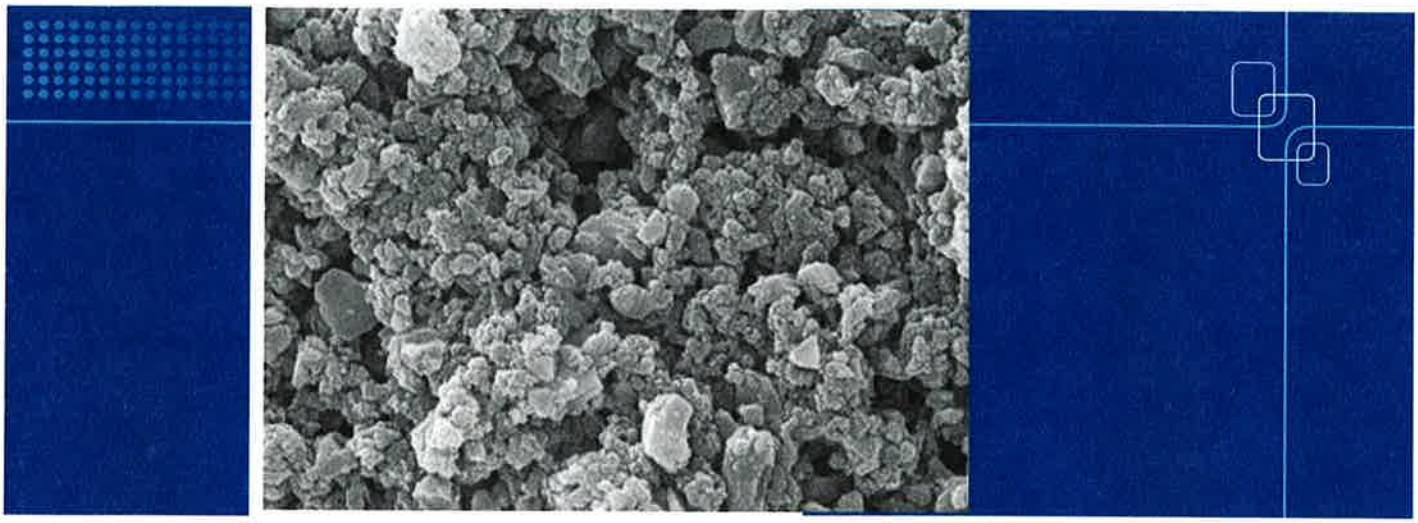
RESEARCH REPORT

VTT-R-02759-12



INT-Testaa trials at week 3 – 4 /2012

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Confidentiality: Public

Report's title	
INT- Testaa trials at week 3 – 4 / 2012	
Customer, contact person, address Jyväskylä Innovation, Petri Nyberg	Order reference
Project name INT-Testaa	Project number/Short name 74055
Author(s) Terhi Saari, Pasi Selenius	Pages 20/19
Keywords SC paper, filler, PPH pigment, SUORA	Report identification code VTT-R-02759-12

Summary

VTT has offered its papermaking research environment (SUORA) for SMEs to give an opportunity to develop their products / technologies together with research institutes and large-sized enterprises, called TESTAA concept. The concept was marketed to international SMEs and one trial week in INT-Testaa project was run together with three SMEs and 3 large size companies.
The goal at week 3 – 4 / 2012 was to produce high filler content SC paper, up to 50 % with new type of pigment polymer hybrid filler (PPH 1080) by Omya. Filler blend Clay & Omya Gloss 500 as a mixture 50 / 50 was used as a reference up to filler content 30%. The filler content was increased from 30 % to 40 & 50 % with PPH filler. The focus was on dewatering in former and press section and paper properties. Variables in the trials besides the filler content was in the HiVac vacuums in the former section and press loads and nip tilt profiles in the press section.

Altogether 38 trial points were done in four trial days. It was easy to reach the high filler contents with this special filler and the machine runnability was good within the whole week.

The results can be concluded:

- Pulps and wet end process stayed stable during the trial week, runnability being good in former and press section
- The average grammage of the trial points were 57 g/m^2, varying between 54 – 58 g/m^2
- Total filler retention was ~ 35 % with filler content 30%, ~ 53 % with filler content 40% and ~56 with filler content 50%.
- The retention of PPH pigment was higher comparing to traditional pigments, which was noticed as higher filler retention in filler contents 40% and 50%.
- The filler distribution through the thickness of the papers showed the higher filler content in all cases on the bottom (wire) side of the sample decreasing on the top side. The evenness of the distribution was increased as a function of filler content which was expected. Higher nip load increased the evenness.
- The dry content increased clearly as a function of filler content after former and press section. The average dry solids content at filler level 30 % after press section was 46%, at filler level 40% 51% and at highest filler level 54%.
- ISO –brightness increased linearly as a function of filler content: the average ISO-brightness at filler level 30 % was 74.6 %, at filler level 40% 76.5 % and at highest filler level 78.7 %.
- Average opacity at filler level 30% was 92.1 % increasing to filler level 40 & 50% to 93.9 %, respectively
- Tensile index (MD) decreased 32 % when filler content increased from 30% to 40%. The drop was somewhat lower from filler level 40 to 50% being 22%.
- The runnability in press to dryer section measured as residual tension decreased roughly same per cent than dry tensile index, but if indexing the values by fibre grammage residual tension decreased 17% and only 4%, respectively.

Confidentiality	Public

Jyväskylä 11.4.2012

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Contents

1 Introduction

VTT has offered its papermaking research environment (SUORA) for SMEs to give an opportunity to develop their products / technologies together with research institutes and large-sized enterprises. This concept is called TESTAA and has launched at 2009. New ideas can be evaluated for their industrial potential in an early stage and developed further. Each company can independently decide how much of the project results they want to make public or inform to the other companies. INT- Testaa project has been focused on marketing the concept to international companies and SMEs and one trial week was planned to run in the project. The project is funded by Jyväskylä Innovation Ltd and VTT.

2 Goal

The goal of INT – Testaa trial at week 3 – 4 / 2012 was to produce high filler content SC paper, up to 50 % with new type of pigment polymer hybrid filler (PPH) and study the drainage behaviour in former and press section. The trials were planned together with Omya, who's fillers were used in the trials.
The focus was on dewatering in former and press section and paper properties. Variables in the trials besides the filler content was in the HiVac vacuums in the former section and press loads and nip tilt profiles in the press section.

