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A METHOD FOR MEASUREMENT OF CHALK DISTRIBUTION IN PAPER SHEETS

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ABSTRACT

The distribution of filler particles in printing papers increases in importance with increasing filler content. Τn this paper it is shown that conclusions about the chalk distribution can be made from formation measurements on sheets before after the chalk and removing respectively. Chalk can be chemically removed using HC1-Propanol an solution.

Measurements of filler distribution have been made on fine paper containing 35-70% chalk. The sheets were formed in a twin wire, roll former laboratory machine at 500 m/min. A three component dry strength/retention aid system was used. starch, one anionic polymer component including and one cationic polymer component. Both when using a strategy for maximum retention and for optimum formation it was found that the local filler grammage was proportional to the local fiber grammage.

Measurements of formation on base paper and roll coated paper respectively revealed that there is a strong negative correlation between local filler grammage and local fiber grammage, which results in very even formation of the coated sheet.

BACKGROUND

There is a trend for increased use of inorganic filler material in fine paper and other printing paper grades. One of the main drawbacks of mixing filler into the sheet is the detrimental effect on mechanical properties. Filler

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particles will decrease fiber-fiber bonding and therefore reduce the strength potential. The distribution of filler material in a paper sheet is, therefore, of great importance and it is surprising that so little has been done to measure this distribution.

EARLIER INVESTIGATIONS

(1) reports results from measurements Corte on handsheets containing chalk. In his measuring equipment he could record local grammage using beta ray absorption. Initially he measured the grammage within 1 x lmm areas of a sample from a handsheet containing filler. 50 x 50mm The chalk was resolved using HCl-saturated butanol, after which the local grammage measurements were repeated. By comparing the two measurements at each location, Corte found a negative correlation between local fiber weight and local filler weight. This means that filler is preferentially located in low fiber weight areas, and therefore contributes to а reduction in grammage variations.

The sheets were made under standard handsheet contitions. Corte also produced handsheets, starting from significantly lower fiber concentrations. He then found an initial increasing filler retention with increasing This result indicates that the structure of t he dilution. sheet affected filler retention in his case and therefore also could have an effect on filler distribution.

Corte also measured the distribution of coating layers. performed his local grammage measurements on a coated He sample and on the same sample after removing the coating. The coating, applied by air-knife, consisted of gelatine micro-capsules, subsequently removed by treatment with methanol. Also in this case a negative correlation was measured between grammage before and after coating removal, interpreted as "infilling" during the coating which he procedure.

STATISTICAL METHOD OF EVALUATION

At STFI/KTH there is, as yet, no equipment available for reproducible local grammage measurement before and after filler removal, in the way used by Corte. To evaluate the formation of sheets, a formation meter based on beta radiography, continuous scanning and power spectrum analysis (2) is available. Compared to the method used by Corte, the STFI-method permits measurement on larger samples with better geometric resolutions and less time consumption. As local grammage values for individual sample points are not saved for further processing it is however not possible to evaluate the point by point difference before and after removing the filler according to the method used by Corte.

By comparing the power spectra with and without filler respectively, however, it is possible to draw conslusions about the average filler distribution in relation to the fiber distribution. In the following the influence of three different filler distributions on formation are described

- A) even filler distribution
- B) filler distribution correlated to fiber distribution
- C) filler distribution independent of fiber distribution

SUPERPOSITIONING OF FIBER AND FILLER DISTRIBUTIONS

Formation

The formation number F is defined as the coefficient of variation of local grammage

$$F = \frac{\sigma(w)}{w}$$
(1)

where w denotes mean grammage.

In the following, w and F will denote grammage and formation respectively for the fiber component while w $_{\rm f}$ and $_{\rm F_f}$ refer to the filler component.

For the complete sheet, w and F will include both fibers and filler.

The Formation Spectrum

The formation spectrum describes the distribution of grammage variance on different floc sizes $(\underline{3})$. If the spectral density is presented with logarithmic scales, a proportional spectral change over the entire floc size range will simply mean a vertical shift of the entire spectrum.

