of opportunity to obtain a quantitative value and use it further as statistical data. Moreover, the size range of stickies adsorbing on a surface of the plastic bottle is not certain. Even though it is assumed that this method indicates stickies in micro-range, in some cases larger particles can be determined as well.

The DCM extraction method is claimed to determine the total amount of stickies. It is a standardized and verified method which makes it more reliable in terms of repeatability. Consistent values obtained by this method have also been observed during elaboration of this project. However, it would be interesting to measure micro-stickies by this method in order to observe whether this value would correlate with other micro-stickies determination methods used in this project. This can be done by screening the pulp and use of rejects for determination of macro-stickies whereas the concentration of micro-stickies can be obtained by subtracting the concentration of macro-stickies from the total stickies concentration. It must however be noted that "DCM extraction method does not directly indicate the amount of stickies in the pulp, but it gives a strong indication to the deposit potential" [31].

The CCT method is said to determine the amount of micro-stickies in the system or, as mentioned, the number of colloidal particles, which is a more suitable designation. Many of the small particles are probably not stickies but for sure a large part of them can be potential stickies.

To sum up, various methods have been tested and the main problem with stickies is presence in the process in different size ranges which makes measurements of them complicated.

Moreover, the morphology of the stickies is prone to undergo changes, i.e. they may disintegrate into smaller particles or agglomerate forming macro-stickies. Therefore, reliable methods for determination of stickies in all size ranges are crucial in order to understand the underlying processes contributing to deposition phenomenon. Consequently, it is of great importance to adopt a credible method for determination of micro-stickies at SCA Edet mill.

Very few duplicates have been performed due to restrictions in time and resources, this should be performed in order to evaluate the method.

12 FINAL CONCLUSIONS

HISTORICAL DATA

Results obtained from analysis of historical data showed that cleaning of wires and felts has been performed more often while running toilet paper compared to wet-strong paper on PM8. The mixture containing pulps from Tower 2, Tower 3 and Chest 7 is the toilet paper quality that is the most prone to contribute to the cleaning occasions.

PROCESS SURVEYS

There are significant variations in critical process parameters, such as temperature, pH, cationic demand and UV-VIS absorbance. pH is an important parameter that needs to be controlled and the impact of pH on the stickies-related problem at SCA needs to be further investigated.

The general trend is that pulps from DIP1 (Tower 1 and Tower 2) contain higher amount of macro-stickies compared to Tower 3 (DIP2). It has been concluded that Tower 3 contains large amount of detrimental substances, mainly micro- or potential-stickies which is based on high amount of DCM extractives, CCT objects in this pulp as well as results from adsorption and dyeing method.

Tower 3 showed a lower cationic demand compared to pulps from DIP1 and it contributes to decrease in cationic demand in the system when running this pulp on the paper machine.

The Krofta is affected by the different qualities and a remarkably high cationic demand is observed in the clear filtrate. The issues regarding the Krofta need to be further investigated.

Lower amount of detrimental substances have been indicated in the system while running wet-strong paper compared to the toilet paper. It is, however, not clear whether this phenomenon occurs due to higher quality pulp used for production of wet-strong qualities, increased retention due to wet-strong additive or due to addition of fresh water.

Chemicals used in papermaking have a large influence on cationic demand in the system. Process additives should be chosen with a great care and possible interactions must be examined before introducing them into the process.

The reliability of methods used for monitoring stickies is still a problematic issue and further examination is needed.

To sum up, the information obtained within the framework of the project indicated several problematic issues regarding stickies in the process. It must be taken into account that stickies-related issues cover many areas, i.e. design of the process, methods for determination of stickies etc. and it is obvious that many questions still remain unanswered. However, obtained results can serve as a good basis for further work for tackling this problem.

13 Future Perspectives

Large process variations are observed and it would be of interest to see how reduced variations would influence the problem with stickies. Improved control of the white water system and separate systems for the different paper machine could also be interesting future work at the mill. One important issue is that very few online meters are used at SCA Edet mill which makes it hard to monitor the process and implement necessary changes before the problem escalates. It would be interesting to try to correlate online measurements with the stickies problem.

A stable pH is important and further investigations are of interest. What is a stable pH? How large variations are accepted and how do they influence the system? One important aspect is the CaCO₃ issue mentioned.

It would also be of interest to study the effect of Tower 3 even more. This can be done by not running the pulp from Tower 3 on PM8 for some time. One future outlook could also be to study the cleaning efficiency on DIP2 and how it can be improved to get a cleaner pulp.

The water in the wire pit contains a high level of detrimental substances which may contribute to process-related problems, such as deposit formation. It could be of interest to study if it can be reduced by adding retention/fixation chemicals.

Process-related problems can sometimes be hard to track. In order to facilitate tracking process delay calculations could be of interest. This means that information regarding time when the pulp enters the deinking line and reaches different process locations as well as problems that have occurred in deinking plant should be available in order to find a source of the issue as quickly as possible.

So far main focus has been set on tracking stickies in the process and finding a way of monitoring them. In future studies more focus could be set to observing the wires where the actual problem occurs. One interesting future work for the mill is to involve operators which work closely to the paper machine. What happens when the stickies start to form? Why do they clean, is it urgent or preventative? Are there small or large stickies? What quality is run and what pulps are included? When did they change quality? Make some kind of template which is easy to fill in and then it is easier to follow up the cleaning occasions.

The problem with stickies originates from the low quality of the raw material entering the mill in the pulping plant. One interesting aspect could be to study the raw material. How much does the quality vary? Perhaps some kind of sorting system could be introduced.

As mentioned, very few duplicates have been performed so far which is preferable from several points of view. Further examination of the methods is needed. It would also be interesting to see how the measured values vary during the day. Examination of methods could include study of both the variations within the triplicate and also the process variations within the day.

Further examination of micro-stickies methods would be interesting. Compare DCM and the CCT method. A daily method for micro-stickies measurements are needed at SCA. Relevance of method and practical issues has to be taken into consideration.

So far a very broad picture of the problem at the mill is obtained, in future work it could be interesting to focus on smaller parts of the process. As an example the Krofta can be studied. How is it affected by the different qualities? Examine the high cationic demand in the clear filtrate. What trends can be seen? Another possible project could be to study samples before and after the wire (machine chest and wire pit).

More chemical analysis of the stickies should be performed in order to get a broader picture about what the stickies really consist of. How much does the composition vary? For example analysis of the DCM extracts could be performed by using for example FTIR in order to see what components the DCM method actually measures.

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APPENDIX A: PROCESS FLOW SHEETS AND CORRESPONDING SAMPLING POINTS

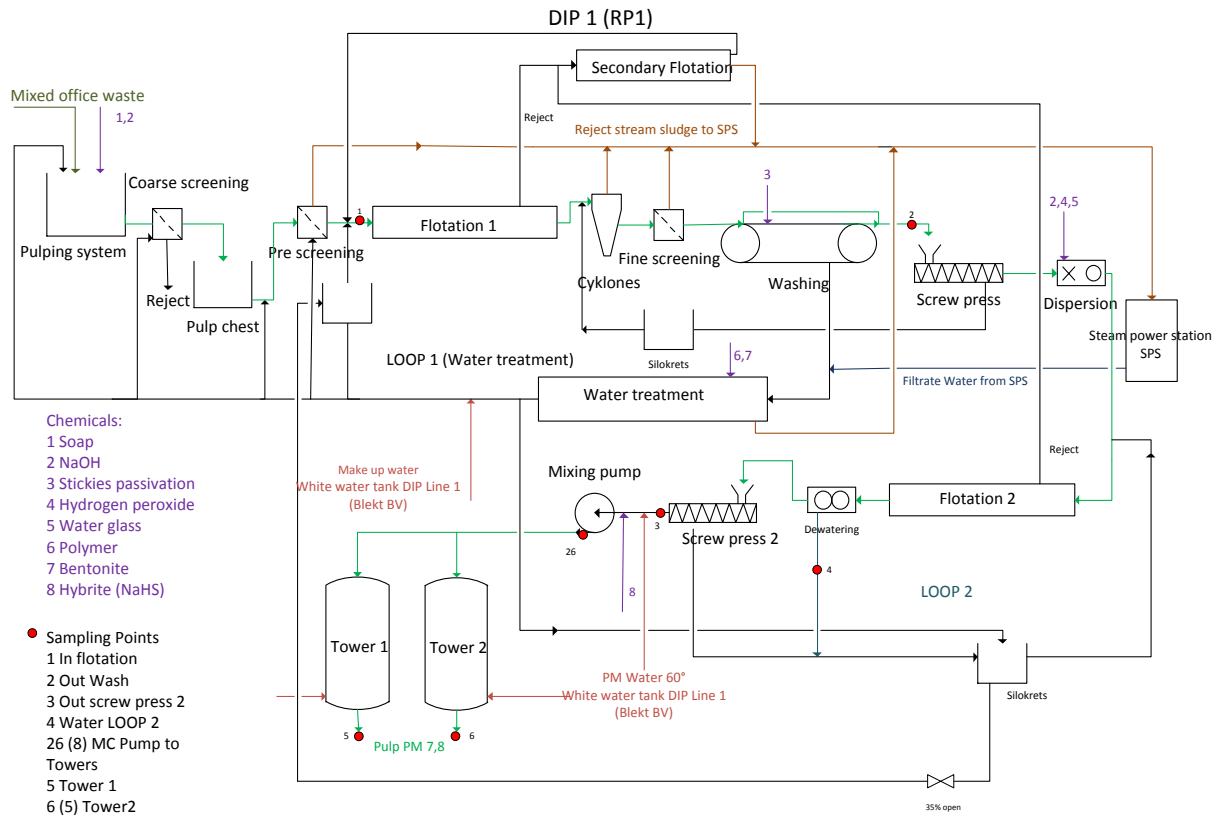


Figure 1 Flow Sheet Deinking line 1 (DIP 1) and corresponding sampling points during stickies survey march 201 2.

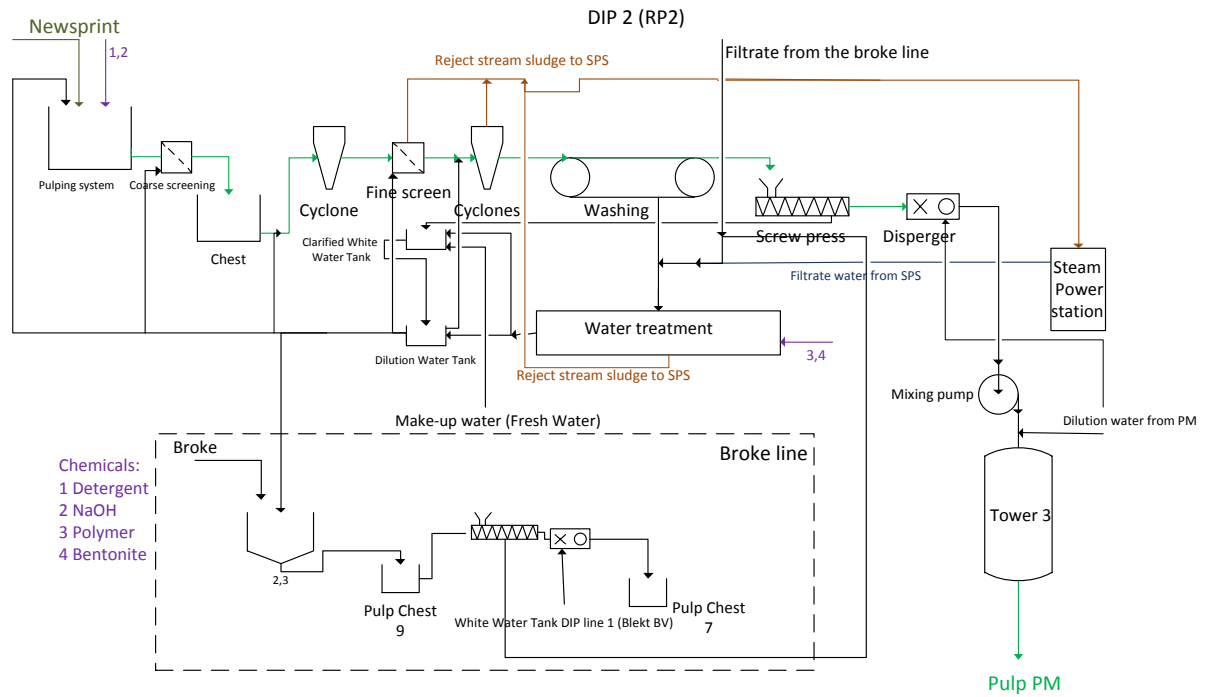


Figure 2 Flow-sheet deinking line 2 (DIP2).

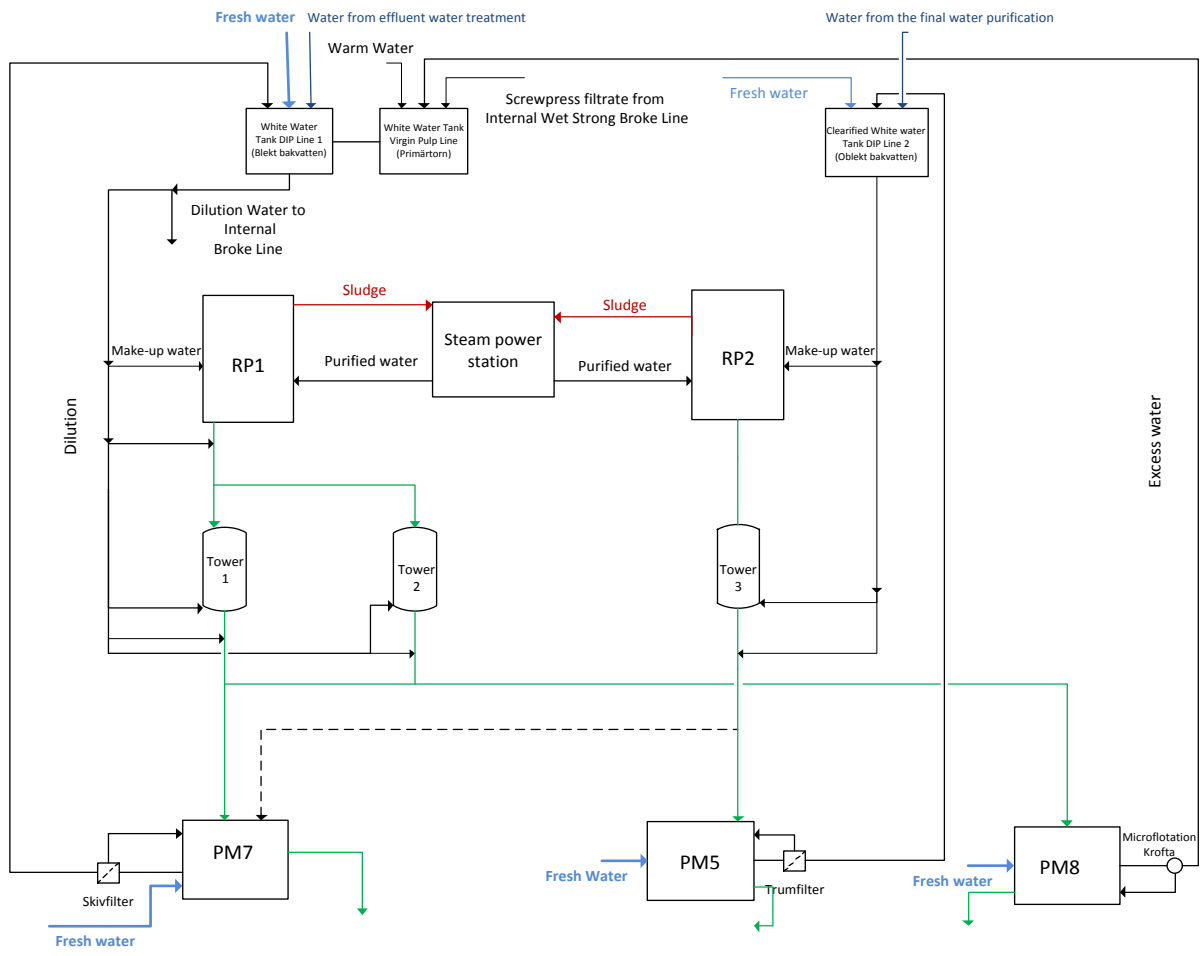


Figure 3 Basic overview of the process water at Edet Mill.

APPENDIX B: RESULTS HISTORICAL DATA 1

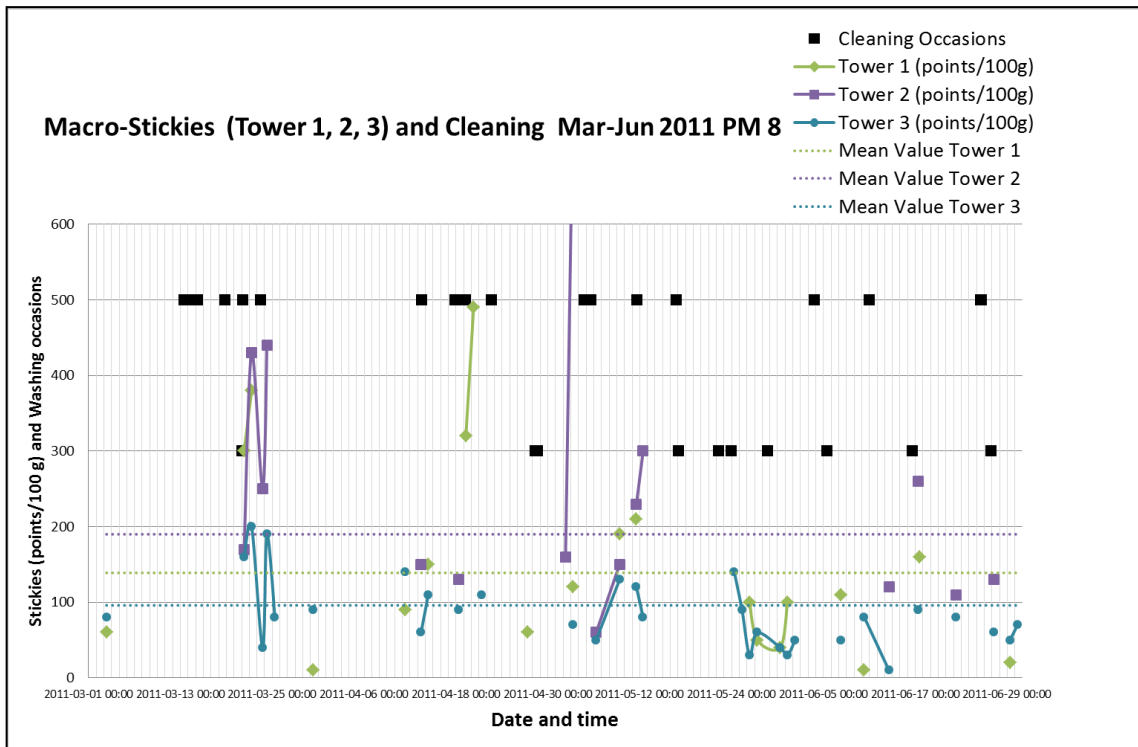


Figure 2 Macro- Stickies measurements (points/100g) for Tower 1,2,3 and Cleaning occasions are plotted for March-June 2011 PM8. The mean values for macro stickies measurements are also present as dotted lines.

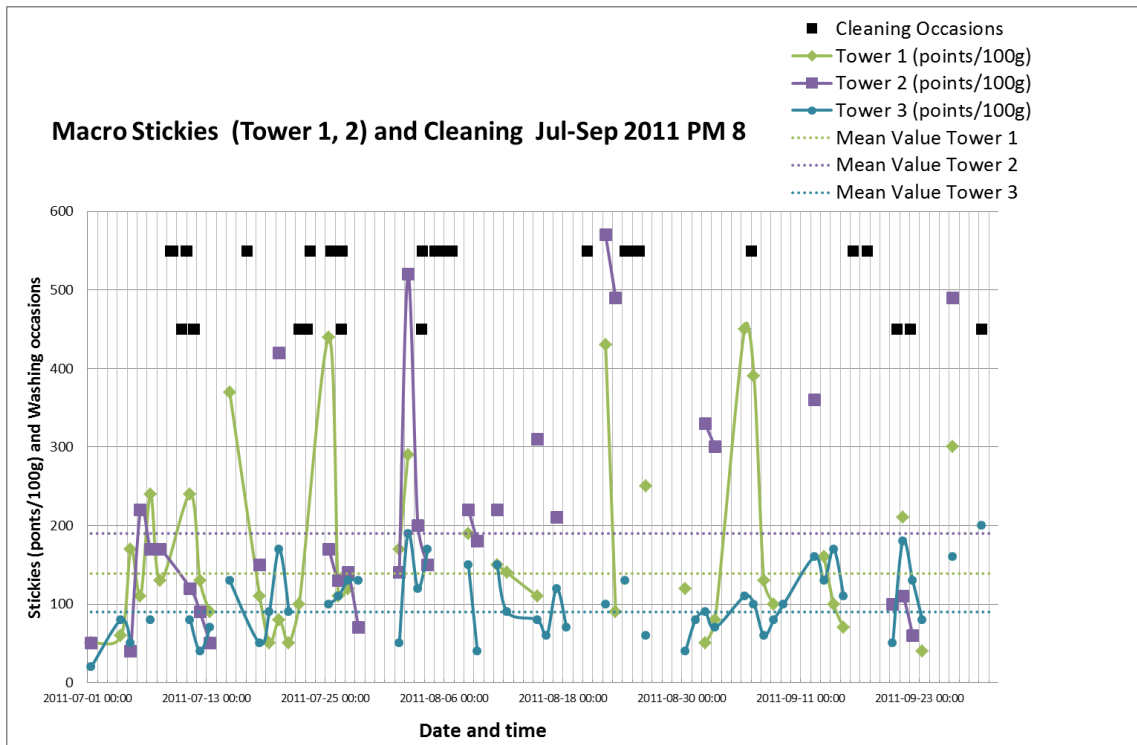


Figure 2 Macro- Stickies measurements (points/100g) for Tower 1,2,3 and Cleaning occasions are plotted for July-September 2011 PM8. The mean values for macro stickies measurements are also present as dotted lines.