3 Participating companies

Three large- sized companies and three SMEs participated to the trials:
- Omya / Fillers / Contact person Timi Hyppänen
- Ashland / Retention systems / Contact person Eemeli Siitonen
- UPM / Papermaking / Contact person Jussi Ventola
- Acosense / Acospector® Acoustic Chemometer / Contact person Felix Törner
- Vasasensor / PressEyes Portable / Contact person Sofia Kocher
- SafePower / Filler dosing control / Contact person Juha Ulmala

4 Trial run plan

The filler content of SC paper was increased from 30 % → 40 % → 50% with PPH – pigment. The grammage target of SC-paper was 52 g/m^2.
At week 3 the actual trial points were done and at week 4 on Monday and Tuesday the environment was run just with the water. Acosense and Safe Power made their measurements at week 3 and Vasasensor did their measurements in press section at week 4. Paper samples were taken as A4-sheets from each trial point.

4.1 Furnish and chemicals

SC pulp mixture, 80 m^3, was delivered from Finnish paper mill. Tap water was used as process water. Each trial day 15 - 20 m^3 of pulp and 20 m^3 tap water was needed in the trial batch. Trials were run in pH –level 8.

Filler blend Clay & Omya Gloss 500 as a mixture 50 / 50 (in proportion to dry content) was used as a reference filler and filler content was increased with this mixture up to 30%. The filler content was increased from 30 % to 40 & 50 % with Omya's PPH 1080 – filler.
Ashland's dual component retention system was used, PerForm PC435 (CPAM) and PerForm SP7200 (anionic micro particle). Retention chemical dosages varied between 70 - 450 g/t for CPAM and 70 - 500 g/t for micro particle depending on the filler target.
Wires (top/bottom) used in the trials were Metso Fabrics GapMaster Pro.

4.2 SUORA concept

Former geometry in the trials was hybrid, fig 1.

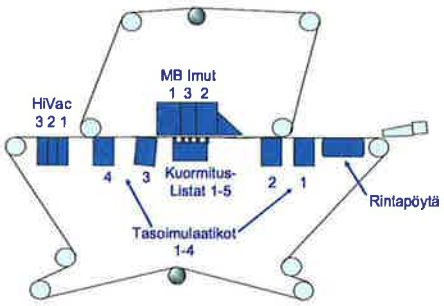


Figure 1. Hybrid-former lay-out used in the trials

Press section (*Figure 2.*) consists of 1-nip press having 350 mm extended nip and a sampling unit.

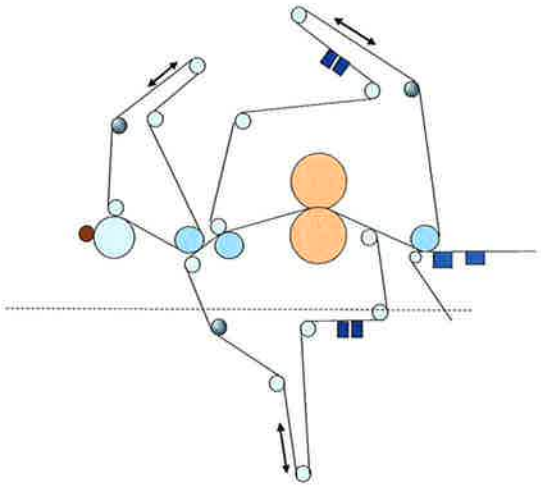


Figure 2. Press section lay-out

Running velocity was 800 m/min. Process variables: HiVac vacuum was variable in forming section being 44 – 65 kPa. Press section variables were press load 1400, 1600 and 1800 kN/m, and nip tilt profile being 2.0, 2.3 and 2.6.

4.3 Trial Schedule

The trial schedule is shown in below. In first week, Tuesday, Thursday and Friday were the actual trial point days, the difference between the days being filler content in the paper web. On Wednesday the retention chemical dosages for actual filler content were defined. Altogether 38 trial points were made and analysed. Trial points 1 – 10 was run on Tuesday, filler content being 30 %, trial points 22 – 30 on Thursday with filler content 40 % and trial points 31 – 38 on Friday with filler content 50%. On Wednesday when the retention chemical dosage was optimised where done trial points 11 – 21.

- Tuesday 17th of Jan
  - Preparation of furnish and retention chemicals
  - Installing devices
  - Reference point (30% filler) = 15% Clay + 15 % Omya gloss
  - Dewatering trials (press tilt, press load, dewatering vacuums)
- Wednesday 18th of Jan
  - Filler load test: 30, 40, 50%
  - Retention chemical dosages & filler level in furnish/water
- Thursday 19th of Jan
  - 40% filler content, dewatering trials (press tilt, press load, dewatering vacuums) = 15% Clay + 15 % Omya gloss + 10% PPH
- Friday 20 of Jan
  - 50% filler content, dewatering trials (press tilt, press load, dewatering vacuums) = 15% Clay + 15 % Omya gloss + 20% PPH
- Monday 23th of Jan
  - Installing devices (Vasasensor)
- Tuesday 24th of Jan
  - Trial day with water

4.4 Analysis and measurements

Furnish measurements were done in the beginning and in the end of every trial day, measurements being: pH, conductivity, freeness and fibre length.

Following measurements were done from each trial point:

- dry content after former and press section
- solids and ash content in the head box and wire pit
- grammage & filler content in paper web

Paper webs were dried with Kodak laboratory dryer (Temperature 120 ^{0}C). Grammage, filler content, density, permeability, tensile strength and optical properties were measured according to SCAN standards.
Runnability measurements with Impact device as well as filler distribution were measured from samples 3, 23, 25 & 34. These samples were sent also to Omya for further analysis.

Filler content calculations

Three different filler types were used: clay, calcium carbonate and PPH (PPH was considered as calcium carbonate). The fillers were mixed with furnish during trials and the amount of filler content was then measured from head box and paper web in each trial points as mentioned above. Defining the amount of different filler types from the paper samples, three laboratory measures were done: dry weights of samples M_A, M_{L1} and M_{L2} (see fig. 3). M_{A1} is a dry weight of sample material (here paper or furnish) and M_{L1} and M_{L2} are ash weights after burning sample materials in 500 and 900 degrees respectively.

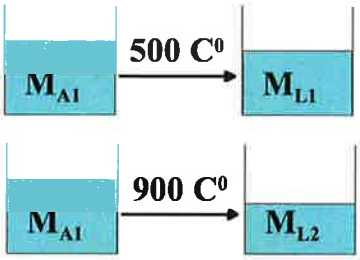


Figure 3. Ashing process was done in two different temperatures to get information about the calcium carbonate and clay filler content in the samples. Weighting was done before (M_A) and after burnings (M_L).