A. Even filler distribution

Let the filler be distributed as an even layer across the sheet. The variance of grammage will then be unchanged compared to the fiber sheet, but mean grammage will increase

$$F = \frac{\sigma(w_0)}{\overline{w}_0 + \overline{w}_f} = \frac{\overline{w}_0}{\overline{w}_0 + \overline{w}_f} \cdot F_0$$

$$\frac{F_0}{F} = \frac{\overline{w}_0 + \overline{w}_f}{\overline{w}_0}$$
(2)

By shifting the fiber spectrum downwards by a factor equal to the ratio of grammage with and without filler the new spectrum will represent the filled sheet.

B. Filler distribution correlated to fiber distribution

Let there be a perfect correlation between local fiber grammage and local filler grammage. The local grammage of the "fiber sheet" will then increase by a proportionality factor at all points when filler is added. The formation number of the filler containing sheet will then amount to

$$F = \frac{\sigma (a \cdot w_0)}{a \cdot w_0} = F_0$$
(3)

In summary: if the filler is locally added in proportion to the fiber weight, the formation spectrum will be unchanged.

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C. Filler distribution independent of fiber distribution

If two independent variables are added their respective variances are added.

Let the variance of fiber and filler distribution be expressed as follows

 $\sigma^{2}(w_{o}) = k_{o} \cdot \overline{w}_{o}$

and

$$\sigma^{2}(w_{f}) = k_{f} \cdot \overline{w}_{f}$$

The formation of the combined fiber and filler distribution can then be written

$$F = \sqrt{\frac{k_{o} \overline{w}_{o} + k_{f} \overline{w}_{f}}{\overline{w}_{o} + \overline{w}_{f}}}$$
(4)

We define the fiber and filler distributions as similar if

$$k_{o} = k_{f} = k$$
 (5)

Equation (5) is inserted into Equation (4)

$$F = \sqrt{\frac{k}{\overline{w}_{o} + \overline{w}_{f}}} = \sqrt{\frac{\overline{w}_{o}}{\overline{w}_{o} + \overline{w}_{f}}} \cdot F_{o}$$

$$\frac{F_{o}}{F} = \sqrt{\frac{\overline{w}_{o} + \overline{w}_{f}}{\overline{w}_{o}}}$$
(6)

Equation (6) means that the spectrum of the fiber sheet is transformed into that of the filled sheet by a downwards shift equal to square root of the ratio of filled sheet weight and fiber weight.

REMOVING FILLER FROM A PAPER SHEET

Measurements have to be performed of the grammage distribution before and after removing of the filler content. The following method was developed for removing the chalk.

A sheet of size 105 x 173mm was placed on a filter litre of a 3 M HCl-propanol solution is poured paper. 0.15 onto and sucked through the sheet. The solution is to be added very gently to avoid damaging the sheet and to make sure that all the solution is sucked through the sheet. The sheet is then dried on a semi-cylindrical drum of 100mm 80°C using radius. The drum is kept at a temperature of electrical heating-spirals. Α tensioned, fine metal wire presses the sheet against the drum. A drying pressure of 1.7kPa was selected, resulting in a drying time of 2 minutes for a 60 g/m^2 sheet. After this drying procedure, the sheet is plane and without wrinkles, which is important for the subsequent formation measurement. Using the above method, more than 90% of the original amount of chalk in a sheet is removed.

RESULTS

Highly Filled Fine Papers

FEX system at STFI (4) extensive tests have been In the run making high filler content fine paper. Sheets were formed at approximately 500m/min according to the twin-wire forming principle. Using this method, roll the fiber suspension will be subjected to little shear during the actual forming process due to the even dewatering pressure. without stationary (pulse generating) dewatering elements. Several retention aid strategies have been tested. In one of them starch (for dry strength) was combined with one anionic and one cationic polymer component.

In one of the test runs, sheet grammage was 100 g/m^2 , filler ratio 35% and filler retention 83%. Figure 1 shows the formation spectra before and after removing the filler respectively.



Fig 1—Formation spectra for filler containing fine paper with optimum retention (100 g/m², 35% filler) and for the same paper without filler

The two formation spectra in Figure 1 are very similar. The conclusion can then be drawn, according to case B above, that the local filler grammage is proportional to the local fiber grammage. The retention aid system can also be optimized for good formation. Figure 2 shows the formation spectra before and after removing the filler respectively, for a well formed sheet of 80 g/m² with 33% filler.



Fig 2—Formation spectra for filler containing fine paper with optimum formation (80 g/m², 33% filler) and for the same paper without filler

The two spectra are nearly identical. This again indicates that the local filler grammage is proportional to the local fiber grammage. Figure 3 shows beta radiographs of sheets with maximum filler retention (Fig. 1) and optimum formation (Fig.2) respectively.