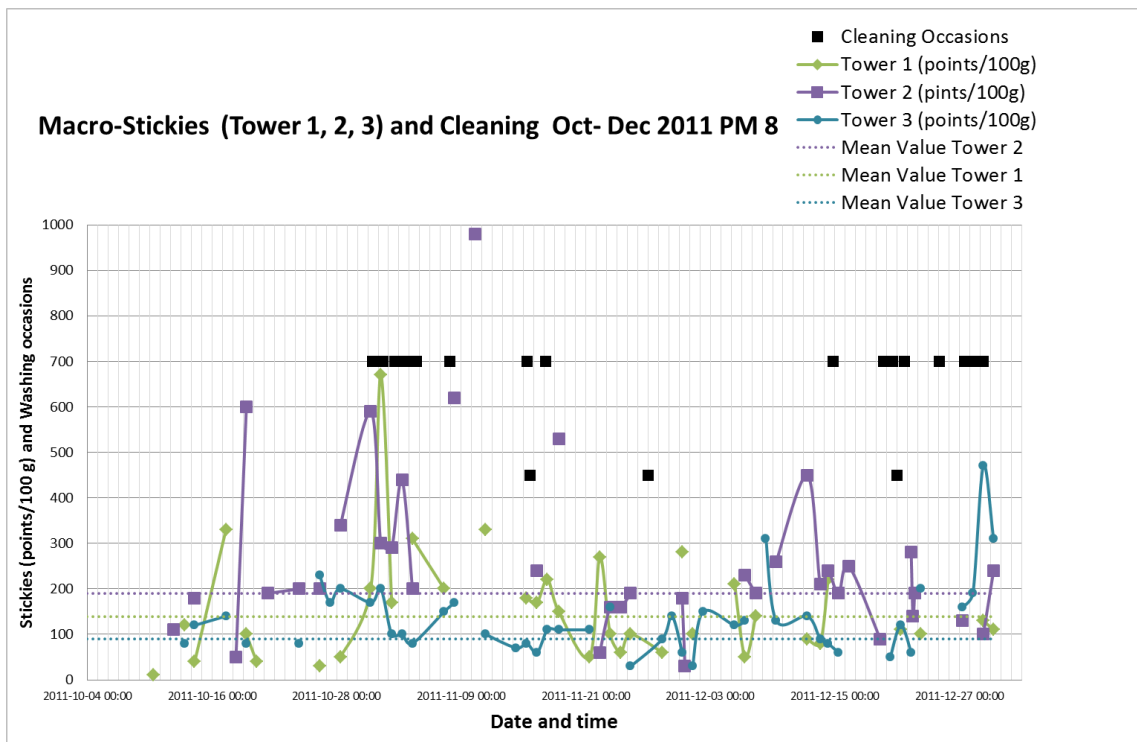


Figure 3 Macro- Stickies measurements (points/100g) for Tower 1,2,3 and Cleaning occasions are plotted for March-June 2011 PM8. The mean values for macro stickies measurements are also present as dotted lines.

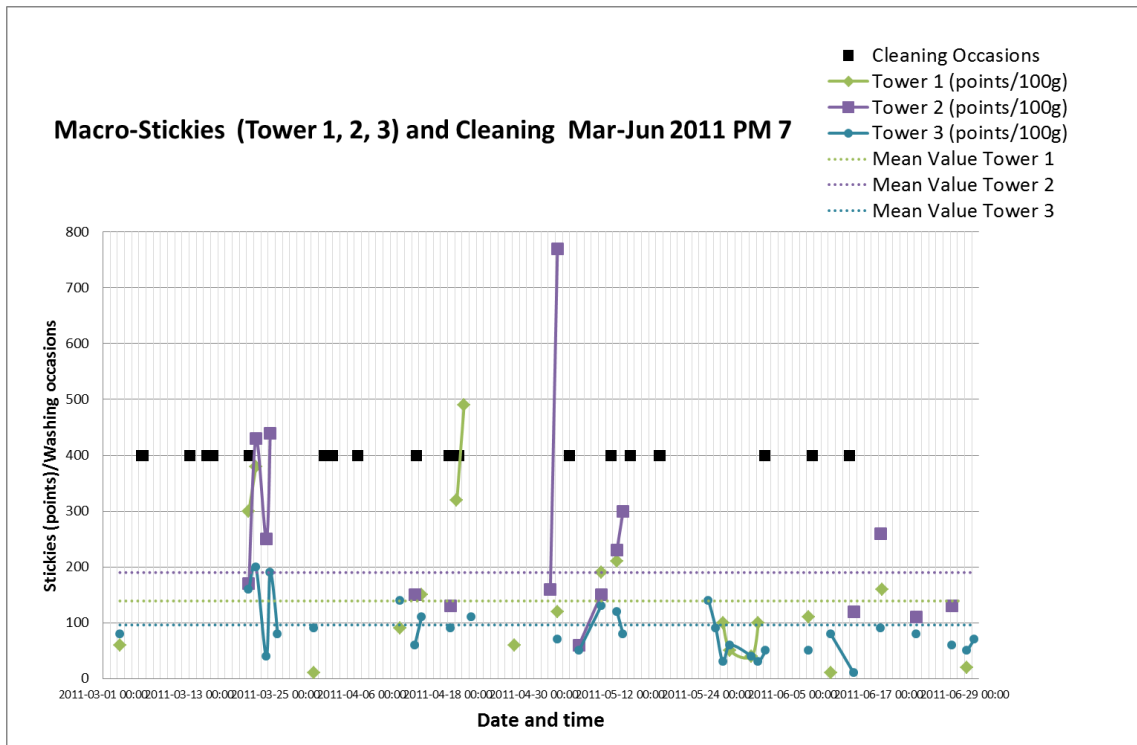


Figure 4 Macro- Stickies measurements (points/100g) for Tower 1,2,3 and Cleaning occasions are plotted for March-June 2011 PM7. The mean values for macro stickies measurements are also present as dotted lines.

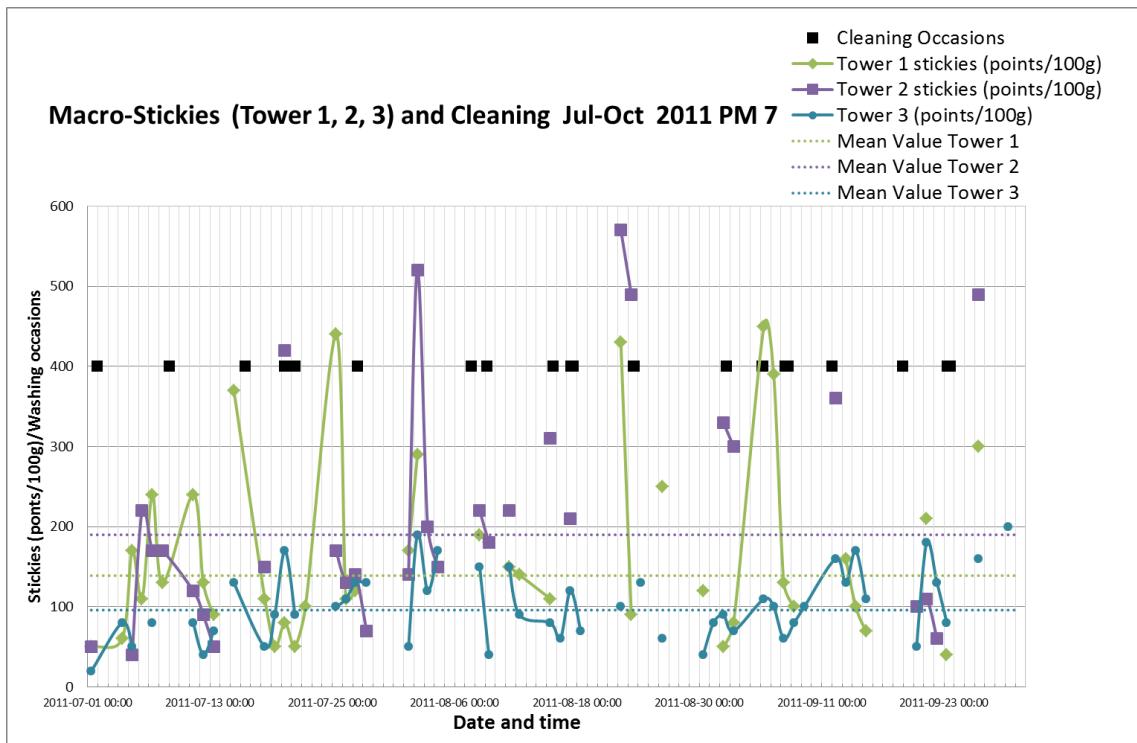


Figure 5 Macro- Stickies measurements (points/100g) for Tower 1,2,3 and Cleaning occasions are plotted for July-September 2011 PM7. The mean values for macro stickies measurements are also present as dotted lines

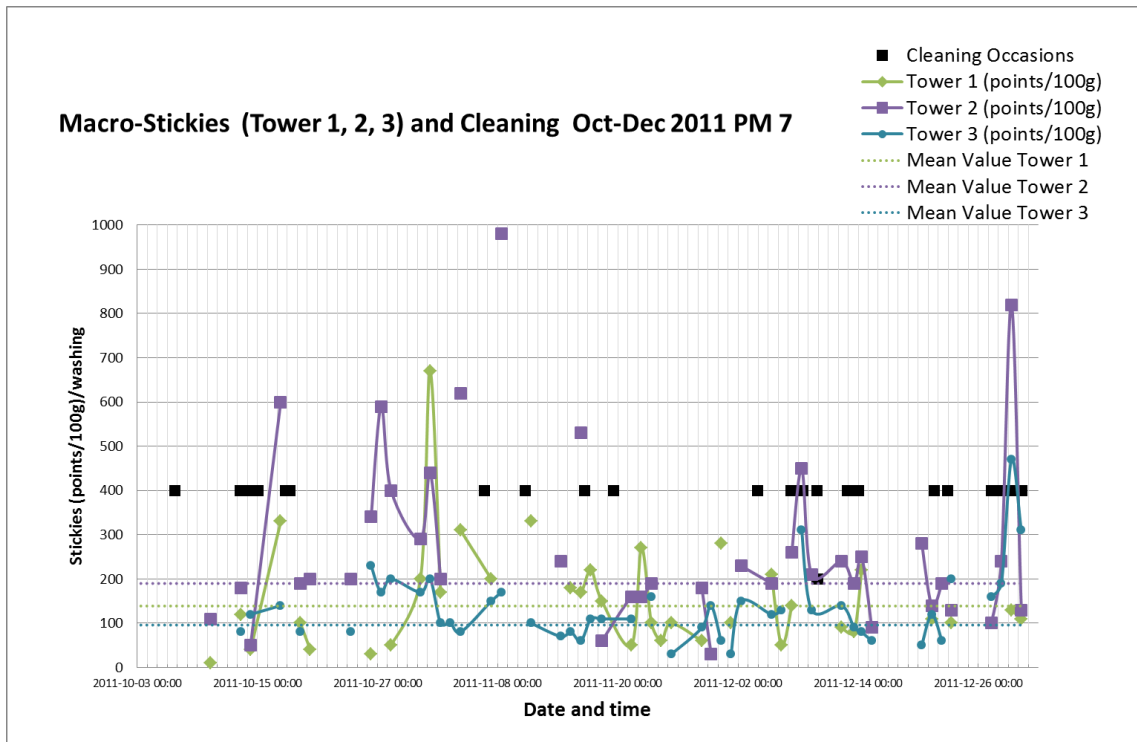
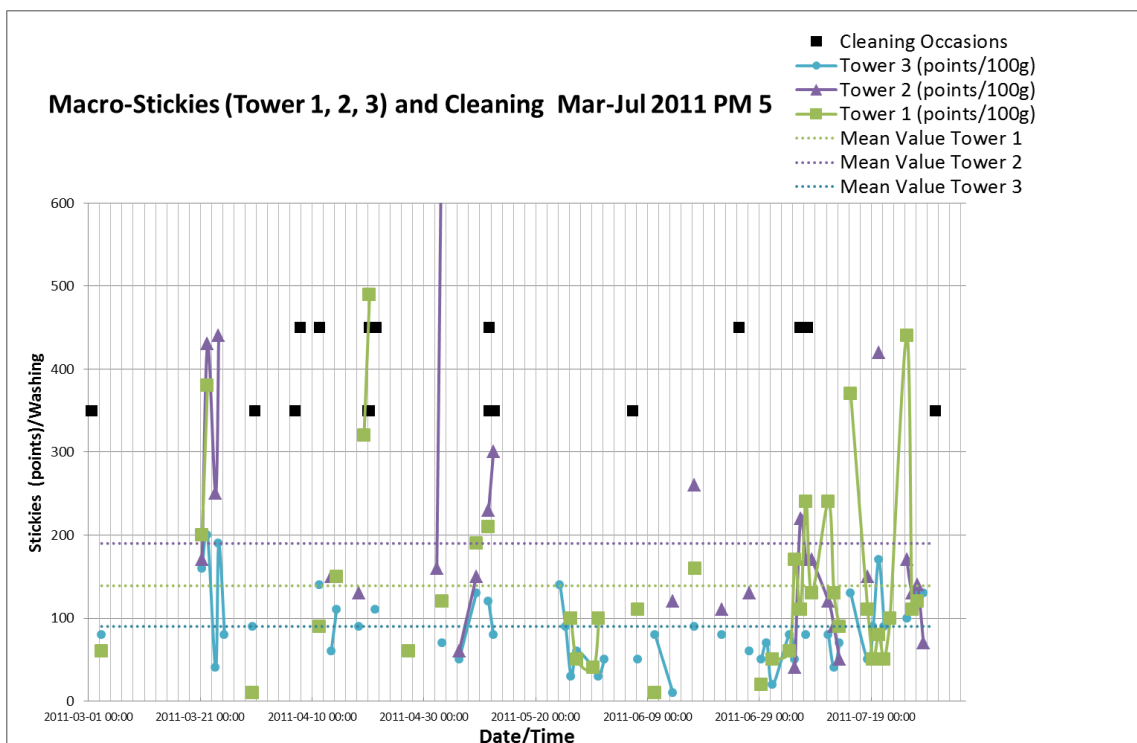


Figure 6 Macro- Stickies measurements (points/100g) for Tower 1,2,3 and Cleaning occasions are plotted for October- December 2011 PM7. The mean values for macro stickies measurements are also present as dotted lines.



Figur 7 Macro- Stickies measurements (points/100g) for Tower 1,2,3 and Cleaning occasions are plotted for March-July 2011 PM5. The mean values for macro stickies measurements are also present as dotted lines.

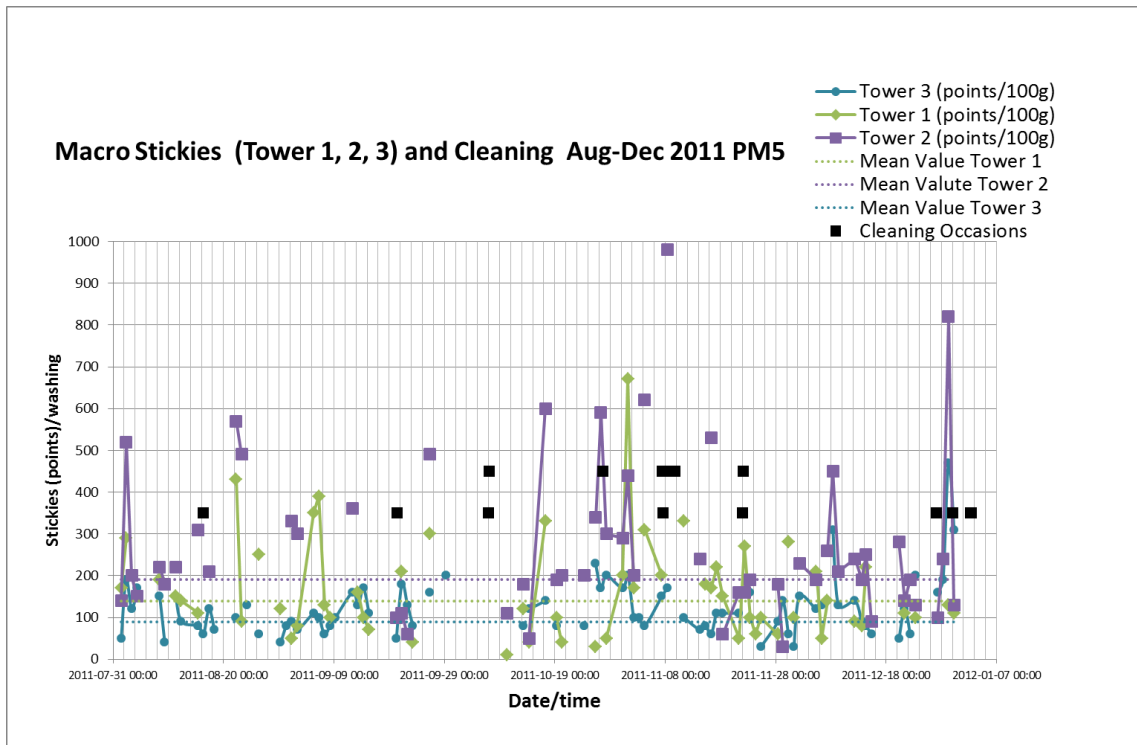


Figure 8 Macro- Stickies measurements (points/100g) for Tower 1,2,3 and Cleaning occasions are plotted for March-July 2011 PM5. The mean values for macro stickies measurements are also present as dotted lines.

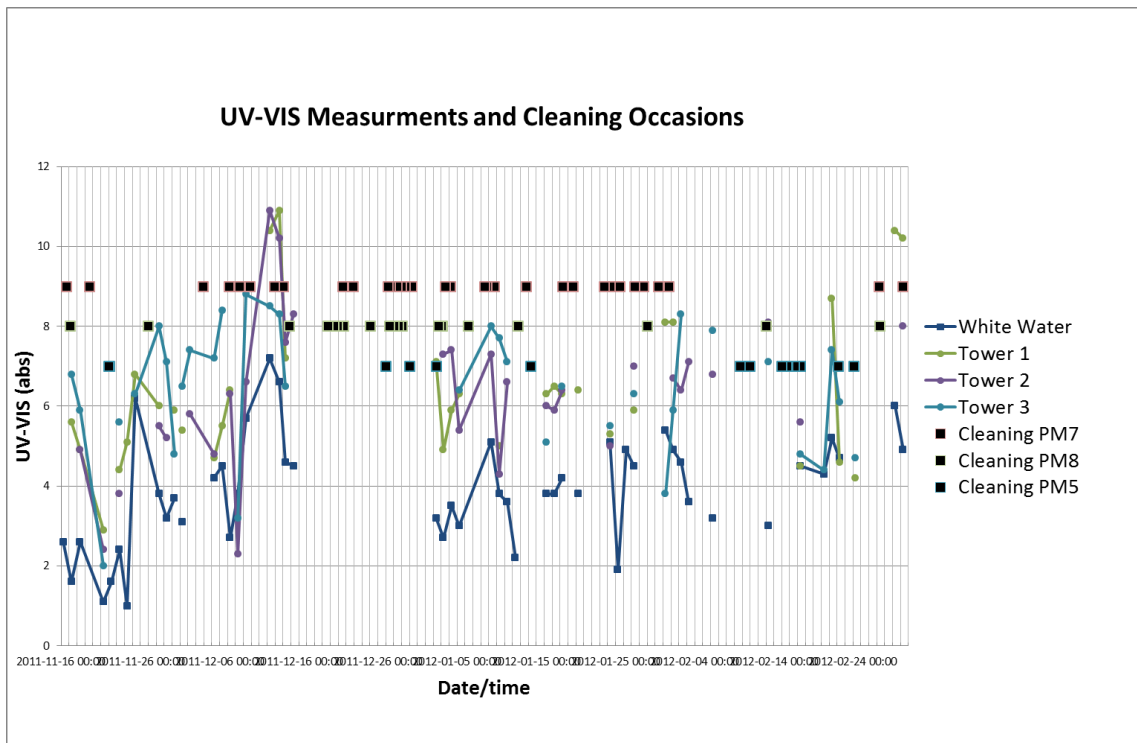


Figure 9 Diagram showing UV-VIS measurements and cleaning occasions on the paper machines.

APPENDIX C: RESULTS HISTORICAL DATA 2 WET-STRENGTH AND PULP DOSAGE

In APPENDIX C wet-strength and Pulp dosage plots for PM8 and PM7 month by month during 2011 are presented in order to observe the influence of pulp dosage (%) on cleaning occasions.

PM8

Pulps used on PM8: Tower 1, Tower 2, Tower 3, Chest 5, Chest 6, Chest 7 and Chest 12.