Using the weighting results and denoting calcium carbonate and clay filler content portions with P_{CaCO3} and $P_{kaol.}$ following equations (1.) - (3.) can be defined:

$$M_{L1} = P_1 M_{A1} + P_2 M_{A1} \text{ (ashing at 500 degrees),} \quad (1.)$$

$$M_{L2} = \frac{P_{CaCO3} M_{A1}}{a} + P_{kaol.} M_{A1} \text{ (ashing at 900 degrees),} \quad (2.)$$

$$P_{CaCO3} + P_{kaol.} = P_{tot.}. \quad (3.)$$

The assumption has made that calcium carbonate will start to decompose above 500 degrees (during ashing process), and its weight is therefore reduced with coefficient a (= 1.78). Clay weight was assumed to remain unchanged. Combining eq. (1.) to (3.) weight portions using sample weighting results can be calculated:

$$P_{CaCO3} = (M_{L1} - M_{L2}) / (M_{A1} - \frac{M_{A1}}{a}) \quad (4.)$$

$$P_{kaol.} = \frac{M_{L1}}{M_{A1}} - P_{CaCO3} \quad (5.)$$

For example in trial point kp31 following weighting results for paper sample have got: M_{A1} = 0.4577g (dry paper), M_{L1} = 0.2283g (ash in 500 degrees) and M_{L2} = 0.1569g (ash in 900 degrees). Substituting these values in equations (3.), (4.) and (5.) the results are: P_{CaCO3} = 0.3559, $P_{kaol.}$ = 0.1428 and $P_{tot.}$ = 0.4987.
The equations (3.), (4.) and (5.) were used to calculate the paper and furnish sample filler content results.

Retention calculations

Retention value R_a is calculated from solid flows
Φ: q1c1ρ1 = q2c2ρ2 + q3c3ρ3,
where q1 is slice flow rate, c1 is a head box consistency, ρ1 is a density of head box fluid, q2 is a removed water flow in forming section, c2 is a pit water consistency, ρ2 is a pit water density, q3 is a web flow rate, c3 is a web consistency and ρ3 is a web density.
Assuming that: q1 ≈q2 and ρ1 ≈ ρ2 ≈ ρ3 = 1000 kg/m3 we get:

$$\frac{\Phi 1}{\Phi 3} \cdot 100\% \equiv R_a = \left(1 - \frac{c2}{c1}\right) \cdot 100\%.$$

Here consistency values are obtained from laboratory values sampled from head box and wire pit water flows.

Retention R_b could also be calculated using paper and head box flow values:

$$R_b = \left(\frac{\Phi_4}{\Phi_1}\right) \cdot 100\% = \frac{bw \cdot v \cdot w}{q1 \cdot c1 \cdot \rho 1} \cdot 100\%,$$

where bw is a basis weight of paper, v is a machine velocity, w = 0.3 m is a head box width.

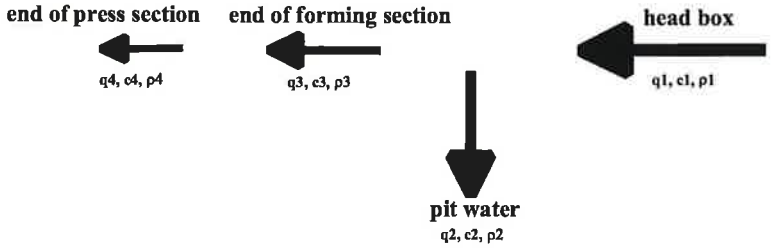


Figure 4. Retention values were calculated assuming that solid flows are conserving and samplings are representative. Measurements that are needed for calculations are: consistencies (head box, pit water), flow rates (head box, pit water) and paper basis weigth.

5 Results

The results of all trial point are presented in appendix 1 (excel – file/ Table).

5.1 Furnish & retention chemical dosages

The furnish from pulp tank was analysed every day to check that it will stay stable during the trials. The results shown in the table 1 shows that no major changes were detected in the pulp.

Table 1 . Furnish analysis from pulp tank

Pulp tank 100 m3		17.1.2012	18.1.2012	19.1.2012	20.1.2012
Temperature	°C	41,1	38,4	42,8	37,8
pH		7,1	6,9	7,3	7,6
Conductivity	µS/cm	2860	2940	2710	2770
Consistency	%	3,17	3,02	2,84	2,84
Ash	%	17,3	17,03	17,74	17,45
Freenes	ml	50	56	52	57
Zeta-potential	mV	-13,9	-18,3	-19,6	-19,1

Ashland's dual component retention chemicals dosages with different filler contents were optimised on Wednesday. Dosages with different filler contents were:

- filler content 30%: PerForm PC435 (CPAM) 70 g/t , PerForm SP7200 (anionic micro particle) 70 g/t
- filler content 40%: PerForm PC435 (CPAM) 200 g/t , PerForm SP7200 (anionic micro particle) 200 g/t
- filler content 50%: PerForm PC435 (CPAM) 450 g/t , PerForm SP7200 (anionic micro particle) 500 g/t

5.2 pH and wire pit analysis

pH level in trials was ~8, measurements from Sortteri tank varied between trial days from pH 7.97 to pH 8.16.

Cationic demand was measured twice a day from wire pit (first and last trial point) (Table 2). Charge in wire pit was staying rather stable in the system during the each trial day, the small difference might come from dilution effect of shower waters. Retention chemical dosages were rather high on the last day (filler content was 50%) which might have an effect on somewhat higher cationic demand.

Table 2 . Cationic demand of the samples

RM3 former pit water tank		Cationic demand, µekv/l
Filler content 30%	KP 1	21,4
	KP9	16,3
Filler content 40%	KP22	26,3
	KP30	16,2
Filler content 50%	KP31	43,4
	KP38	38,3

5.3 Grammage and filler content & distribution

The average grammage of trial points during trial week was 57 g/m^2, varying between 54 – 58 g/m^2 (figure 5). Density was increasing as a function of filler content (figure 6). Average density with filler content 30 % was 486 kg/m^3, with filler content 40 % 506 kg/m^3 and with filler content 50% 537 kg/m^3.

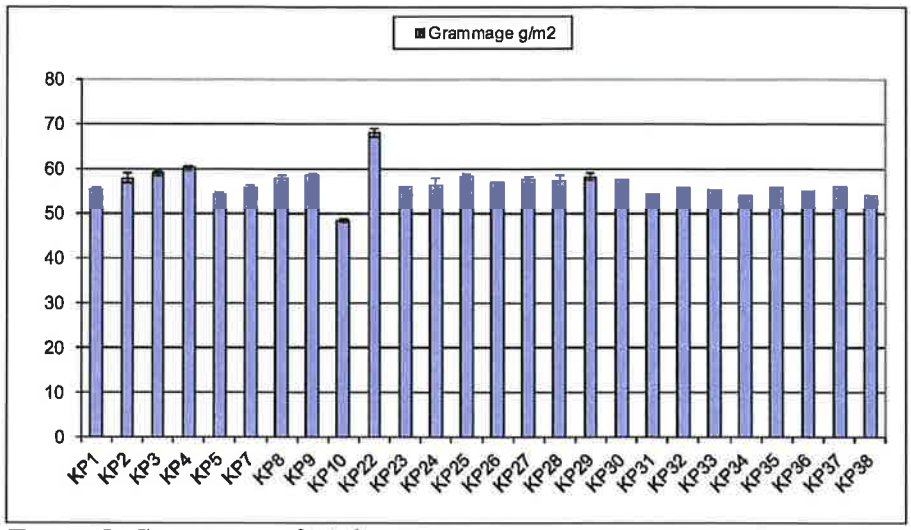


Figure 5. Grammage of trial points

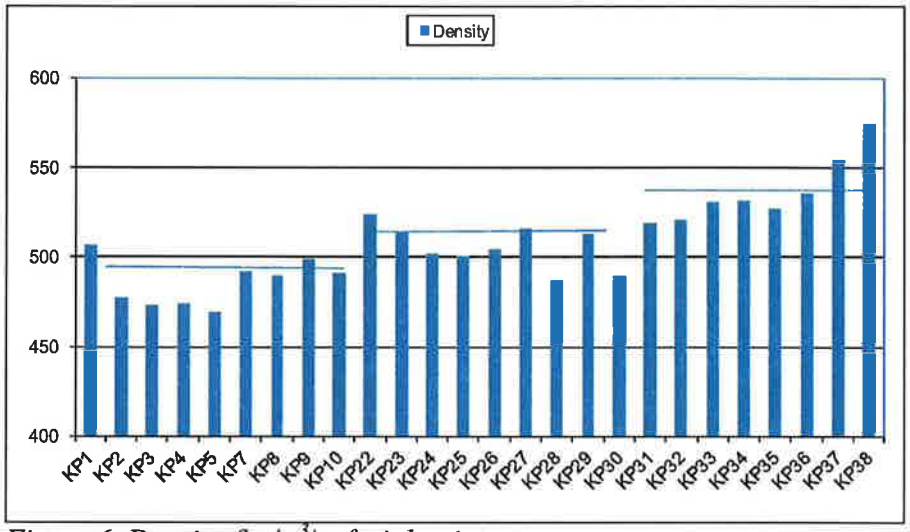


Figure 6. Density (kg/m^3) of trial points

Filler content of trial points are shown in figure 7. Target filler content was reached every trial day, first day the average filler content was 28 %, on second day 41 % and on Friday 51%. With lower filler contents (30 and 40%) filler content was decreased ~2 % units during trial day, however with filler content 50 % it was increased with the same amount.

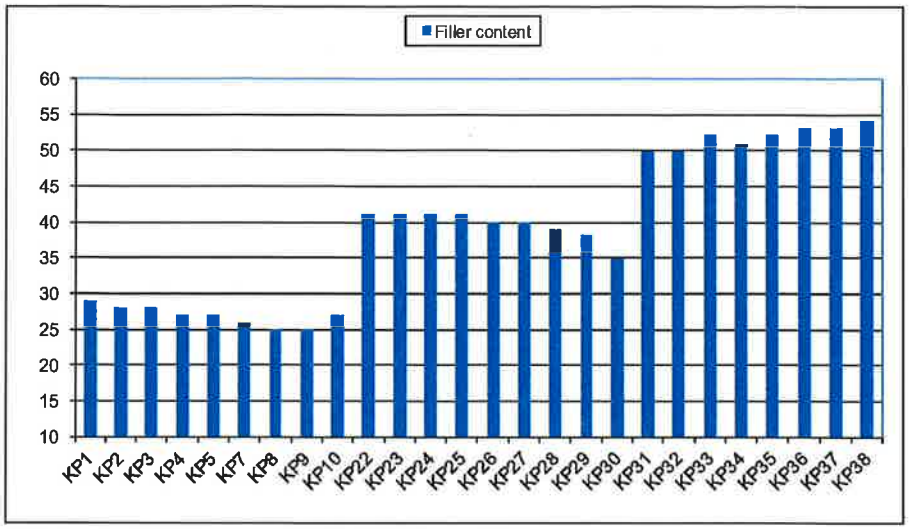


Figure 7. Filler content (%) of trial points

Filler retentions are shown in figure 8. Filler retentions are calculated as shown in part 4.4 as total filler retention, retention of clay and calcium carbonate and their ratio. Total filler retention was ~ 35 % with filler content 28%, ~ 53 % with filler content 41% and ~56 with filler content 51%. Filler retention ratio clay /carbonate decreased as a function of filler content, when the relative proportion of carbonate increased, indicating better retention of carbonate vs clay. The retention of PPH pigment was higher comparing to traditional pigments, which was noticed as higher filler retention in higher filler contents (~54% vs ~35%) (figure 9).