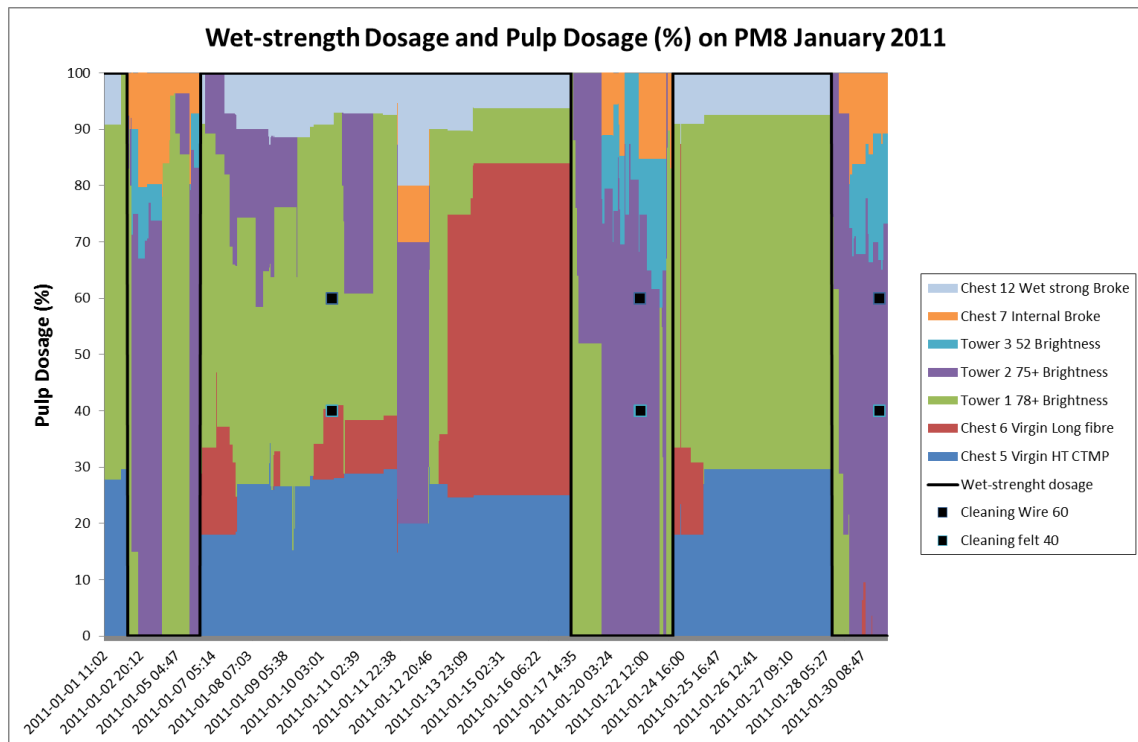


Figure 1 Diagram January 2011 PM8. On the vertical axis the pulp dosage of the individual pulps is given in percent of the total pulp dosage and felt and wire cleanings are presented as black squares at 40 and 60 respectively. Each black mark represents one cleaning occasion. The wet strength dosage is also plotted and when the black line is at 100 it means that wet-strength agent is added to the process, amount of agent added is not taken into account.

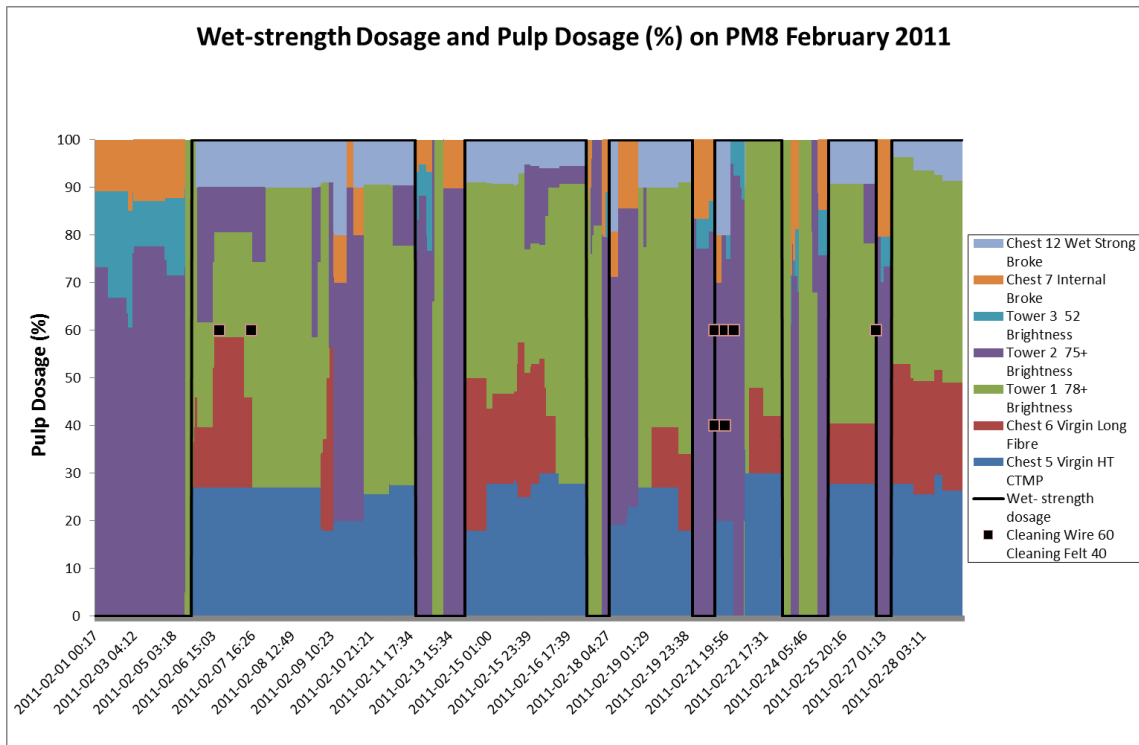


Figure 3 Diagram February 2011 PM8.

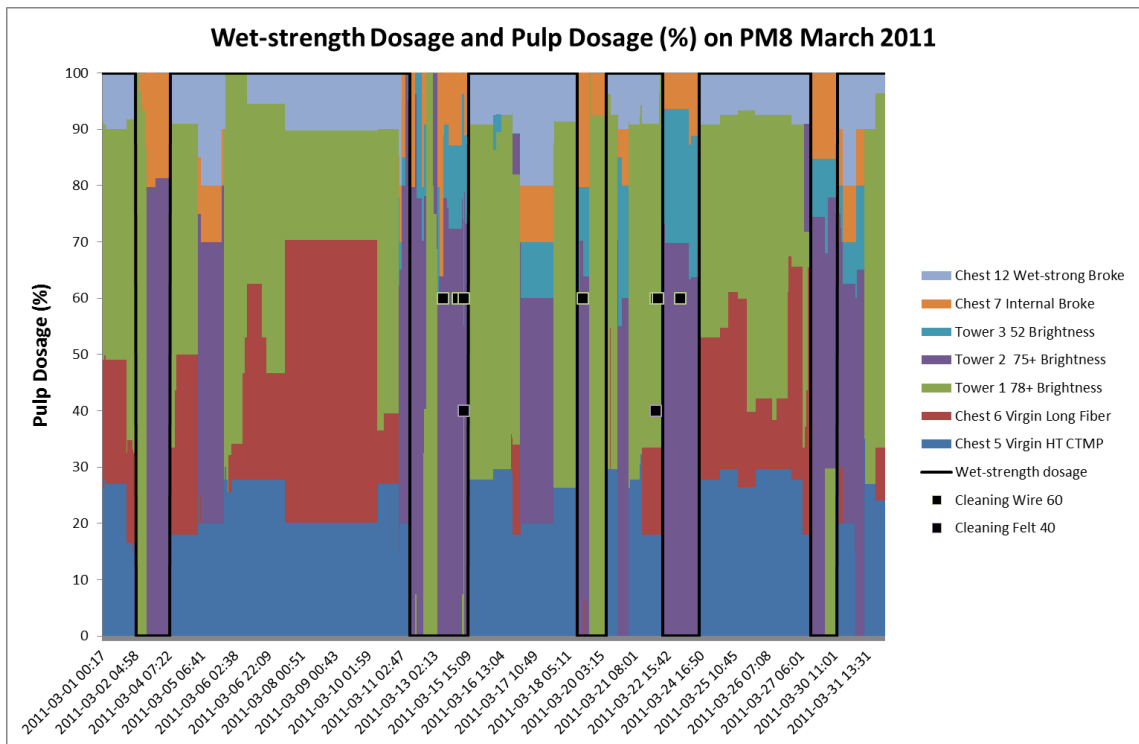


Figure 3 Diagram March 2011 PM8

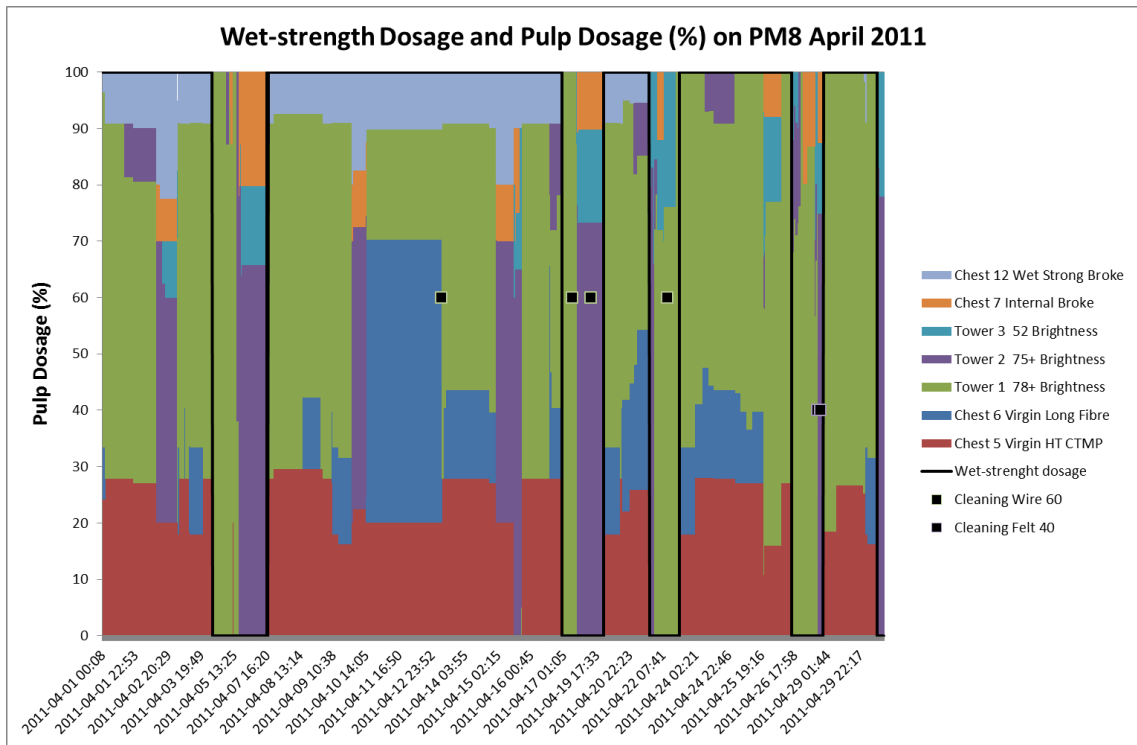


Figure 4 Diagram April 2011 PM8

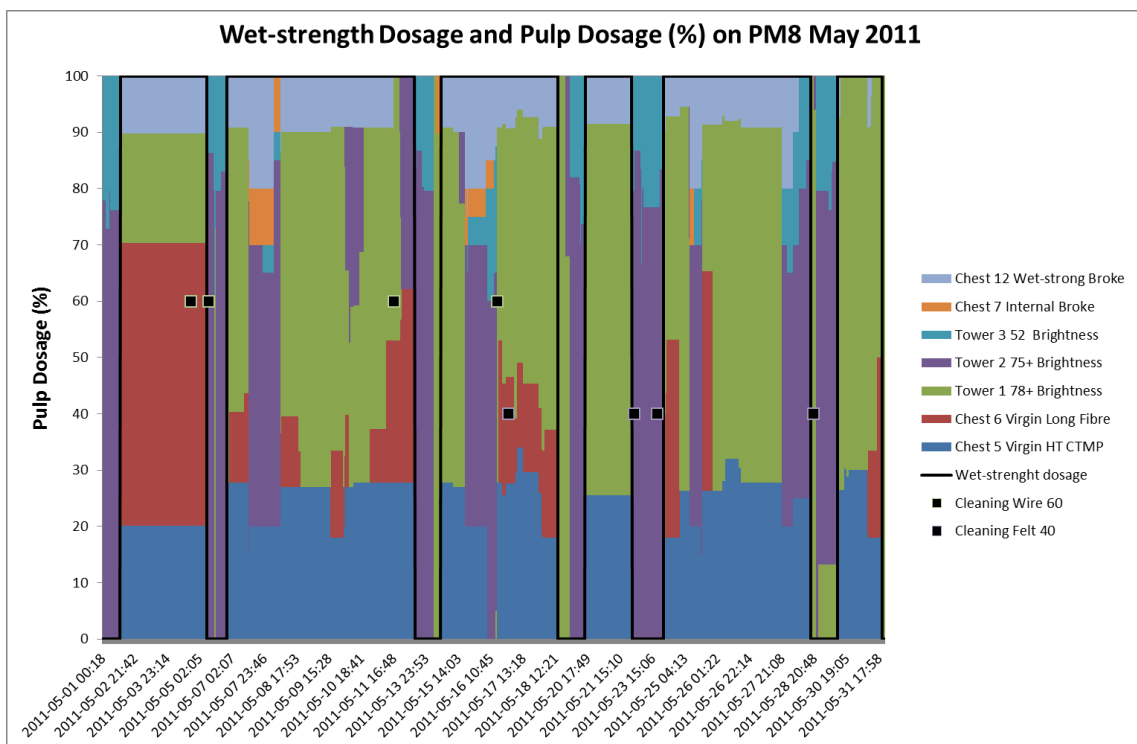


Figure 5 Diagram May 2011 PM8

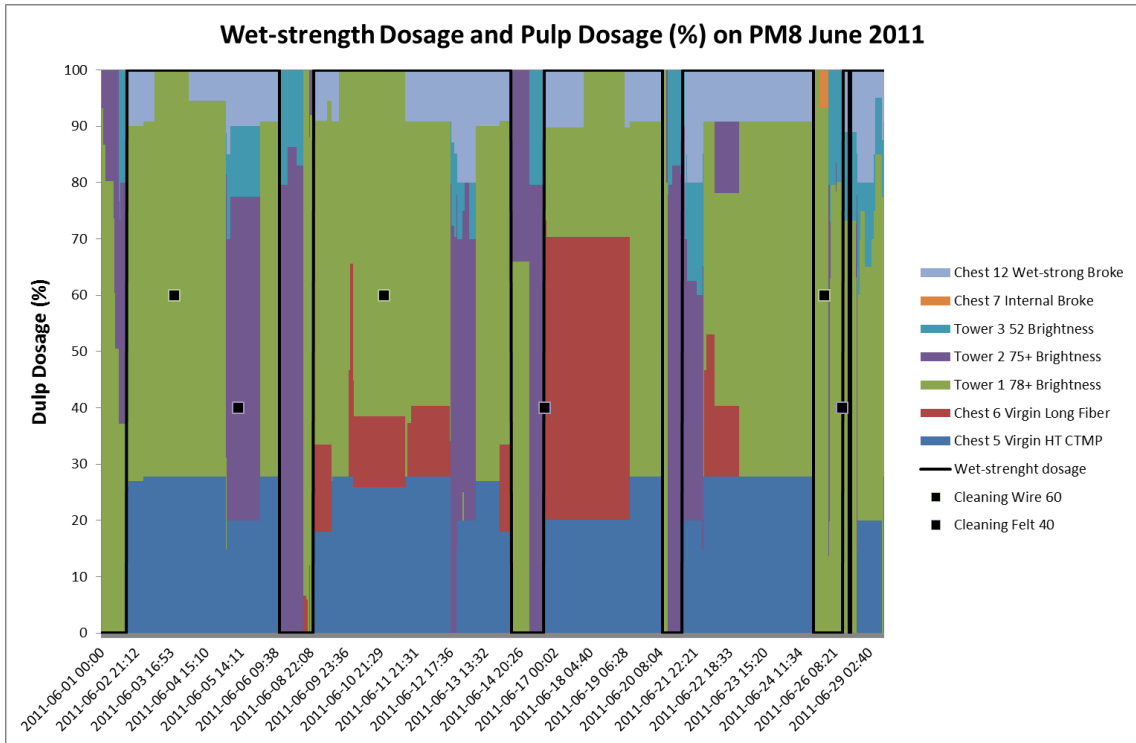


Figure 6 Diagram June 2011 PM8

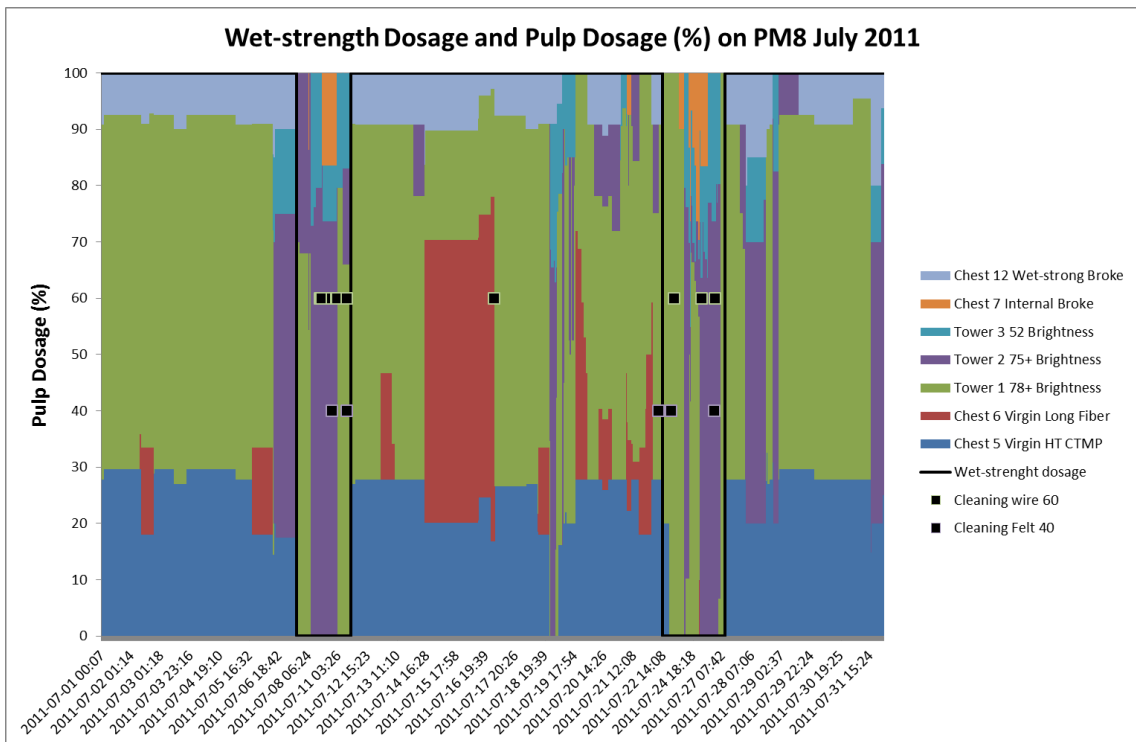


Figure 7 Diagram July 2011 PM8

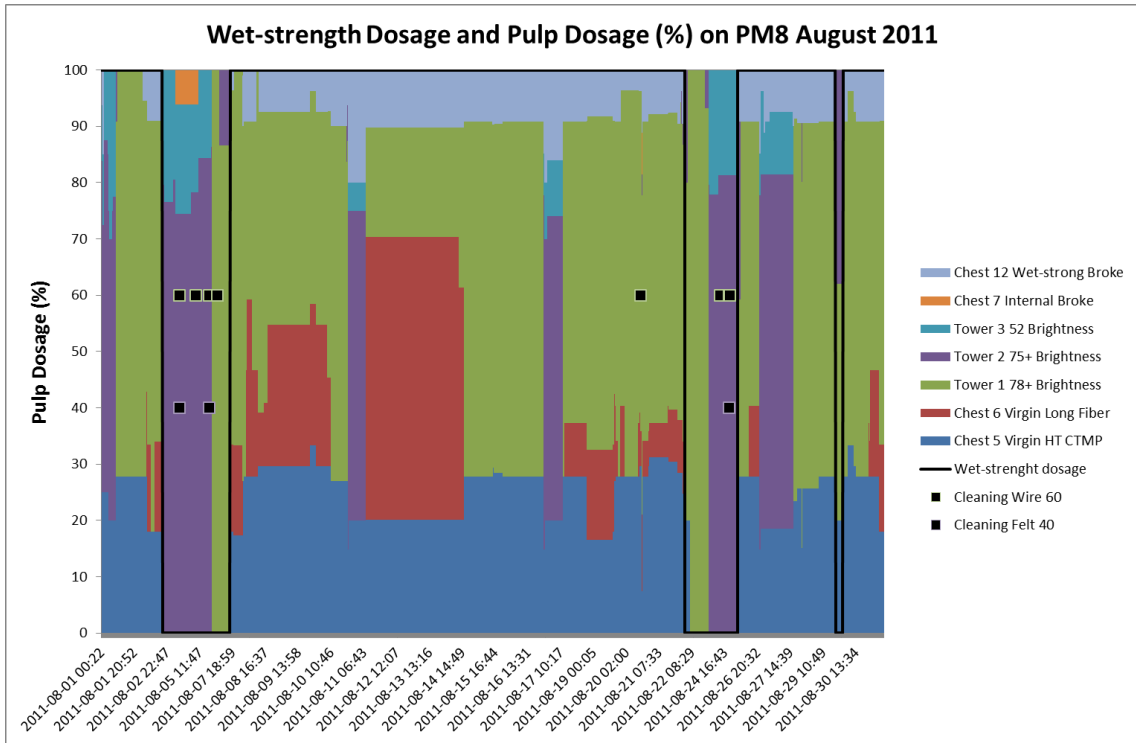


Figure 8 Diagram August 2011 PM8

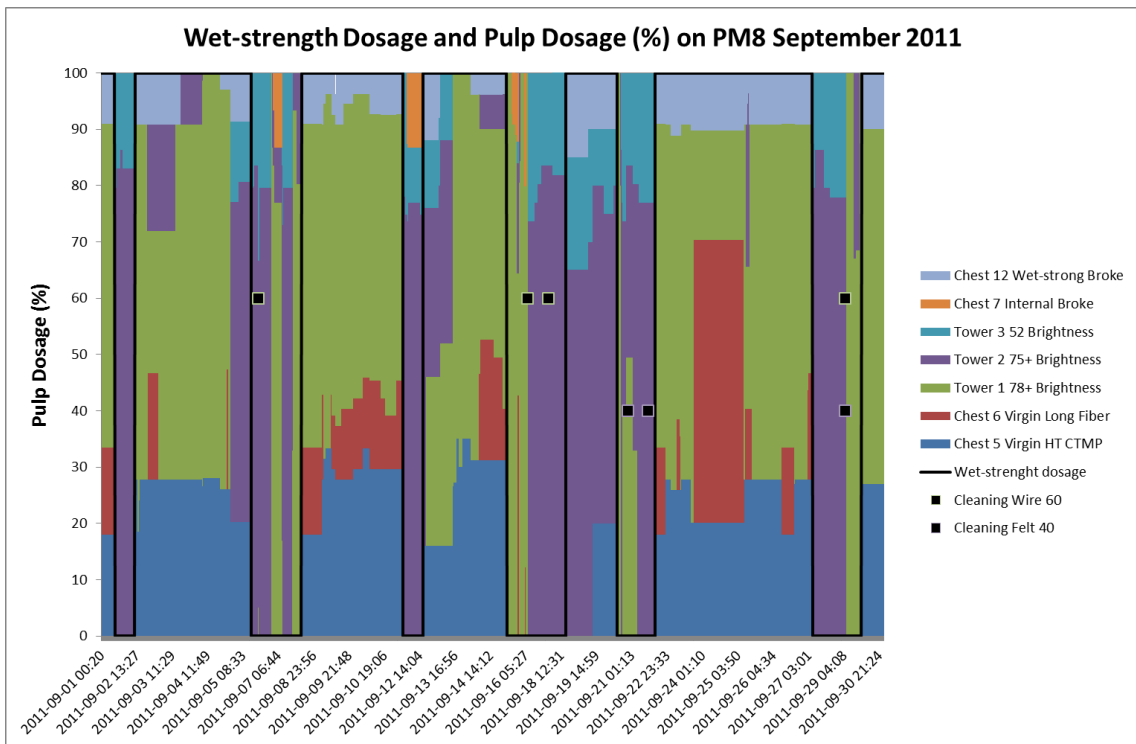


Figure 9 Diagram September 2011 PM8

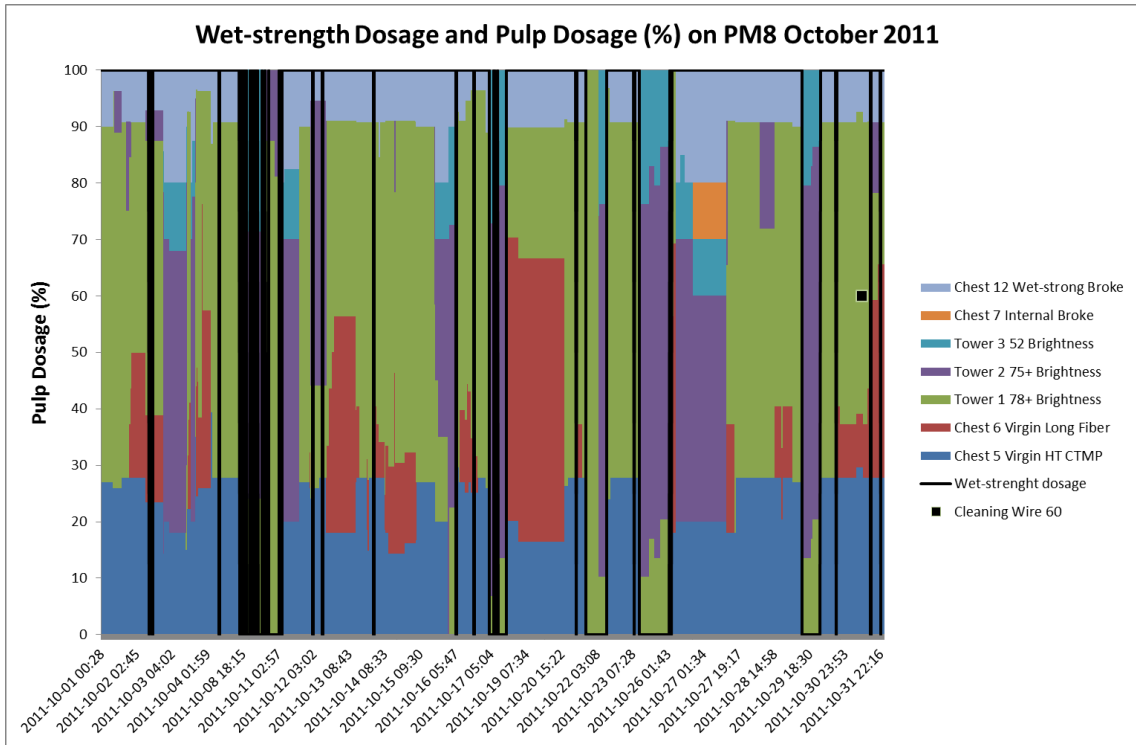


Figure 10 Diagram October 2011 PM8

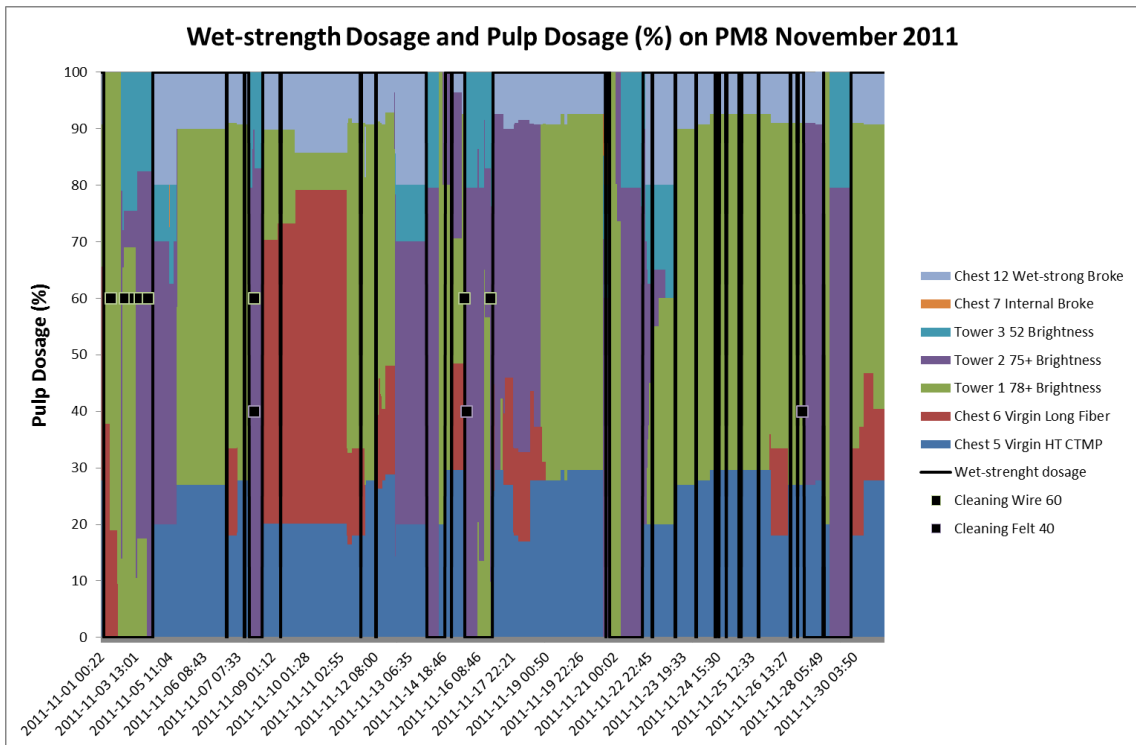


Figure 11 Diagram November 2011 PM8

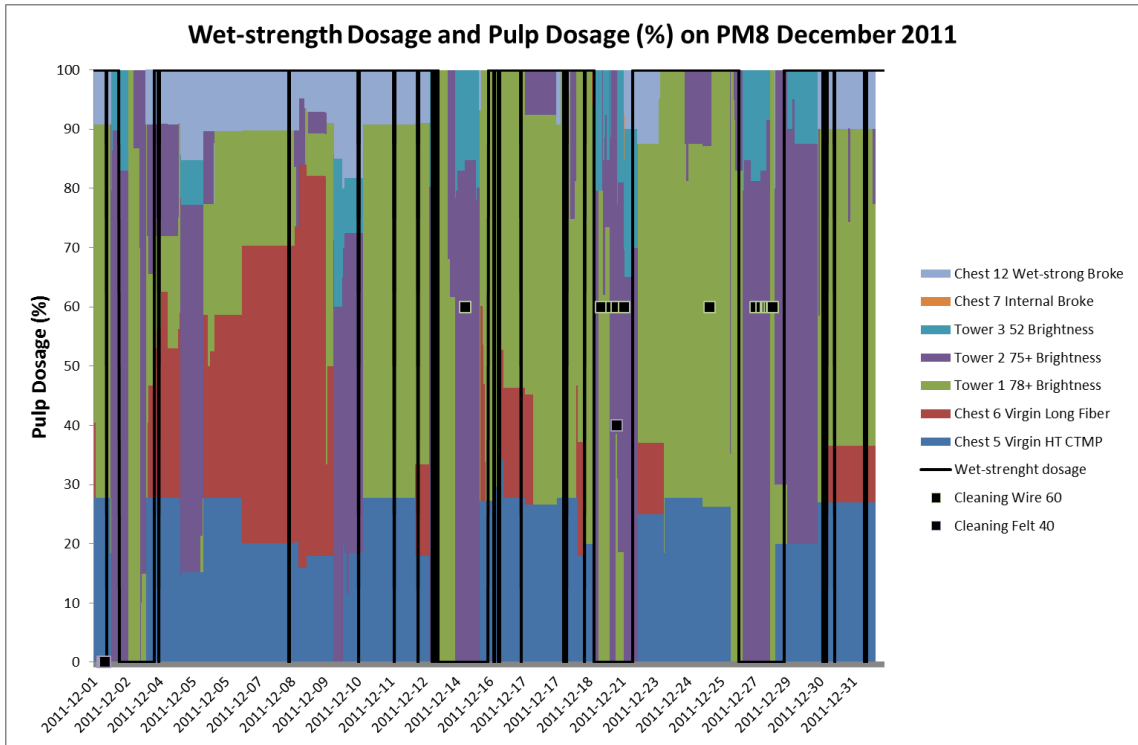


Figure 12 Diagram December 2011 PM8

PM7

Pulps used on PM7: Tower 1, Tower 2, Tower 3, Chest 4, Chest 5, Chest 6, Chest 7 and Chest 8.