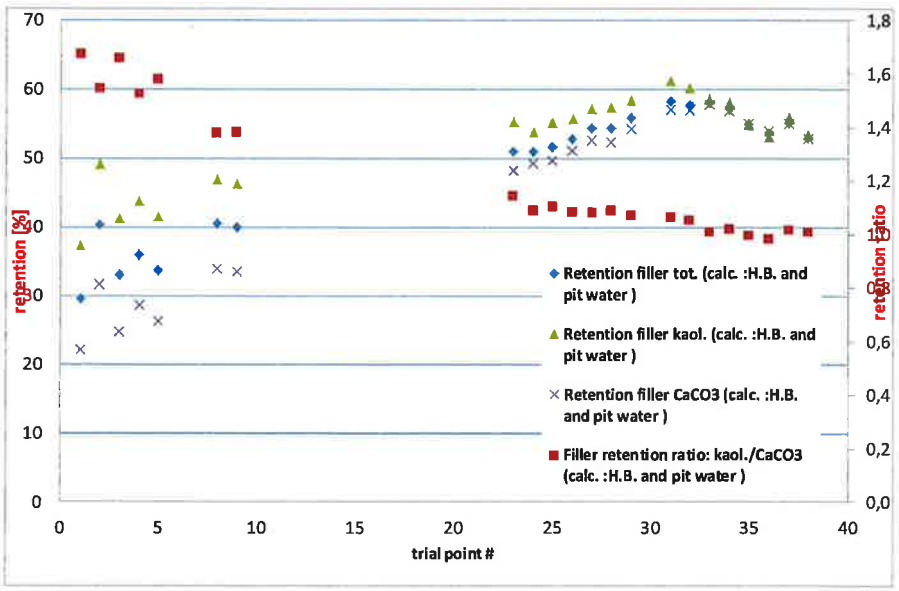


Figure 8. Filler retention as a function of trial points

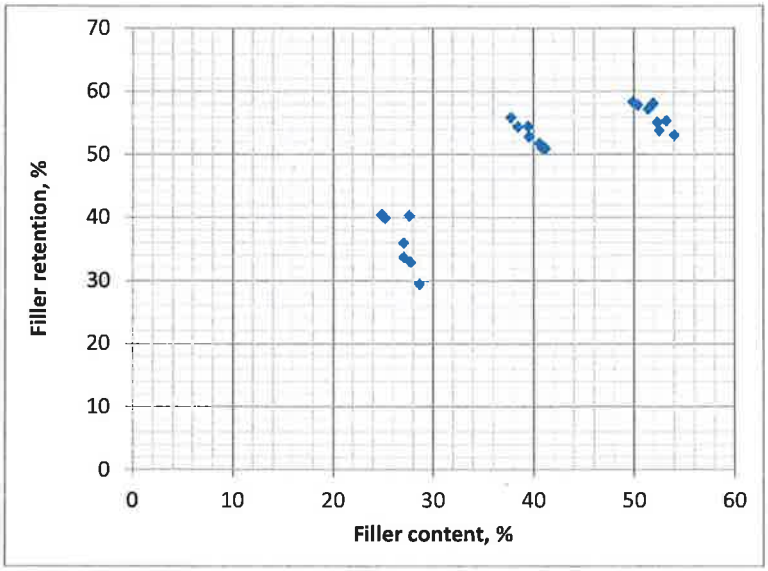


Figure 9. Filler retention as a function of filler content

Filler distribution through the thickness of paper was measured from samples 3, 23, 25 and 34 to get information of the effect of filler content on filler distribution. (figure 10). Also the effect of press nip load on filler distribution in filler level 40 % was studied (samples 23 & 25). In the analysis paper samples were ripped to the layers buy tape ripping method and layers was ashed in the oven 500 ^{0}C. The running parameters in former and press section were same in the samples 3, 25 and 34 being HiVacuum ~— 65 kPa, nip load 1600 kN/m and tilt 2,3. In the sample 23 the nip load was 1800 kN/m. The measured filler contents in the sample 3 was 28 %, sample 23 & 25 41% and in the sample 34 51%.
It can be noticed the filler content was higher in all cases on the bottom (wire) side of the sample decreasing on the top side. The evenness of the distribution was increased as a function of filler content which was expected. Higher nip load increased the evenness (1800 kN/m/ sample 23 vs 1600 kN/m sample 25).

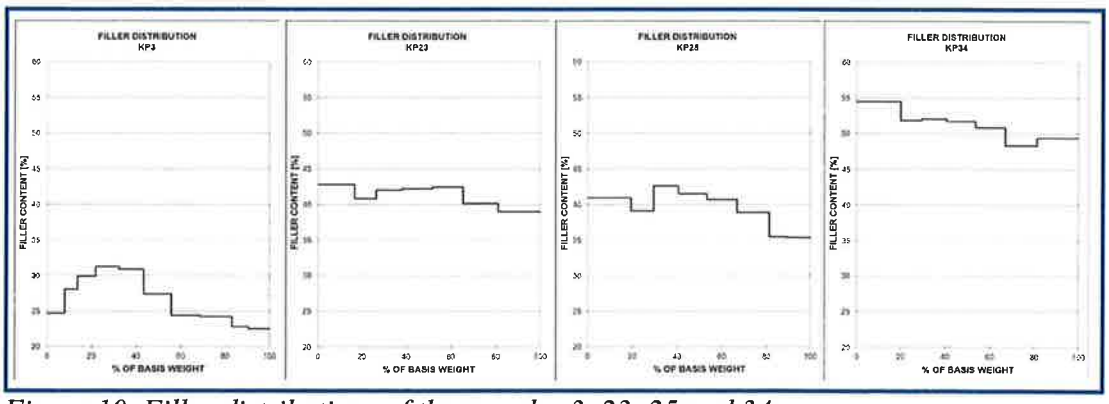


Figure 10. Filler distributions of the samples 3, 23, 25 and 34

Mercury porosimetry analysis was done for same four samples at Omya development lab in Oftringen. At figure 11 are shown the mercury intrusion curves. Samples 3 and 25 have the highest total specific pore volumes, sample 34 the smallest → pore volume decreased as a function of filler content. Higher nip load in press section has an effect on pore volume, decreasing the pore volume (1800 kN/m/ sample 23 vs 1600 kN/m sample 25).

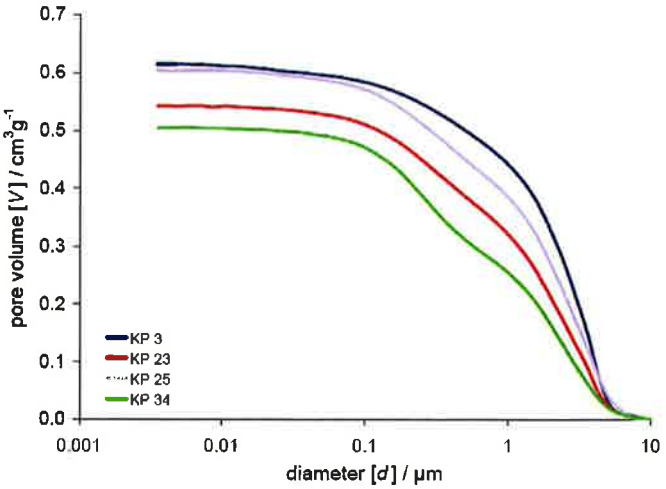


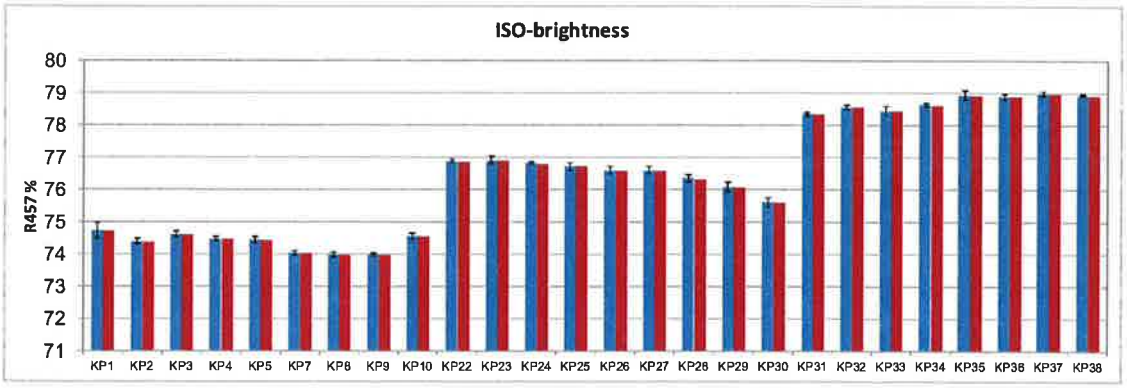
Figure 11. Mercury intrusion curves of the paper samples truncated at 10 µm

5.4 Dry content after former and press section

Dry contents were measured after press section in each trial day and after former section on Thursday and Friday. The dry content increased clearly as a function of filler content. The average solids content after former section at filler level 40 was 20.4 % and at filler level 50 % 22.7 %.
The average dry solids content at filler level 30 % after press section was 45.9%, at filler level 40% 51,0 % and at highest filler level 53.8%.