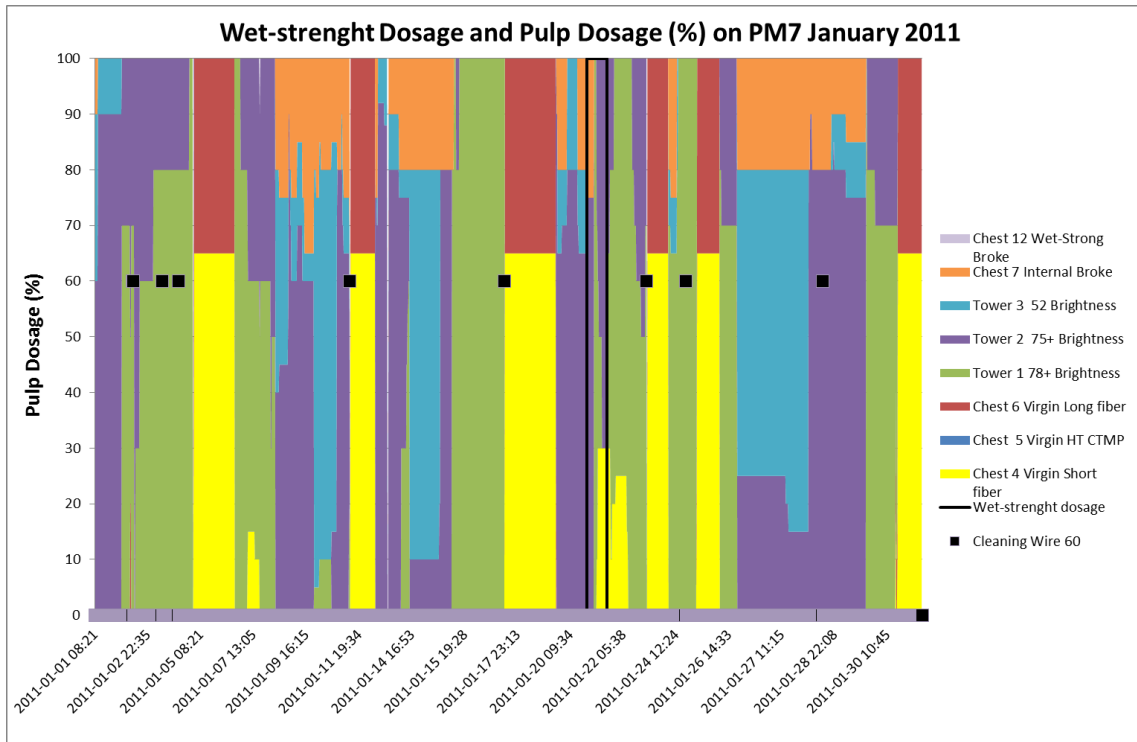


Figure 13 Diagram January 2011 PM7

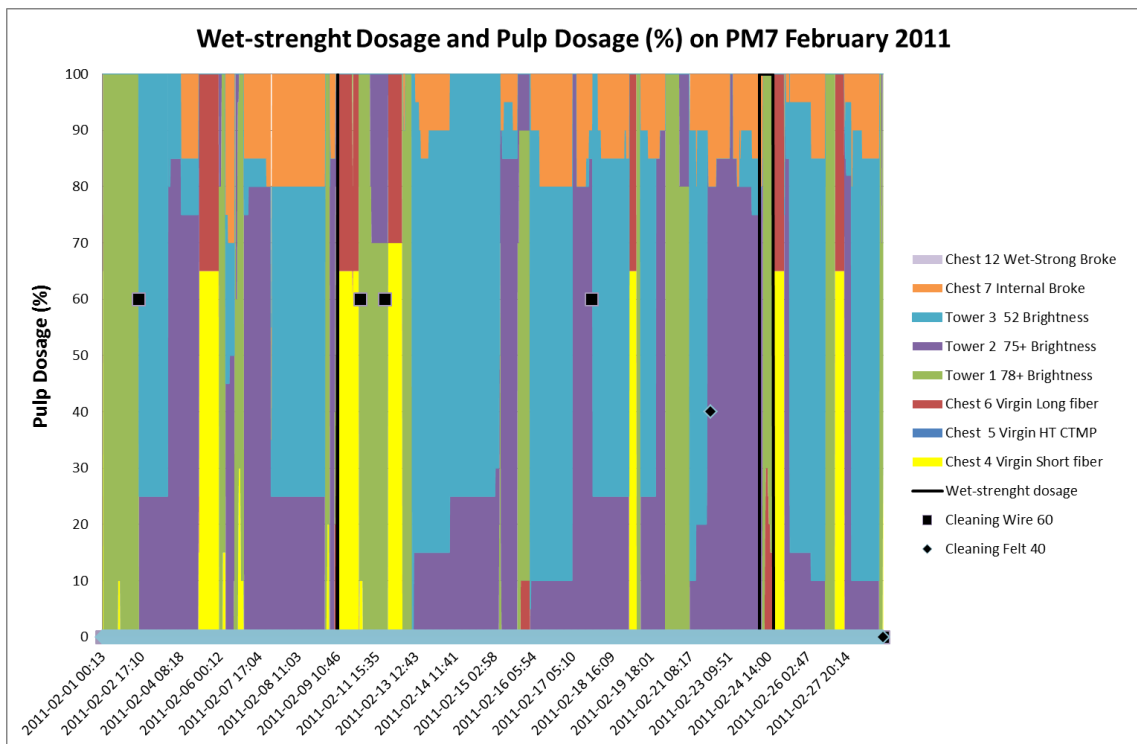


Figure 14 Diagram February 2011 PM7

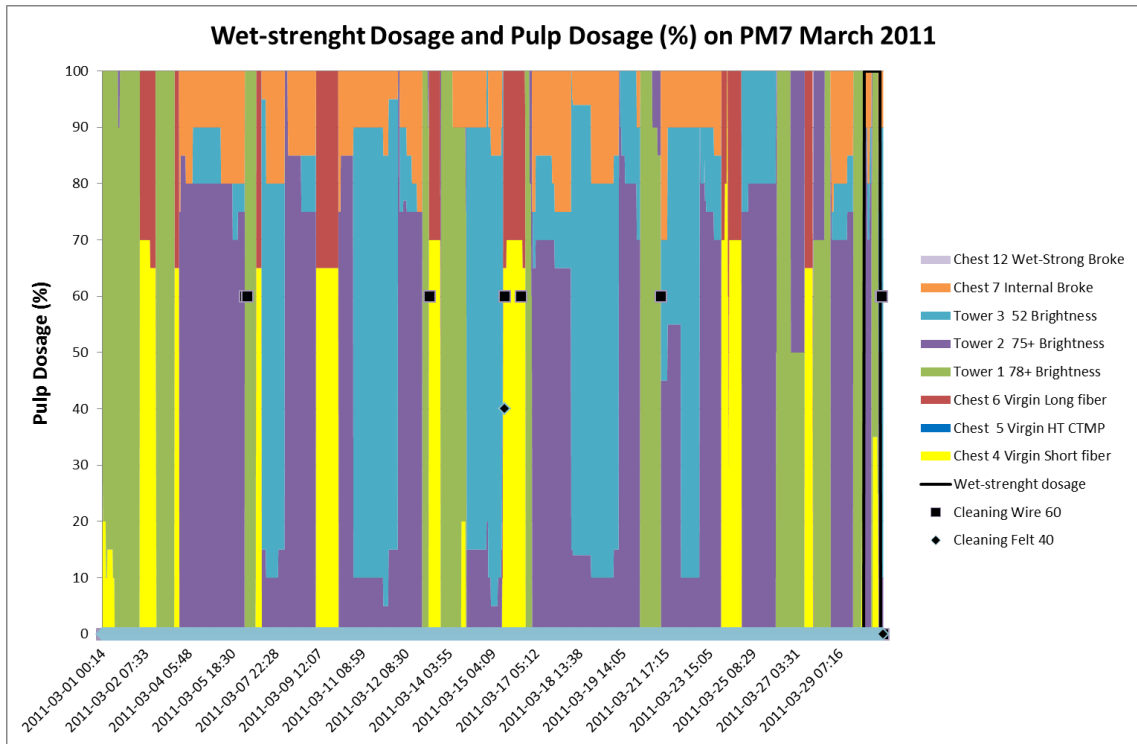


Figure 15 Diagram March 2011 PM7

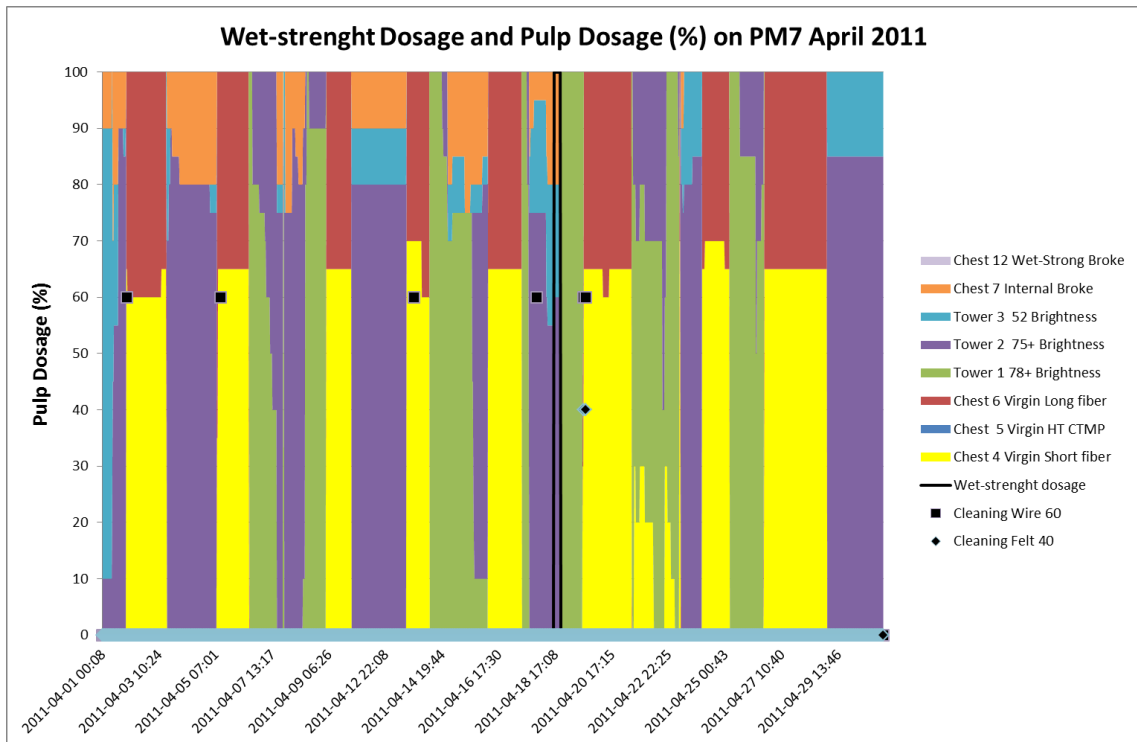


Figure 4 Diagram April 2011 PM7

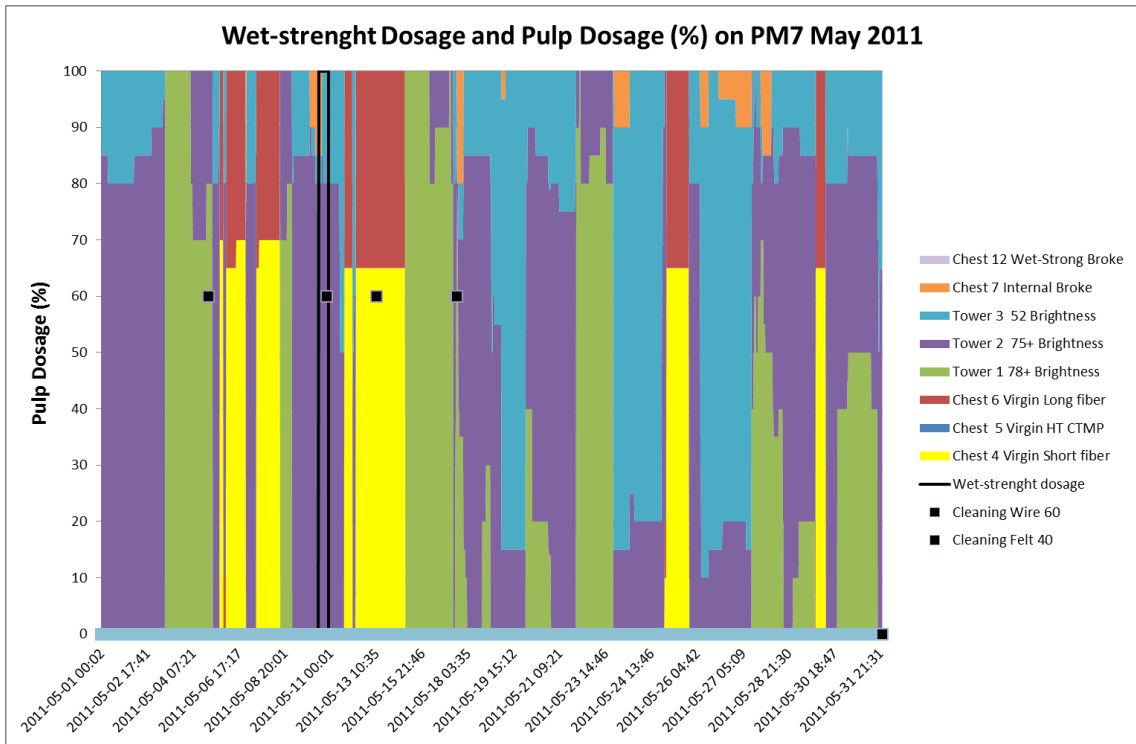


Figure 175 Diagram May 2011 PM7

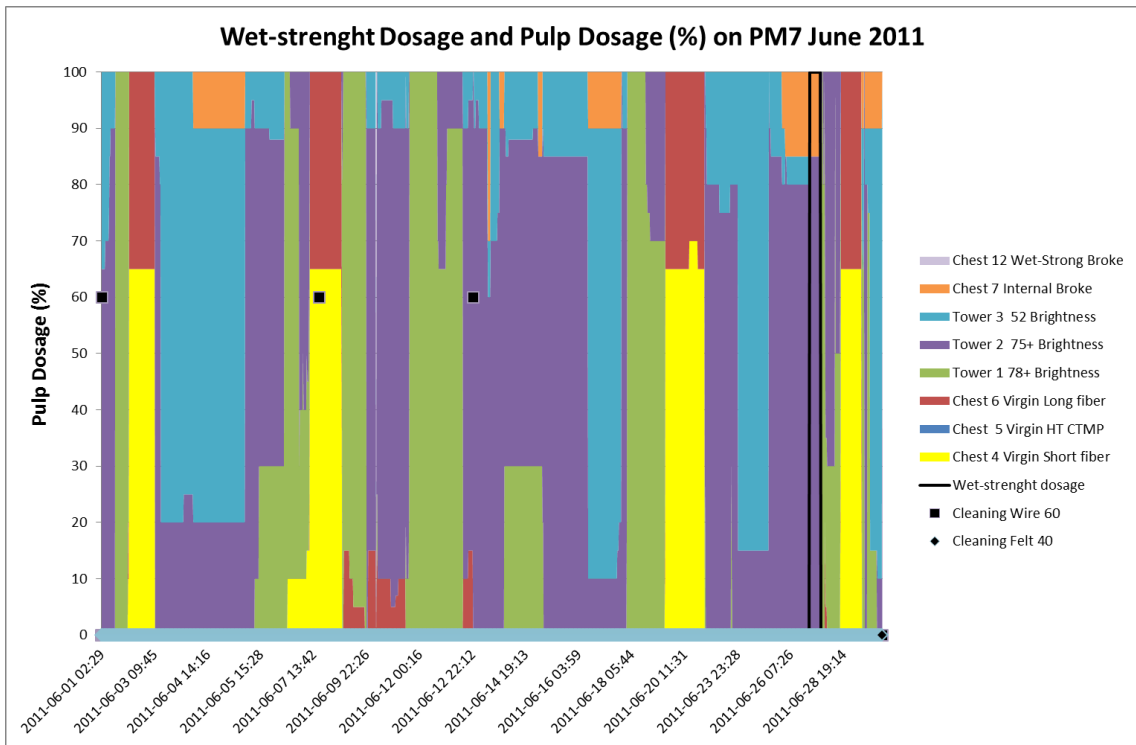


Figure 18 Diagram June 2011 PM7

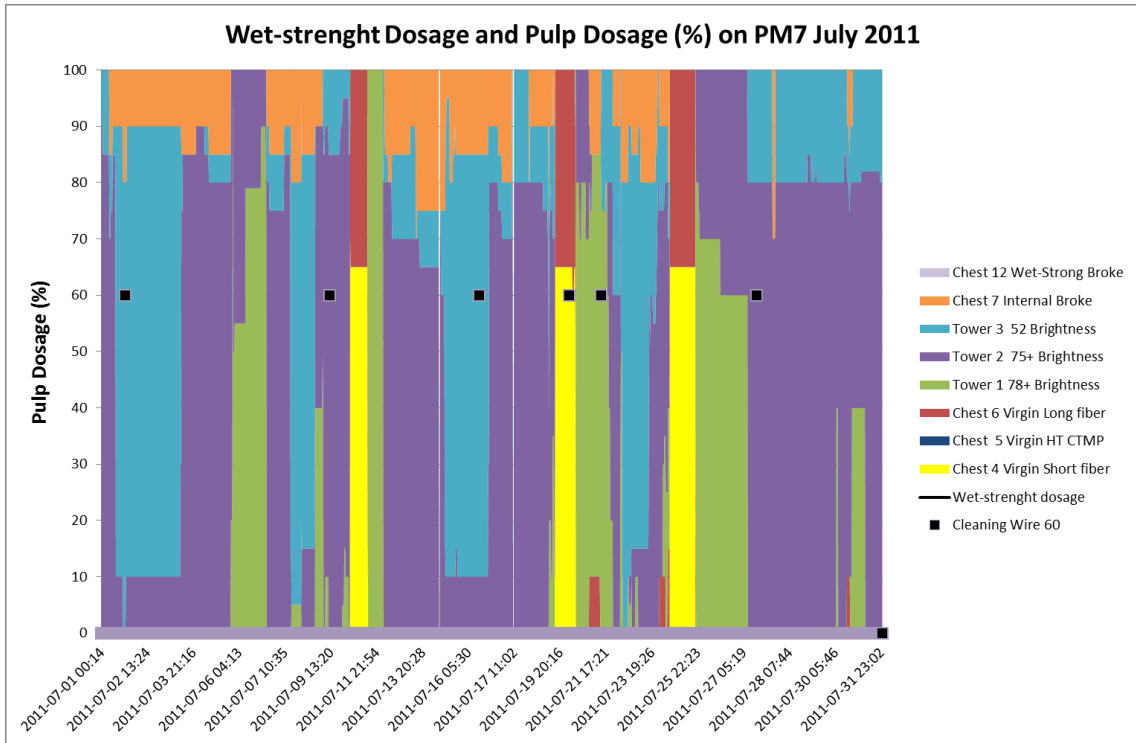


Figure 19 Diagram July 2011 PM7

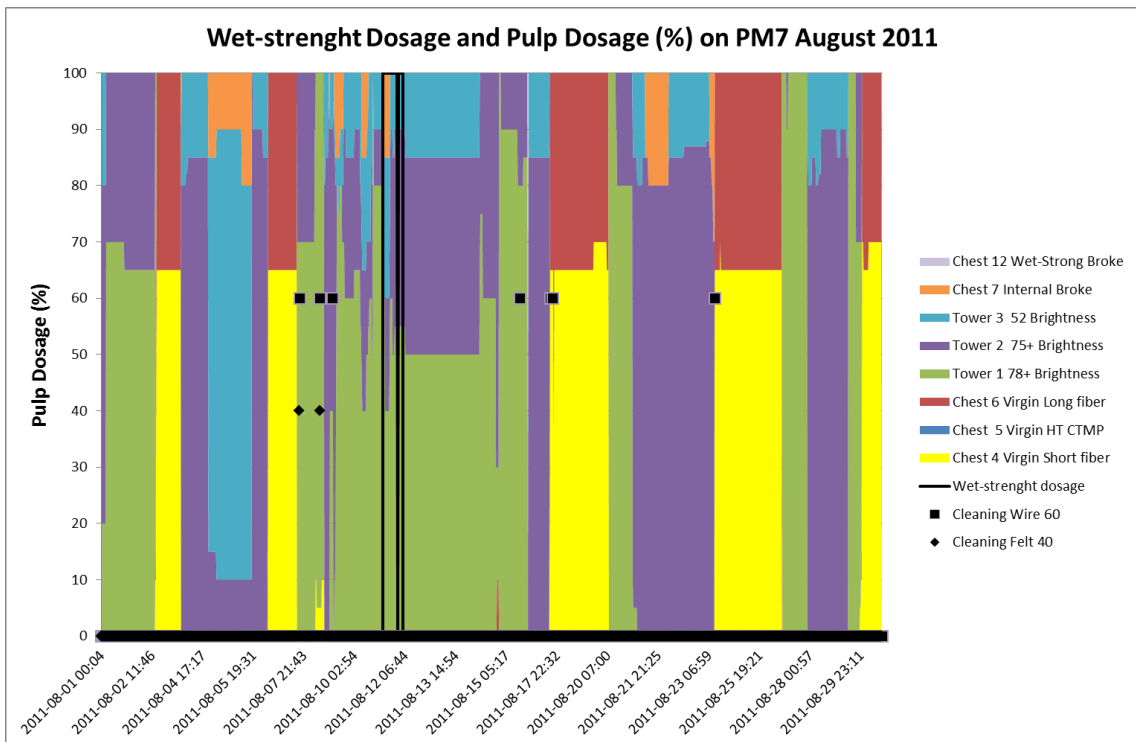


Figure 20 Diagram August 2011 PM7

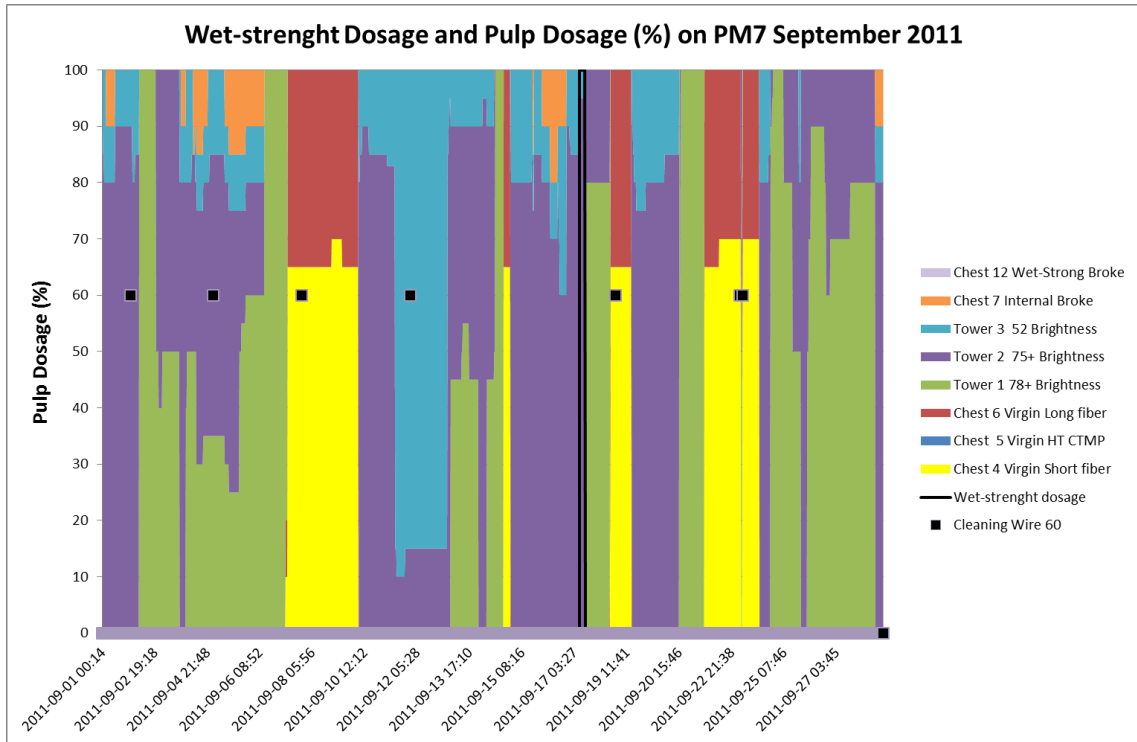


Figure 21 Diagram September 2011 PM7

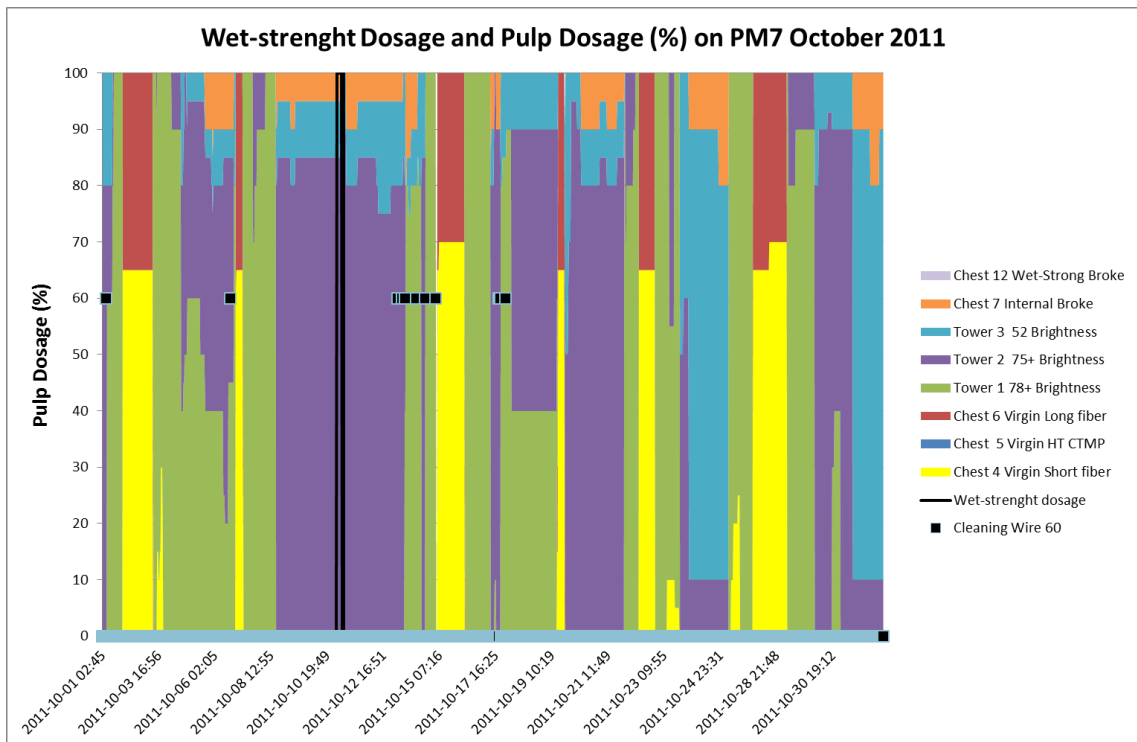


Figure 22 Diagram October 2011 PM7

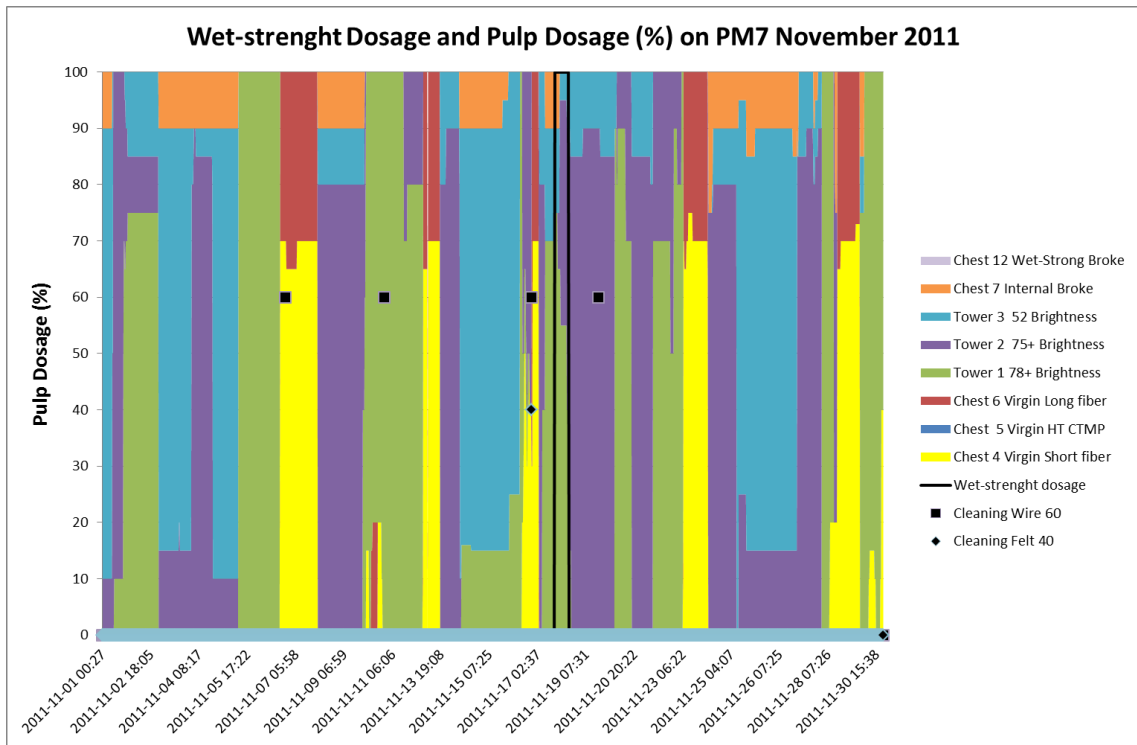


Figure 23 Diagram November 2011 PM7

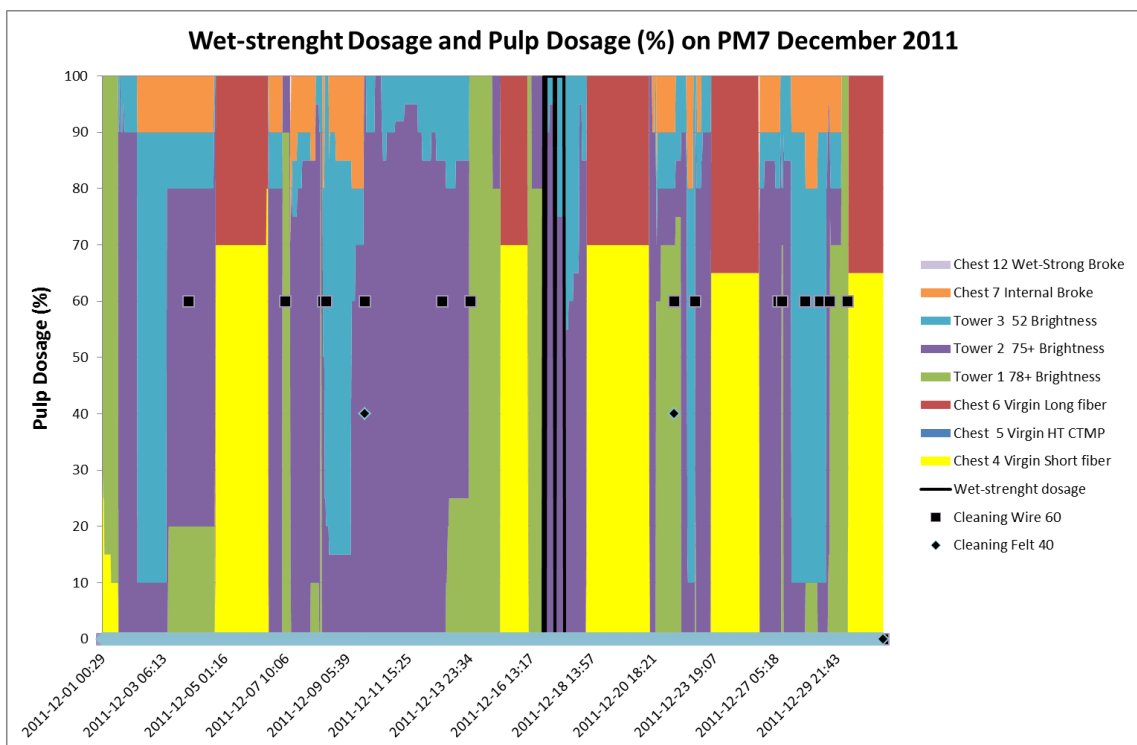


Figure 24 Diagram December 2011 PM7

APPENDIX D: RESULTS PROCESS SURVEY 1

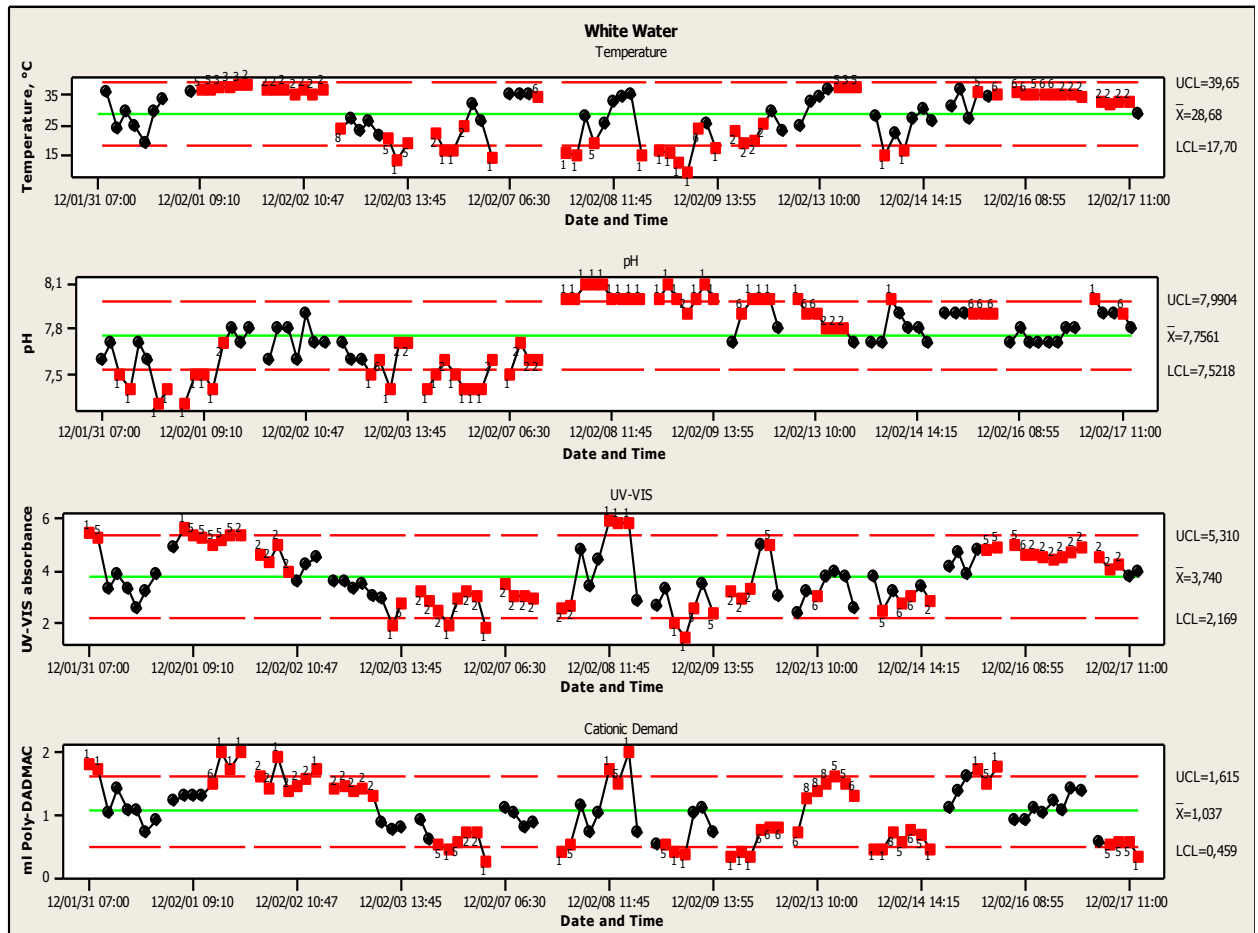


Figure 1. Variation in measured process parameters for the white water (Blekt BV).

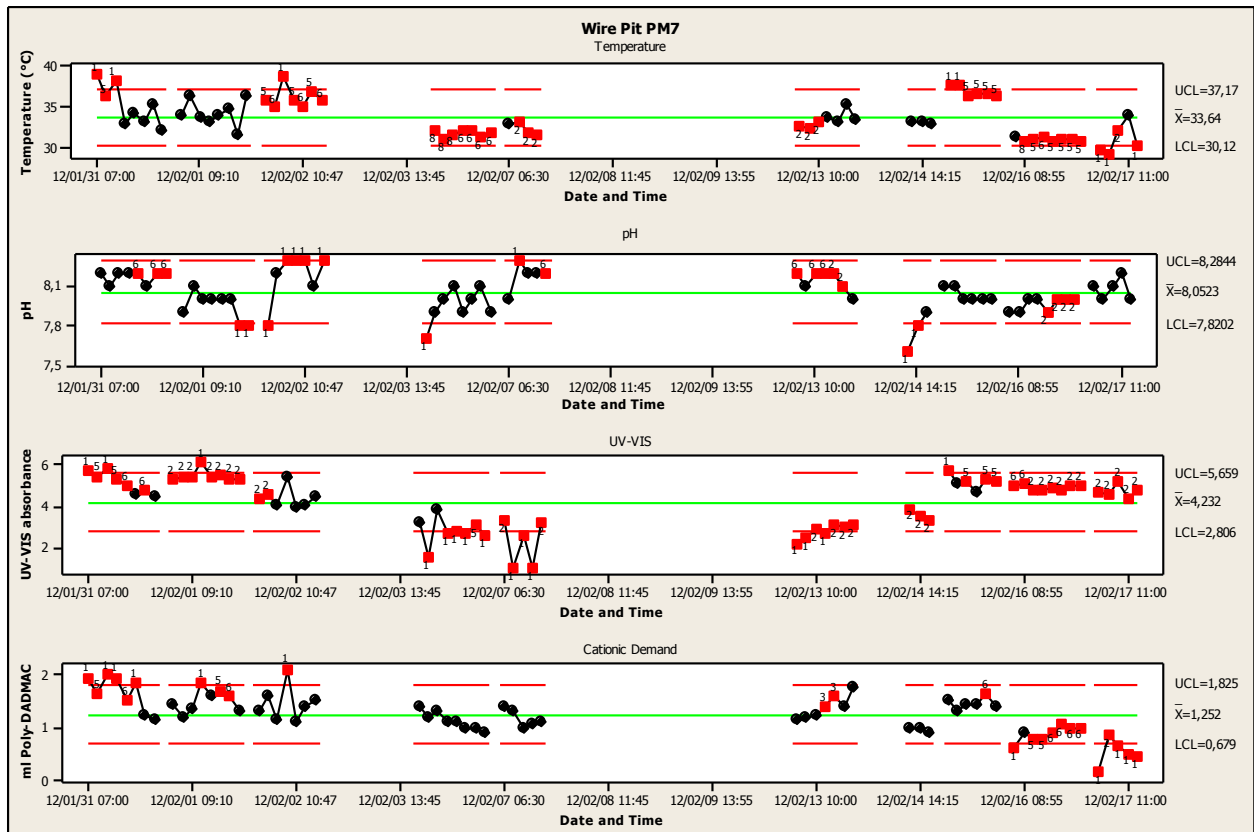


Figure 2. Variation in measured process parameters for Wire Pit PM7.

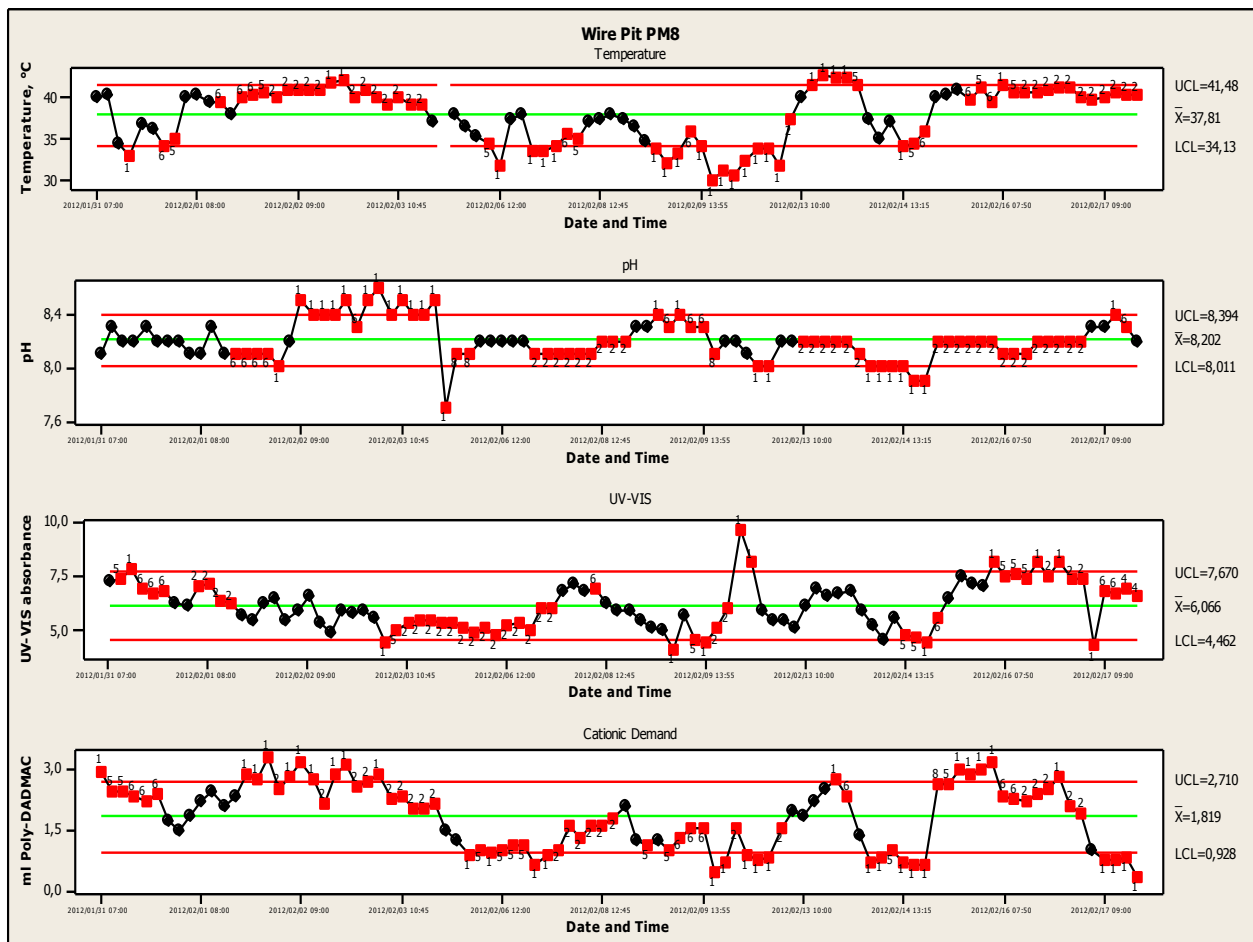


Figure 3. Variation in measured process parameters for Wire Pit PM8

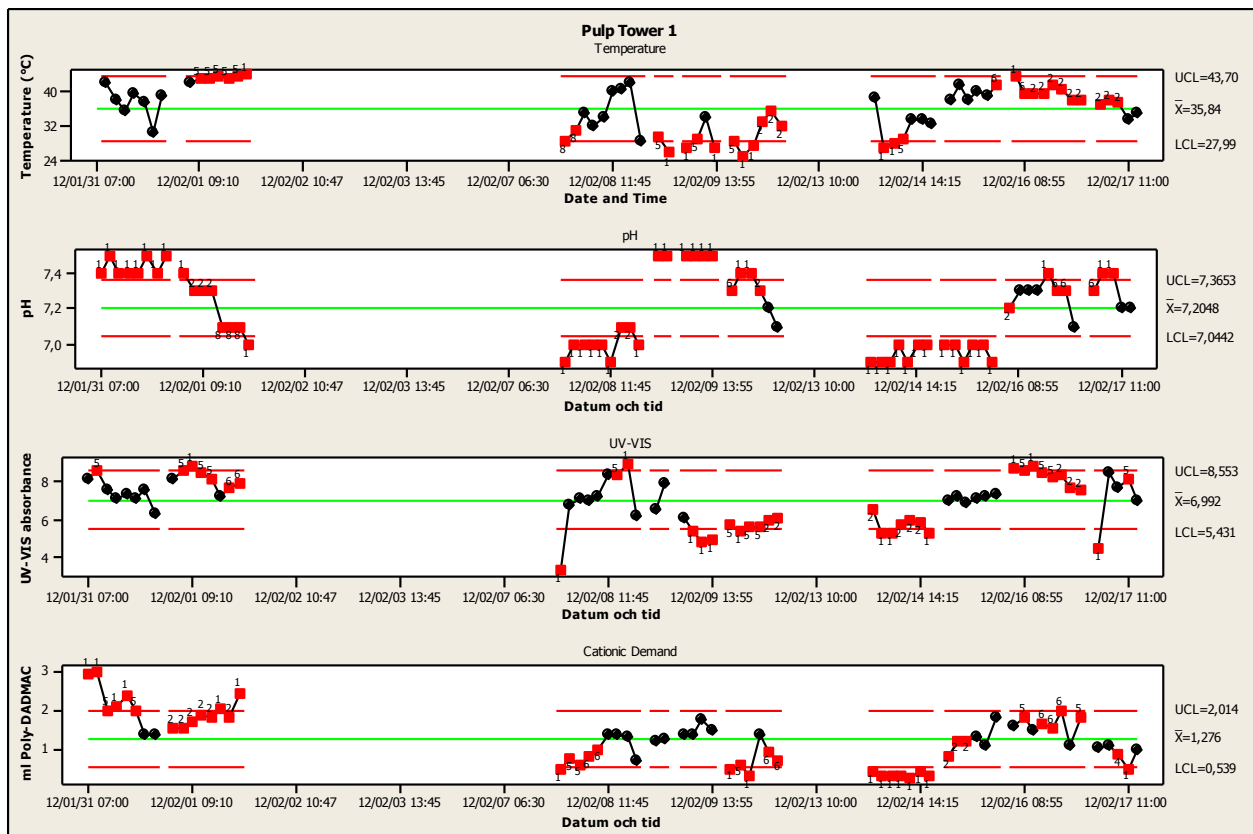


Figure 4 Variation in measured process parameters for Pulp Tower 1.

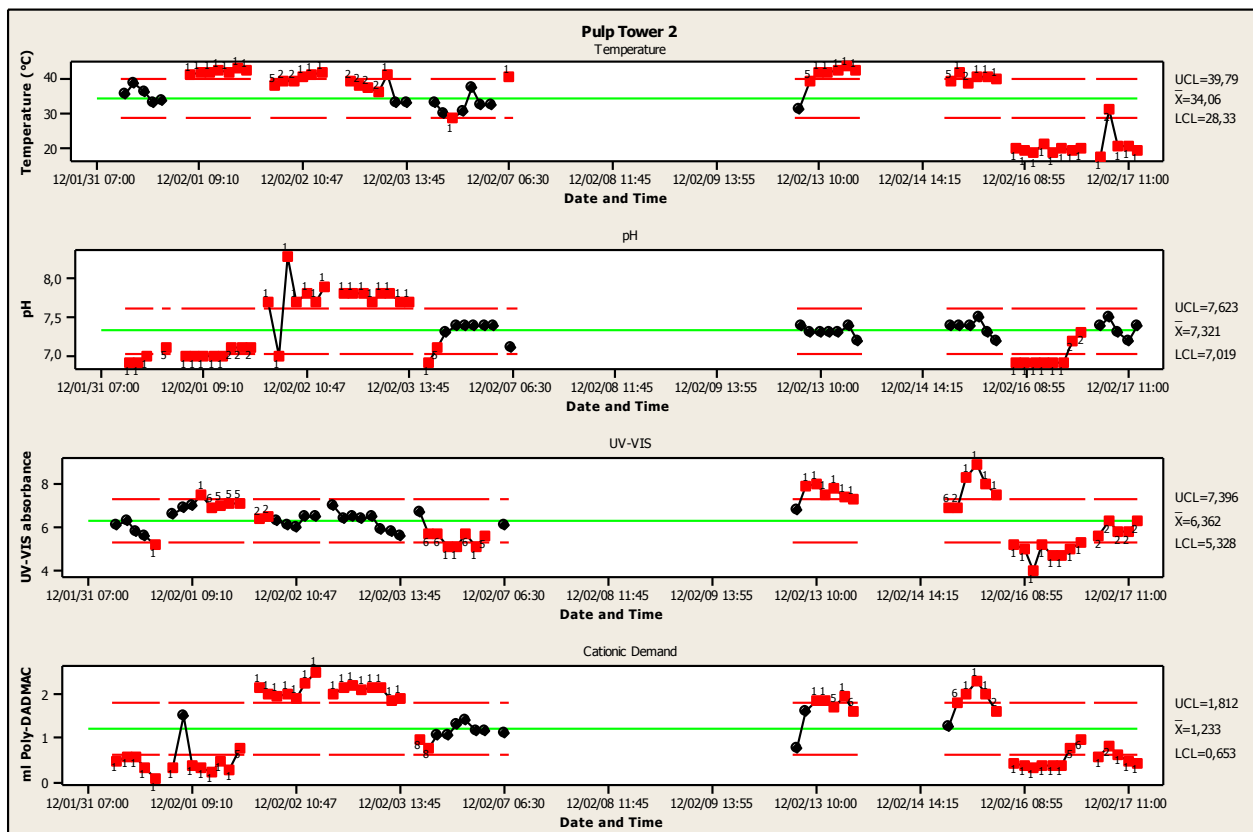


Figure 5 Variation in measured process parameters for Pulp Tower 2.

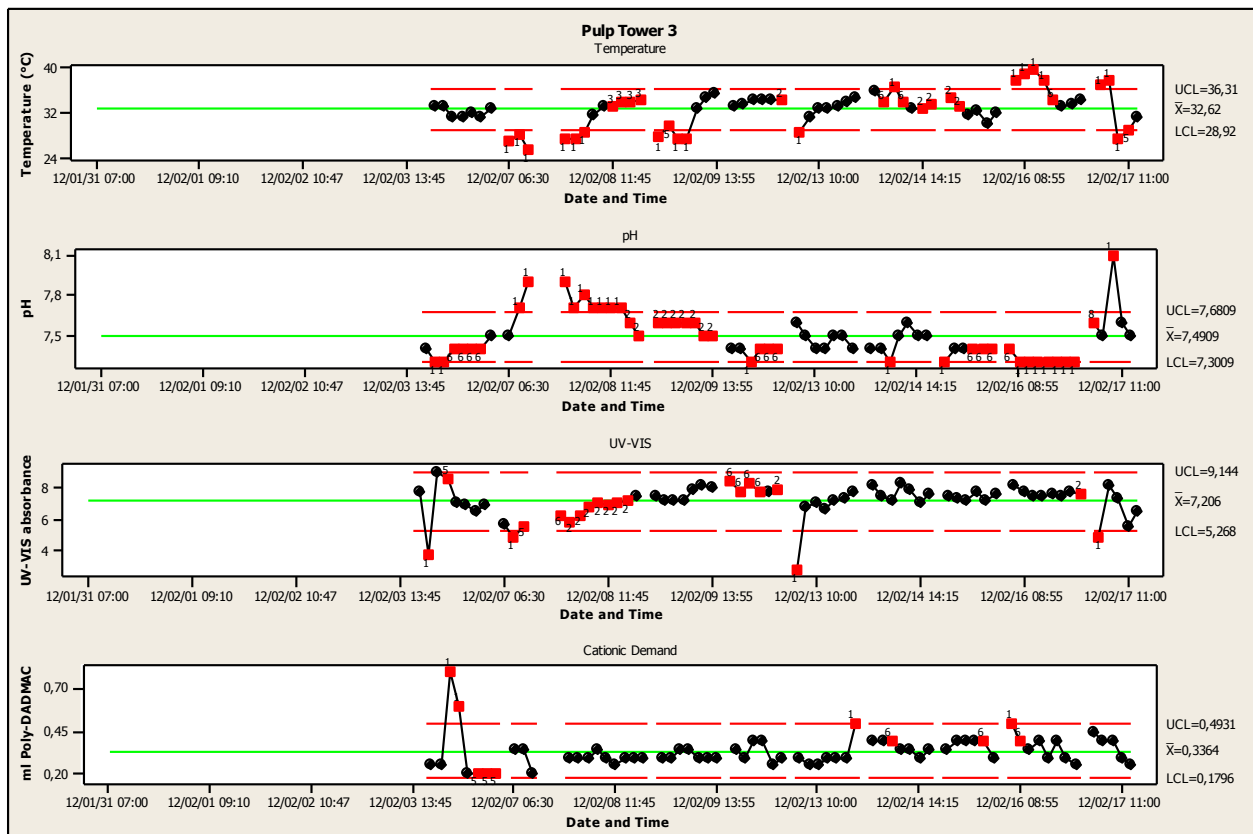


Figure 6 Variation in measured process parameters for Pulp Tower 3.