5.5 Optical properties

ISO –brightness and opacity of the samples are shown in a figure 12. The average ISO-brightness at filler level 30 % was 74.6 %, at filler level 40% 76.5 % and at highest filler level 78.7 %, increasing 2 units from each step. Average opacity at filler level 30% was 92.1 % increasing to filler level 40 & 50%, being 93.9 % in both cases.



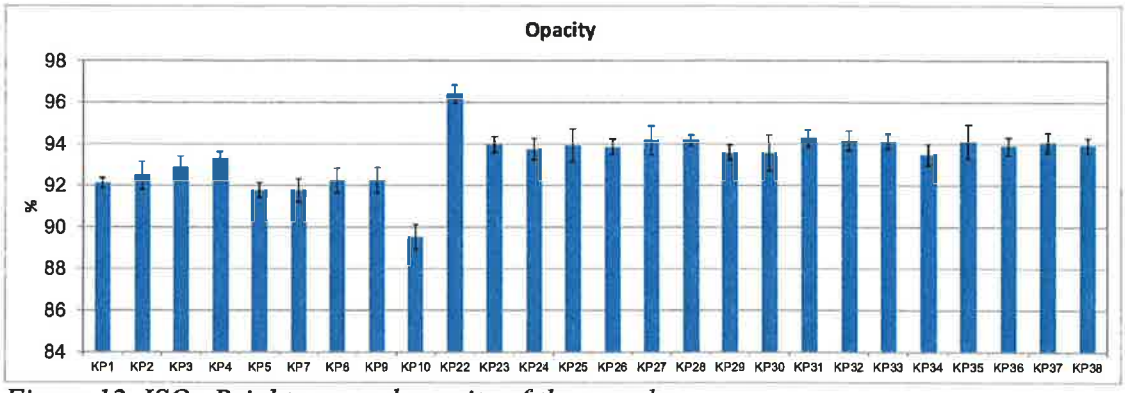


Figure 12. ISO –Brightness and opacity of the samples

ISO-brighness, opacity and light scattering coefficients are shown as a function of filler content (total filler content and as a function of calcium carbonate) in figures 13 – 15. Correlation between ISO brightness and filler content is almost linear.

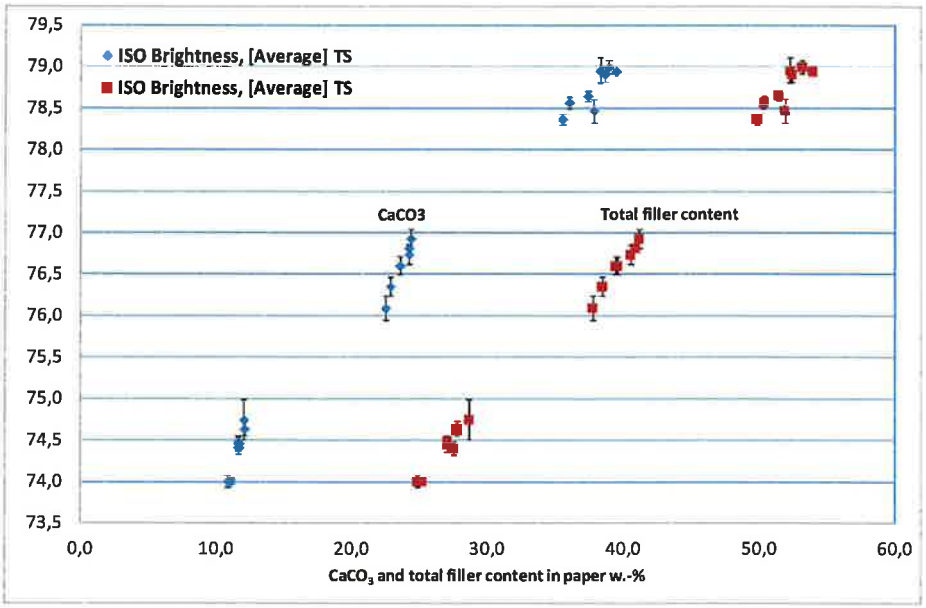


Figure 13. Iso-Brightness of trial points of Tuesday, Thursday & Friday as a function of filler / CaCO3 content

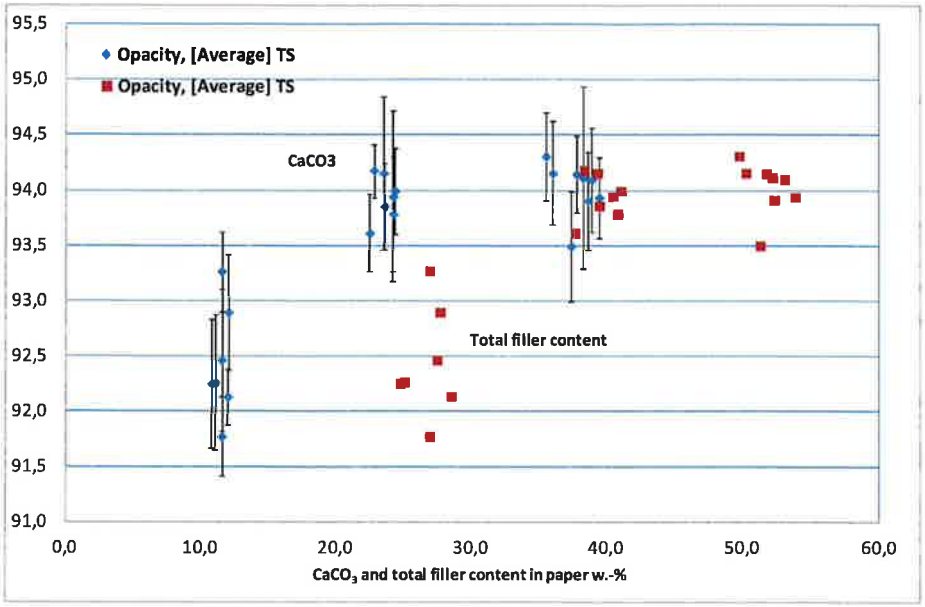


Figure 14. Opacity of trial points of Tuesday, Thursday & Friday as a function of filler / CaCO3 content