APPENDIX E: RESULTS STICKIES SURVEY MARCH 2012

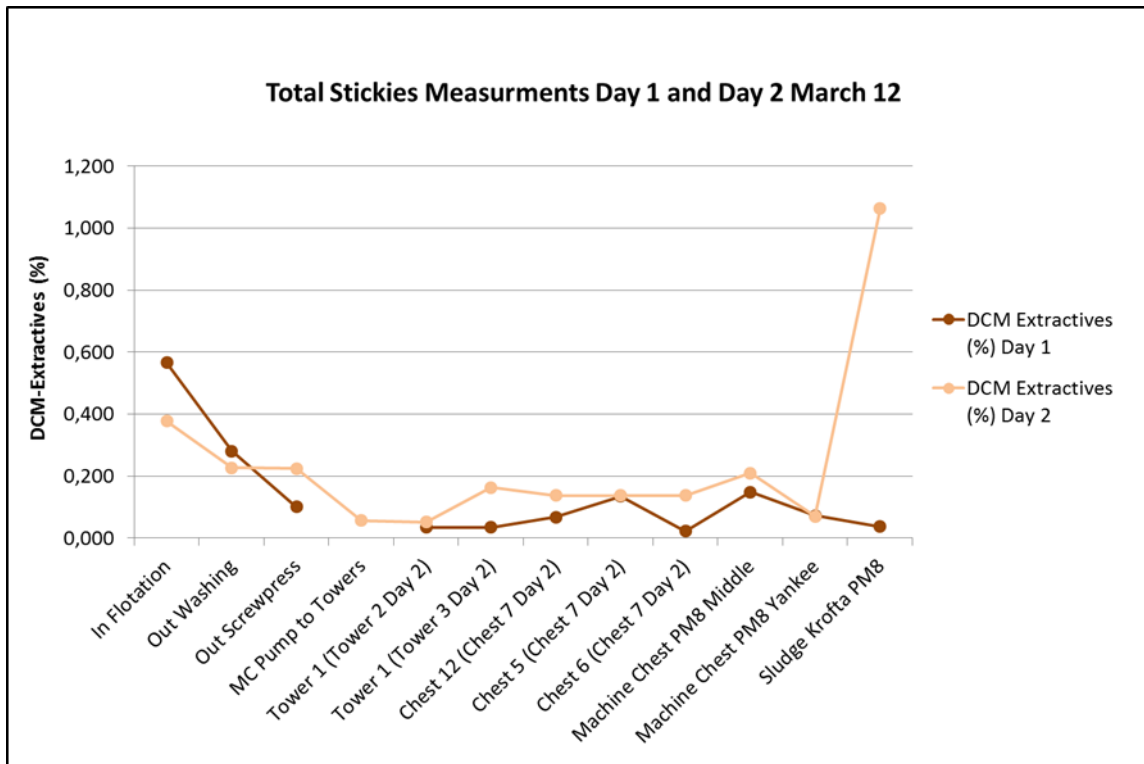


Figure 1 Results total-stickies measurements Day 1 and Day 2 Stickies Survey March 2012.

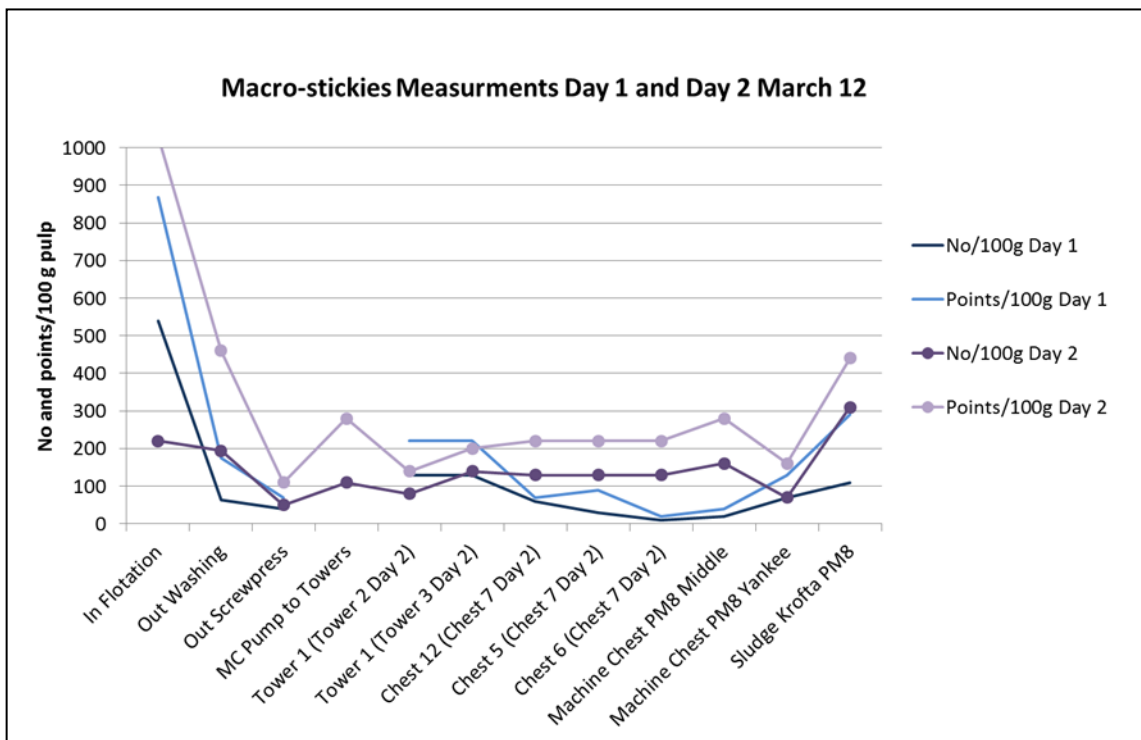


Figure 2 Results Macro-Stickies measurements Day 1 and Day 2 Stickies Survey March 2012.

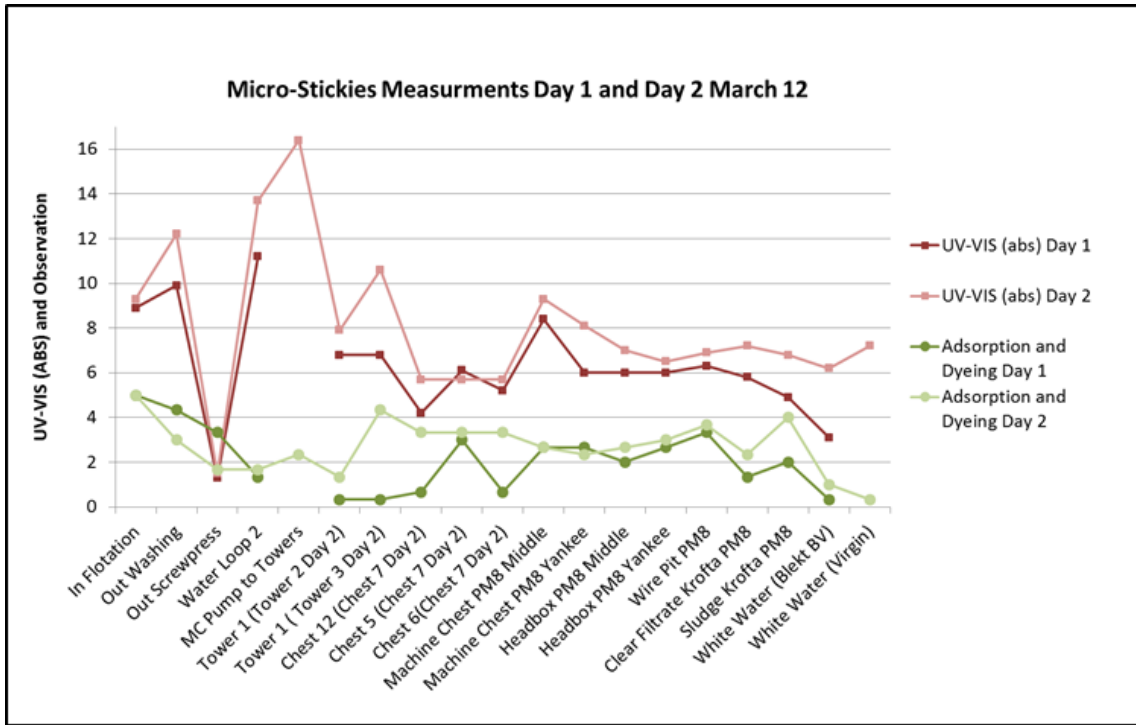


Figure 3 Results micro-stickies measurements Day 1 and Day 2 Stickies Survey March 2012.

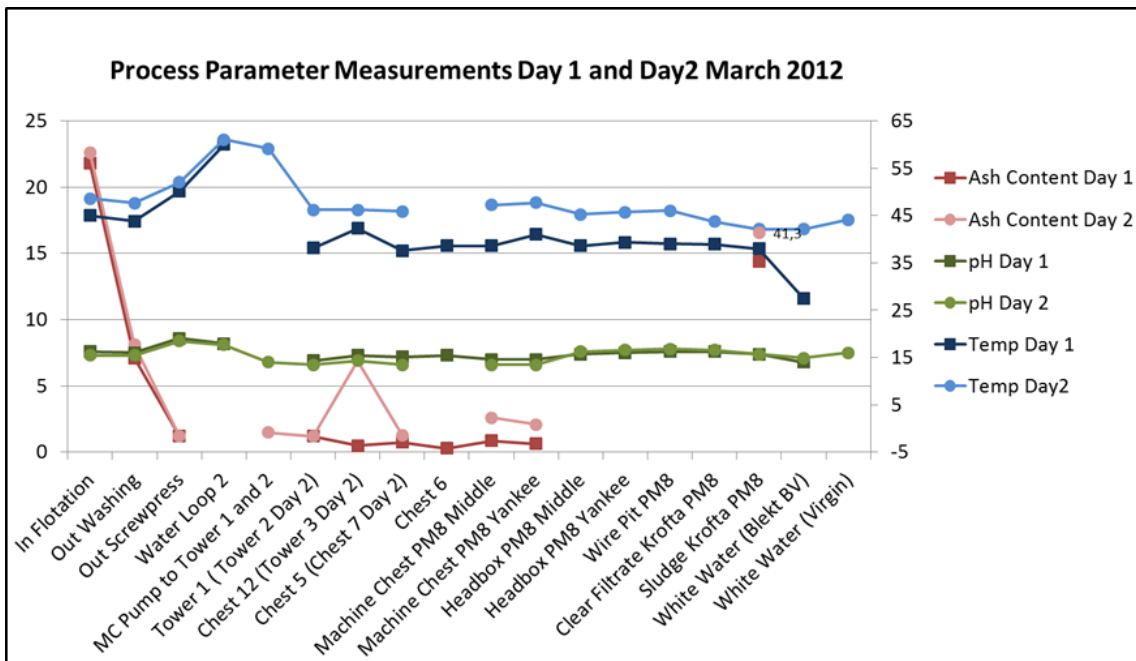


Figure 4 Diagram showing variations in process parameters. Ash content and pH are presented on the left hand axis. Temperature on the right and axis.

APPENDIX E: RESULTS METHOD INVESTIGATION

ADSORPTION AND WEIGHING EXPERIMENTS

Table 1 Results from blank samples when using 250mL bottles. Average and standard deviation is presented in bottom of the table

Date	Weight Before (g)	Weight After (g)	Difference
2012-04-12	22,3745	22,3707	-0,0038
	22,0588	22,0597	0,0009
	22,1912	22,1881	-0,0031
	22,2639	22,2613	-0,0026
	22,2564	22,2503	-0,0061
	22,063	22,0522	-0,0108
2012-04-13	22,3975	22,3705	-0,027
	22,1601	22,1465	-0,0136
	21,7352	21,7281	-0,0071
	22,1004	22,0956	-0,0048
	21,9507	21,9426	-0,0081
	22,1686	22,1588	-0,0098
	21,867	21,8426	-0,0244
Average			-0,0092538
Standard deviation			0,0079259

Table 2 Results from trials running adsorption measurements with 250 mL bottles and pulp taken into flotation.

Sample	Conc	Weight before	Weight after	Diff	Diff + 0,0093	Amount of pulp (g)	g stickies/ g air dried pulp	g stickies/ 100g air dried pulp
1	1,45	22,2711	22,2855	0,0144	0,0237	8,7000	0,0027	0,2724
2	1,45	22,2741	22,2565	-0,0176	-0,0083	8,7000	-0,0010	-0,0954
3	1,45	22,1881	22,1815	-0,0066	0,0027	8,7000	0,0003	0,0310
4	1,45	22,3583	22,3561	-0,0022	0,0071	8,7000	0,0008	0,0816
5	1,45	22,0623	22,0580	-0,0043	0,0050	8,7000	0,0006	0,0575
6	1,49	21,9998	22,0035	0,0037	0,0130	8,9400	0,0015	0,1454
7	1,49	22,2591	22,2584	-0,0007	0,0086	8,9400	0,0010	0,0962
8	1,49	22,2503	22,2529	0,0026	0,0119	8,9400	0,0013	0,1331
2. 1	1,77	22,0673	22,0605	-0,0068	0,0025	10,6200	0,0002	0,0235
2. 2	1,77	22,2677	22,2693	0,0016	0,0109	10,6200	0,0010	0,1026
2. 3	1,77	22,1384	22,1245	-0,0139	-0,0046	10,6200	-0,0004	-0,0433
2. 4	1,77	22,3705	22,3763	0,0058	0,0151	10,6200	0,0014	0,1422
2. 5	1,77	22,1465	22,1524	0,0059	0,0152	10,6200	0,0014	0,1431
2. 6	1,77	21,7281	21,7357	0,0076	0,0169	10,6200	0,0016	0,1591
2. 7	1,22	22,0956	22,0918	-0,0038	0,0055	7,3200	0,0008	0,0751
2. 8	1,22	21,9426	21,9562	0,0136	0,0229	7,3200	0,0031	0,3128
2. 9	1,22	22,1588	22,1962	0,0374	0,0467	7,3200	0,0064	0,6380
Average				0,0022	0,0115		0,0013	0,1338
Standard deviation				0,0125	0,0125		0,0016	0,1634

Table 3 Results from blank samples when using 125 mL bottles. Average and standard deviation is presented in bottom of the table.

Date	Weight Before (g)	Weight After (g)	Difference
2012-04-16	13,6135	13,612	-0,0015
	13,6975	13,6824	-0,0151
	13,6569	13,6516	-0,0053
	13,7162	13,7134	-0,0028
	13,7478	13,7451	-0,0027
	13,5017	13,4966	-0,0051
	13,905	13,9015	-0,0035
Average			-0,0051429
Standard deviation			0,0042527

Table 4 Results from trials running adsorption measurements with 125mL bottles and pulp taken into flotation.

Sample	Conc	Weight before	Weight after	Diff	Diff + 0,0051	Amount of pulp (g)	g stickies/ g air dried pulp	g stickies/ 100g air dried pulp
3.1	1,52%	13,4973	13,5013	0,0040	0,0091	0,0152	0,0001	0,0150
3.2	1,52%	13,9874	13,9909	0,0035	0,0086	0,0152	0,0001	0,0141
3.3	1,52%	13,9650	13,9631	-0,0019	0,0032	0,0152	0,0001	0,0053
3.4	1,52%	13,8266	13,8277	0,0011	0,0062	0,0152	0,0001	0,0102
B1	1,52%	13,6120	13,6203	0,0083	0,0134	0,0152	0,0002	0,0220
Average				0,0030	0,0081		0,0001	0,0133
Standard deviation				0,0038	0,0038		0,0001	0,0062

APPENDIX F: RESULTS STICKIES SURVEY MAY 2012

In appendix F results from stickies survey may are presented including measurements of process parameters and stickies determination methods.

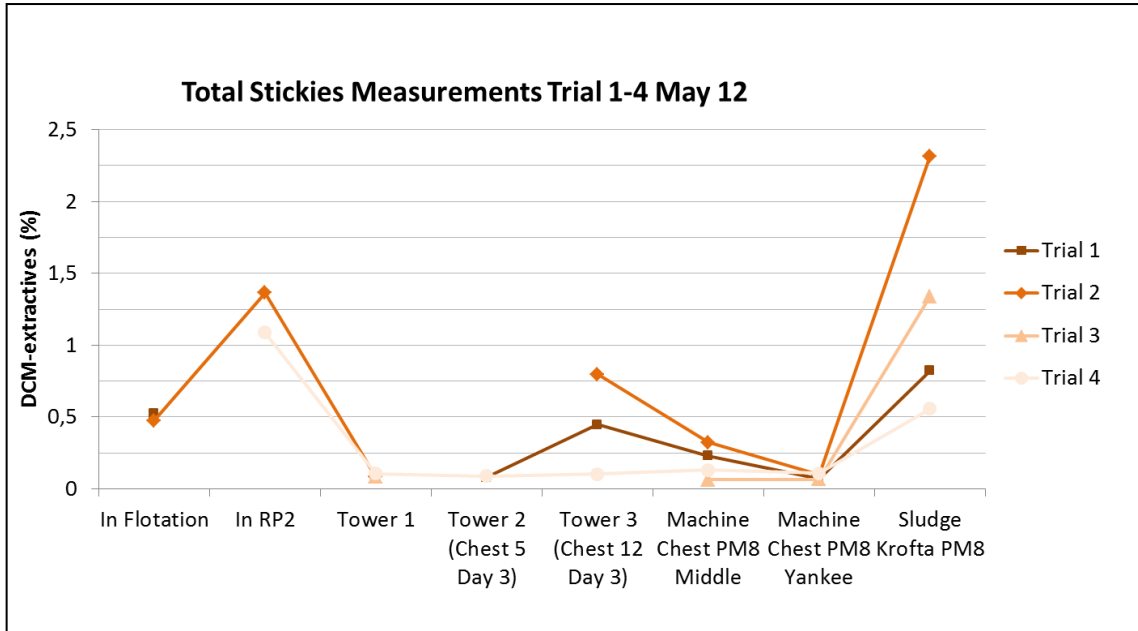


Figure 1 Results from DCM-extraction measurements.

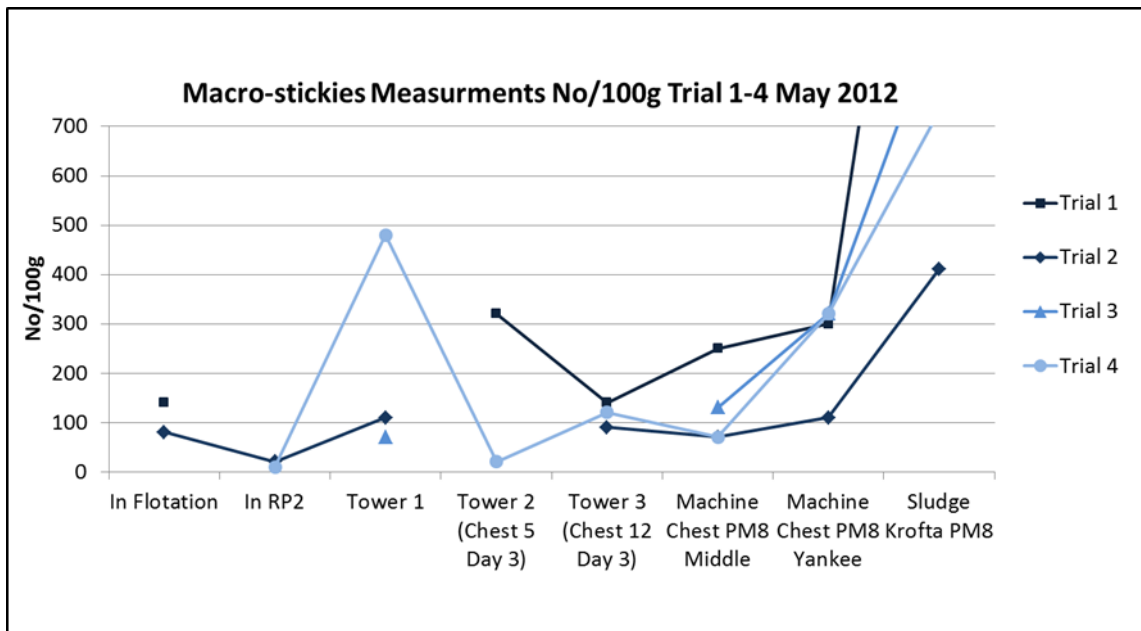


Figure 2 Results from Macro-stickies measurements in terms of No/100g.

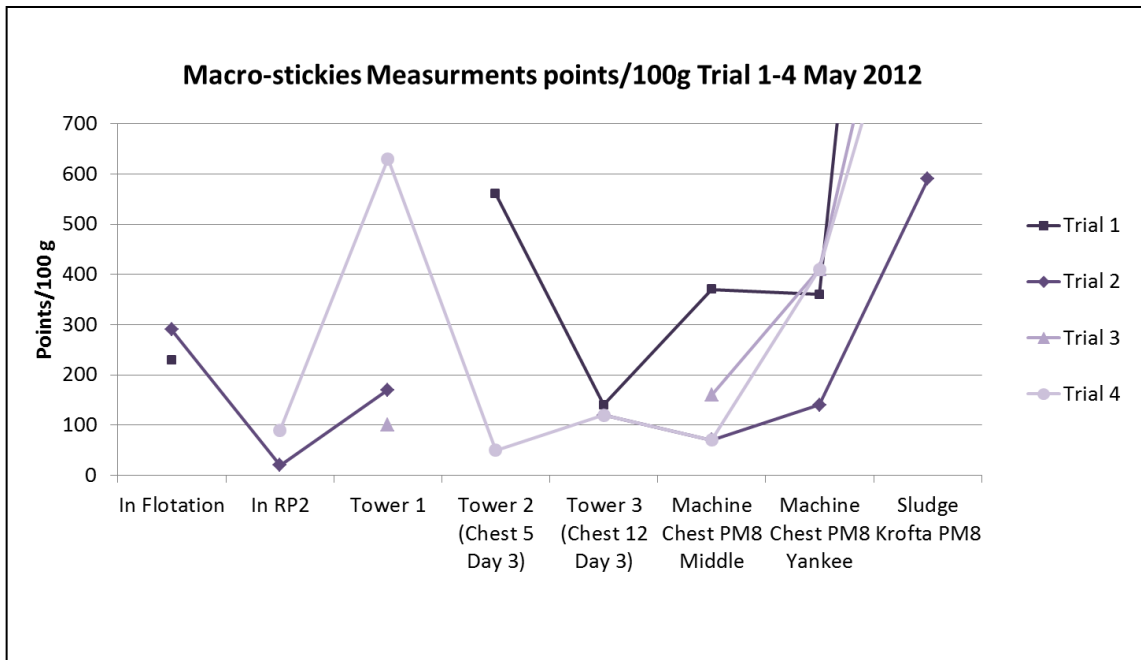


Figure 3 Results from Macro-stickies measurements in terms of points/100g.