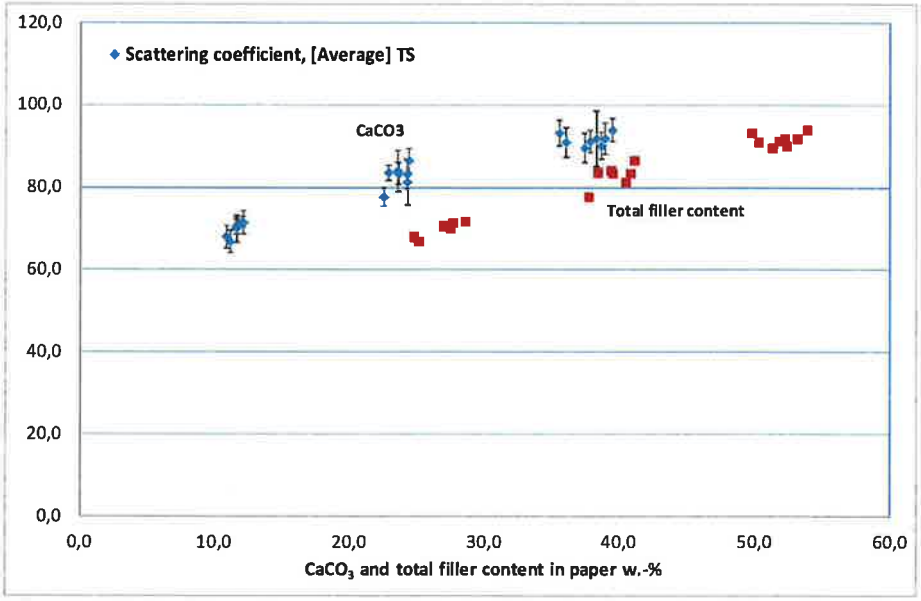


Figure 15. Light scattering coefficient of the trial points of Tuesday, Thursday & Friday as a function of CaCO3 and total filler content.

5.6 Tensile properties / dry samples

Tensile strength properties were measured from the samples. Average tensile index in MD/CD in different filler levels are shown in the table 3.
Tensile index (MD) decreased 32 % when filler content increased from 30% to 40%. The drop is somewhat lower from filler level 40 to 50% being 22%.
Average strain at break (MD) of all the trial points are in the same level being 1.3 – 1.4. Modulus of elasticity (MD) decreased from 2626 N/mm^2 (filler level 30%) → 1949 N/mm^2 (filler level 40%) → 1624 N/mm^2 (filler level 50%).

Table 3. Average tensile index values of the samples of different filler levels

Filler content	Tensile Index (Nm/g) MD	dev	Tensile Index (Nm/g) CD	dev
30	34,8	2	17,9	1,2
40	23,7	1,7	14,1	1,2
50	18,6	1,3	10,1	0,7

Tensile index and modulus of elasticity are shown as a function of filler content (total filler content and calcium carbonate content) in figures 16 – 17.

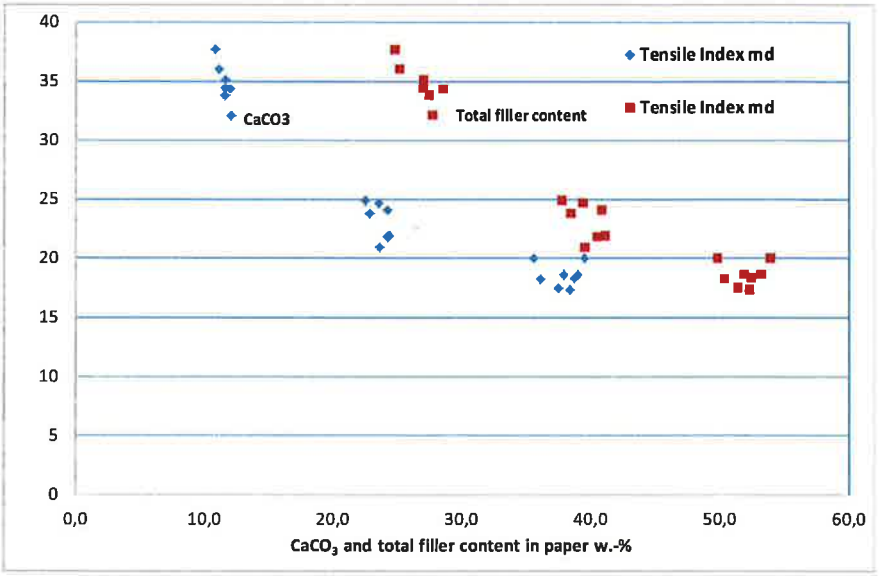


Figure 16. Tensile index (MD) of trial points of Tuesday, Thursday & Friday as a function of CaCO3 and total filler content

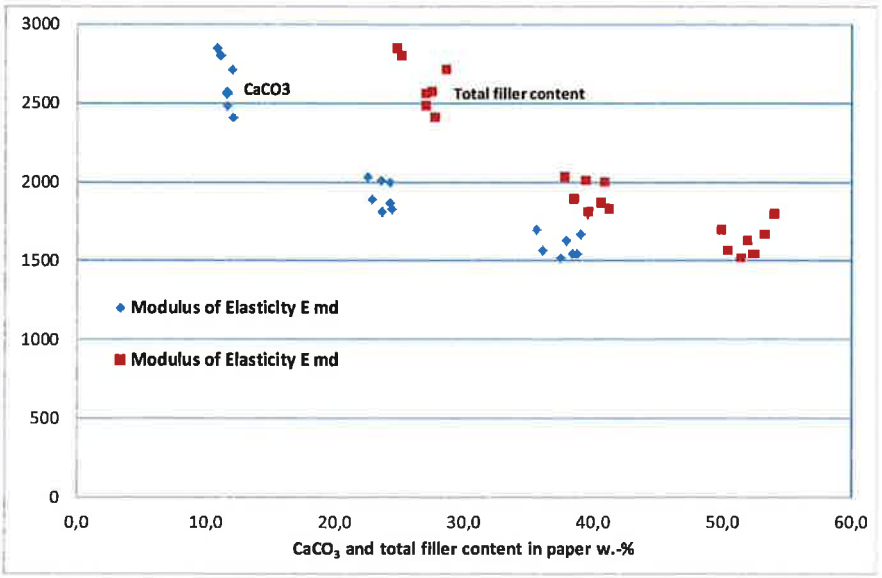


Fig 17. Modulus of elasticity of trial points of Tuesday, Thursday & Friday as a function of CaCO3 and total filler content

5.7 Wet strength & relaxation

Dynamic tensile strength and relaxation properties of the wet pressed samples were measured with Impact device to get information of the runnability of the webs in press to dryer section. In the experiment the strain velocity is 1 m/s, (1000 %/s). The relaxation properties (residual tension) were measured at 2 % strain. Tensile strength and residual tension is shown as a function of solids content in figures 18 & 19. No changes could be defined in strain at break. When comparing the results at dry solids content 50 % (Table 4) it can be noticed that tensile strength decreased 45 % when increasing the filler content 30 % → 40 % and 18% when increasing the filler content 40 % → 50%, for residual tension the decreasing per cents being 31 % and 17 %, respectively. The dry content after press section increased as a function of filler content (as discussed in part 5.4) which compensate to some extent the runnability in press to dryer section. The changes in tensile and residual tension properties have also calculated by indexing the values by fibre grammage of the samples (the effects of fibre bonding on tensile properties) and it can be concluded that the tensile properties decreased 21 % when filler content increased 30 % → 40 % and only 5 % from 40 % → 50%. Residual tension results being 17 % and 4 %, respectively.

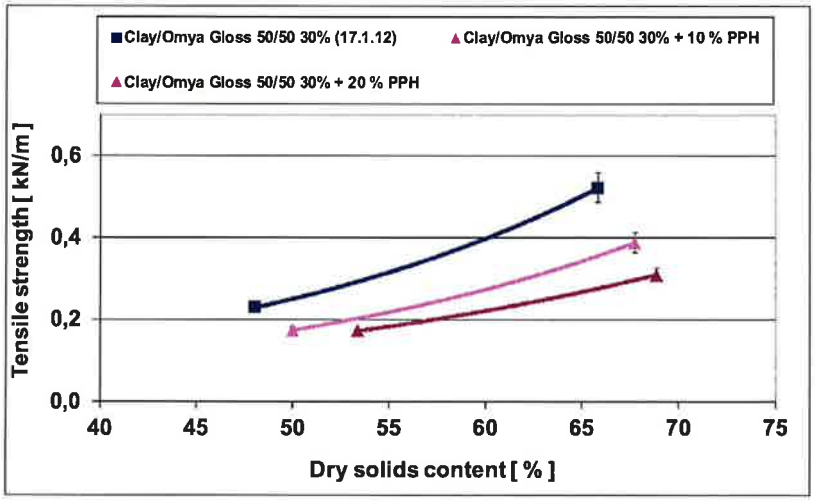


Figure 18. Tensile strength of the samples at different filler levels as a function of dry solids content

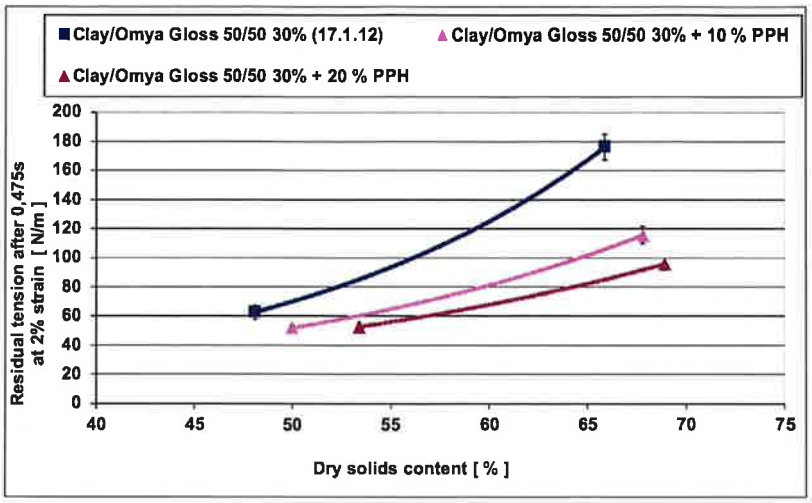


Figure 19. Residual tension of the samples at different filler levels as a function of dry solids content

Table 4. Tensile strength and residual tension at different filler contents as such and indexed by fibre grammage

	Tensile strength at d.s.c. 50 % N/m	Residual tension at d.s.c. 50 % N/m	Tensile strength d.s.c 50 % index by fibre grammage Nm/g	Residual tension d.s.c. 50% index by fibre grammage Nm/g
Filler content 30%	260	74,8	6,8	1,97
Filler content 40 %	170	51,9	5,4	1,64
Filler content 50 %	140	43,3	5,1	1,59

6 Conclusions

The goal of INT – Testaa trial at week 3 – 4 / 2012 was to produce high filler content SC paper, up to 50 % with new type of pigment polymer hybrid filler (PPH 1080) by Omya. Filler blend Clay & Omya Gloss 500 as a mixture 50 / 50 was used as a reference up to filler content 30%. The filler content was increased from 30 % to 40 & 50 % with PPH filler.

The focus was on dewatering in former and press section and paper properties. Variables in the trials besides the filler content was in the HiVac vacuums in the former section and press loads and nip tilt profiles in the press section.

Altogether 38 trial points were done in four trial days. It was easy to reach the high filler contents with this special filler and the machine runnability was good within the whole week.

The results can be concluded:
- Pulps and wet end process stayed stable during the trial week, runnability being good in former and press section
- the average grammage of the trial points were 57 g/m^2, varying between 54 – 58 g/m^2
- Total filler retention was ~ 35 % with filler content 30%, ~ 53 % with filler content 40% and ~56 with filler content 50%.

- The retention of PPH pigment was higher comparing to traditional pigments, which was noticed as higher filler retention in higher filler contents
- The filler distribution through the thickness of the papers showed the higher filler content in all cases on the bottom (wire) side of the sample decreasing on the top side. The evenness of the distribution was increased as a function of filler content which was expected. Higher nip load increased the evenness.
- The dry content increased clearly as a function of filler content after former and press section. The average dry solids content at filler level 30 % after press section was 46%, at filler level 40% 51% and at highest filler level 54%.
- ISO –brightness increased linearly as a function of filler content: the average ISO-brightness at filler level 30 % was 74.6 %, at filler level 40% 76.5 % and at highest filler level 78.7 %.
- Average opacity at filler level 30% was 92.1 % increasing to filler level 40 & 50% to 93.9 %, respectively
- Tensile index (MD) decreased 32 % when filler content increased from 30% to 40%. The drop was somewhat lower from filler level 40 to 50% being 22%.
- The runnability in press to dryer section measured as residual tension decreased roughly same per cent than dry tensile index, but if indexing the values by fibre grammage residual tension decreased 17% and only 4%, respectively.