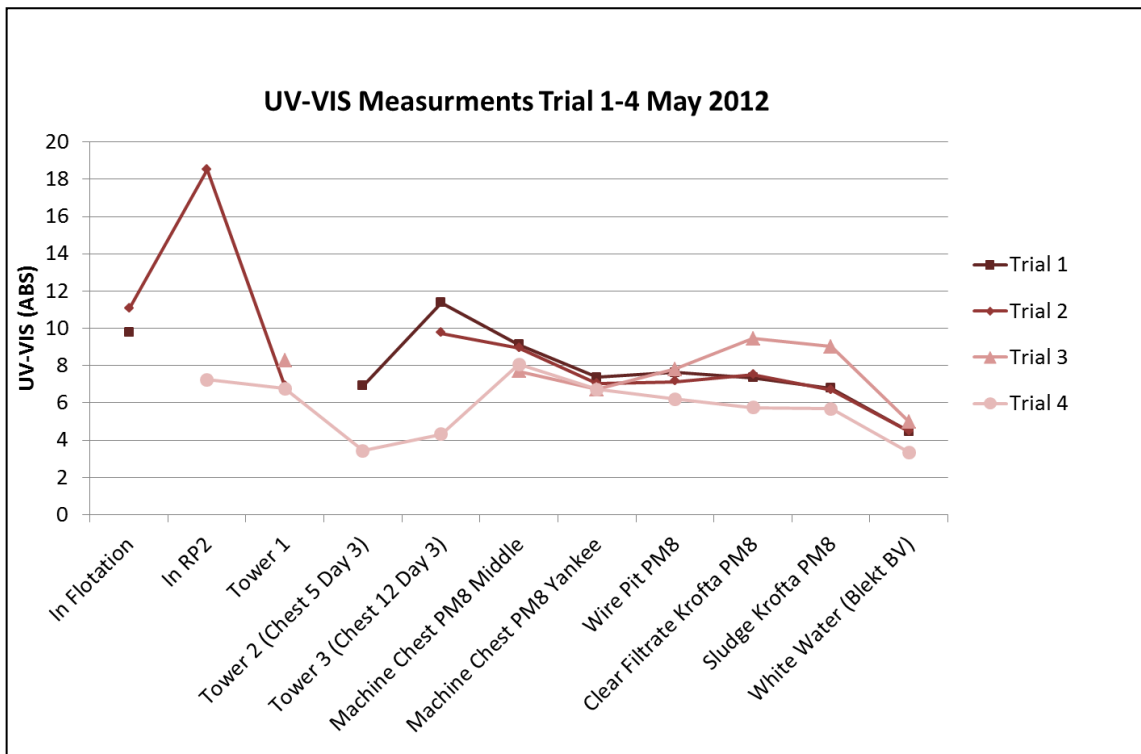


Figure 4 Results UV-VIS measurements Stickies Survey May 2012.

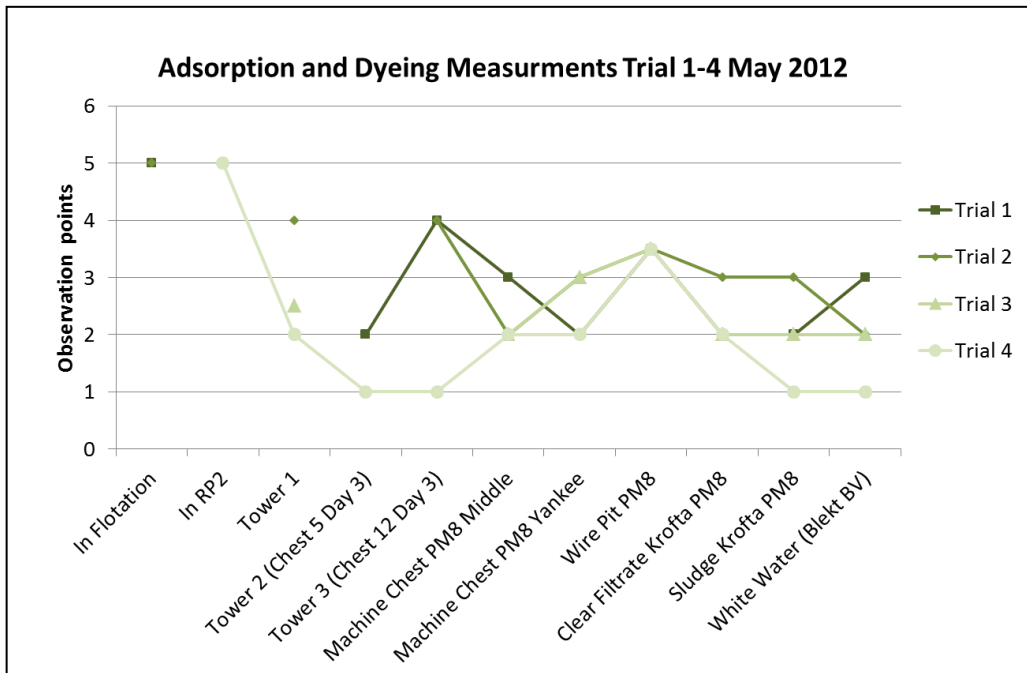


Figure 5 Results from Adsorption and Dyeing Measurements during Trial 1-4.

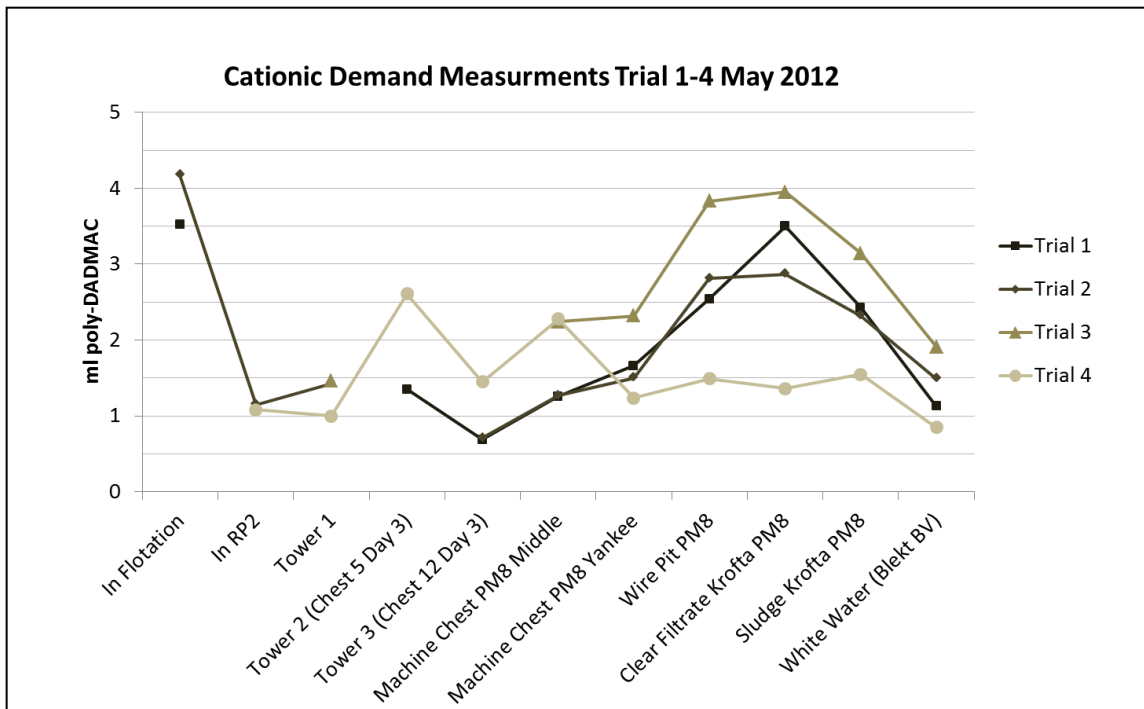


Figure 6 Results from Cationic Demand Measurements during Trial 1-4.

CCT RESULTS

CCT-results for the positions Clear Filtrate, White Water and Wire Pit is presented in the Figure 8- 12. In Table 1 the intervals for respective class is presented.

Table 1 Intervals for class 1-4.

Class	Size (μm)
Class 1	0 - 0.4
Class 2	0.4 - 0.8
Class 3	0.8 - 1.6
Class 4	1.6 - 20

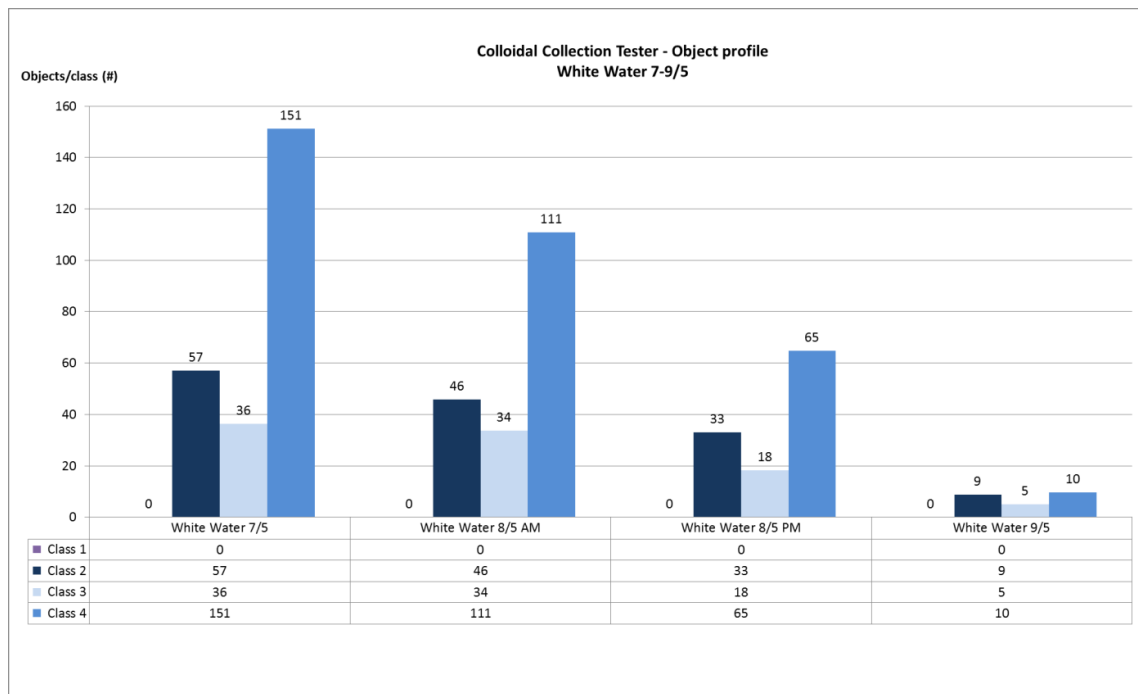


Figure 7 Number of objects in each size class for the White water 7-9/5.

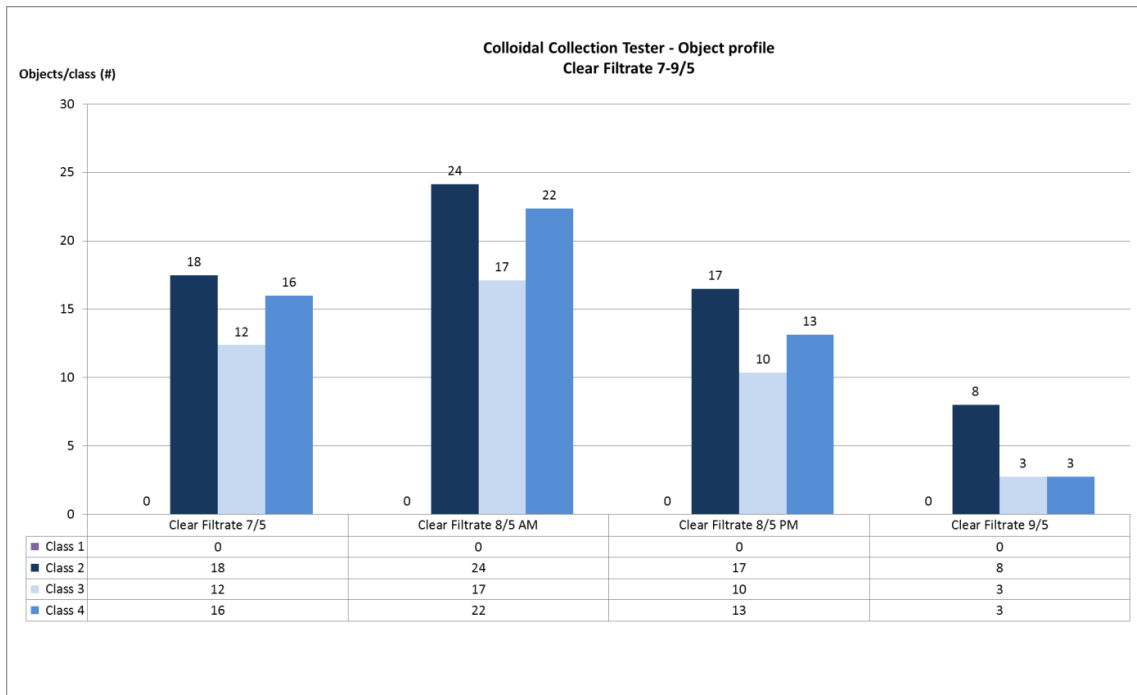


Figure 8 Number of objects in each size class for the Clear filtrate 7-9/5.

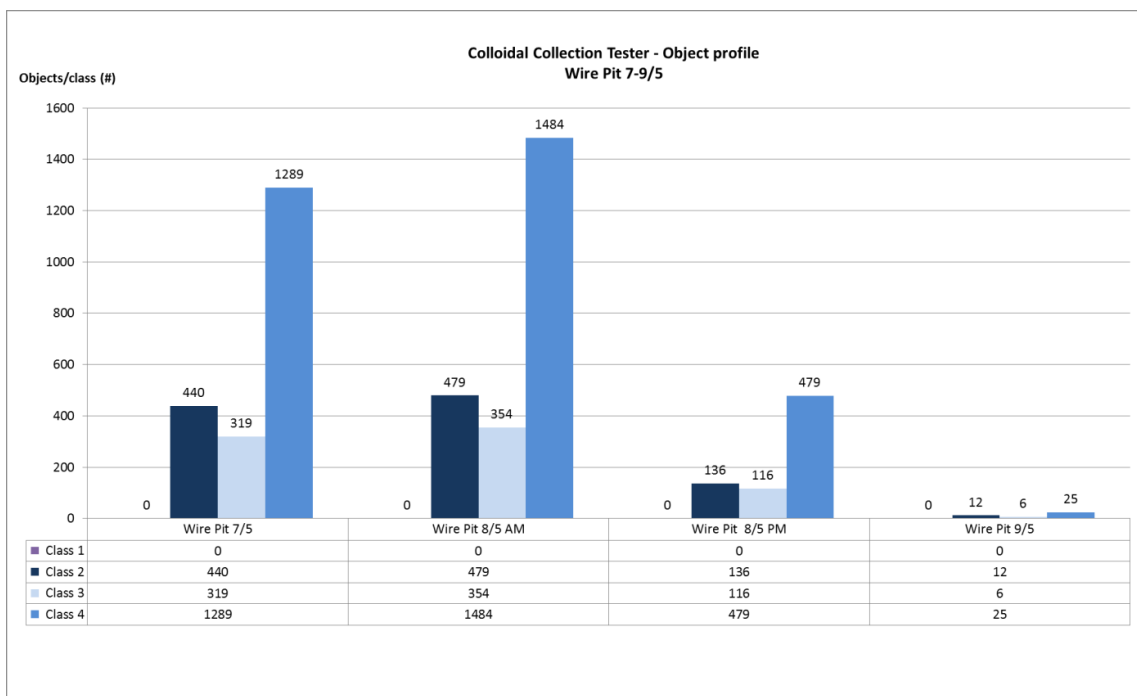


Figure 9 Number of objects in each size class for the Wire pit 7-9/5.

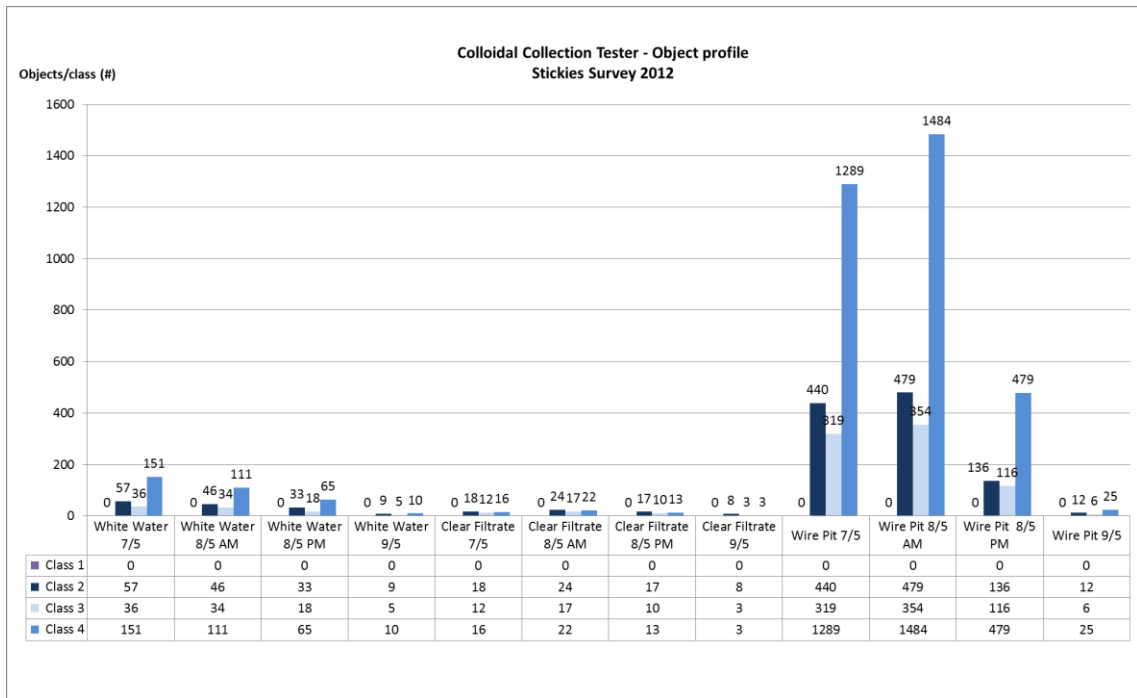


Figure 10 Number of objects in each size class for the White water, Clear Filtrate and Wire pit 7-9/5.

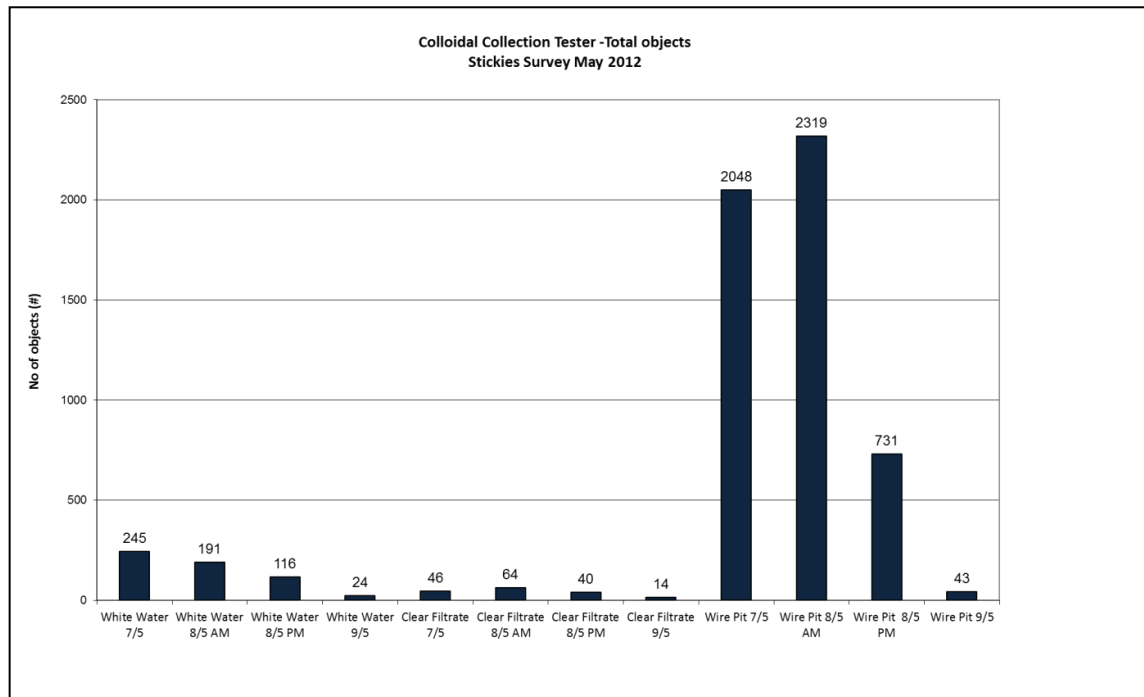


Figure 11 Total objects in samples from White water, Clear filtrate and Wire Pit 7-9/5.