Wagberg (5) has studied the flocculation progress after the addition of retention aids in a flow loop. He observed that filler is adsorbed onto the fibers, but no separate filler aggregates are formed. Figure 4 shows an example of filler added to the fibers.

The filler distribution in Figure 4 supports the previous result that filler in a sheet is distributed proportionally to the local fiber grammage.



Fig 3—Beta radiographs of fine paper with maximum retention (left) and optimum formation (right). Dark areas represent high grammages. Calibration steps to the left



Fig 4-Addition of retention aids causes filler to adsorb to fibres



Fig 5—Formation spectra for base paper (82 g/m²) and coated paper (124 g/m²). Middle curve represents base sheet with even layer of 42 g/m₂ added

Coating Distribution

In Figure 5 formation spectra for a base paper and the corresponding coated paper are shown. The coating layers are applied using rolls.

The upper spectrum represents the base paper. The middle spectrum represents the base sheet to which an ideally even coating has been added according to principle A in the previous section. The shift in spectrum was calculated using Equation (2).

The measured spectrum of the coated sheet is well below that calculated assuming even coating distribution. This indicates a strong negative correlation between filler and fiere grammage, i.e. "infilling".

The coating method used by Corte (1) was of "air-knife" type, which is known to give an even coating layer. This can explain the rather small evening out effect recorded by Corte.

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Transcription of Discussion

A Method for Measurement of Chalk Distribution in Paper Sheets

by B. Norman and O. Axell

Dr. D. Caulfield USDA, Forest Prod. Lab, Madison, U.S.A.

I agree that a Beta ray emitter is the only way to get density distribution measure meaningful mass of а formation. If formation is taken to mean the visual appearance or look through, then that measurement contains valuable data that we don't quite understand yet. Because involves light scattering phenomena, it tells it us about surface distribution and also about something connectivity of mass distribution in the Z-direction. As Dr. Baum pointed out yesterday, this is a very important point.

Prof B. Norman I cannot see how you could evaluate Z-direction variations from a planar picture like that. The most important variable is the local grammage. If, to this, you add other things such as local structuring, absorbtion and other parameters, this is even more useful. If you do not know the local grammage and have to rely on optical things which are calender dependent, then you are on very loose ground.

Prof J. Marton Westvaco, Laurel, U.S.A.

You showed two transmission pictures of two differently formed papers. Did you improve the formation using chemistry or had one deteriorated less than the other?

Norman The good paper with good formation was one order of magnitude better than normal paper and the other one was worse.

Dr. R.W. Davison Hercules, Wilmington, U.S.A.

Regarding the Figure 4 showing the flocculated filler attached to pulp fibres, was that taken from the Wagberg paper in reference 5, or did you take that picture yourself?

Norman Yes, the photographer is here and you can talk to him later.

Davison What was the stirring speed for the pulp fibre slurry when these aggregates of filler are apparently attached to the fibre? It must be very low for this to occur.

Norman It is a pipe loop with a flowing suspension at speeds of 2 m per second in this particular experiment, but you can discuss the hydrodynamic conditions with Wagberg later.

B.A. Thorp James River, Richmond, U.S.A.

What is the basis weight of the two sheets in Figure 3 on page 585?

Norman Somewhere between 80 and 100 g/m^2 , the better formed sheet was slightly heavier.

A. Komppa Jaakko Poyry Oy, Helsinki, Finland

We have determined filler distribution in the sheet using titanium dioxide and clay as a filler. We punched out small pieces of paper 1 mm in diameter and determined the weight of the pieces before and after ashing them. The pieces were cut from the flocs and from between the flocs from the web in general. Three different forms of and filler distribution were noted. First, ash content was larger in the flocs than in between the flocs. Secondly, where ash content was smaller in the flocs and thirdly, where ash content was the same all over the sheet. Can you comment on that?

There are two factors that are very important Norman for The only reference we found was by Corte this. who detected a negative correlation between filler and fibre. He used a well diluted fibre suspension with no retention aid. In that case, the sheet was acting like a filter medium for the filler, therefore it is only natural that more filler will collect in the lower weight areas where the drainage is higher. In the case you quote, I am not sure what sort of hydro dynamic situation you used. You can get different results with different forming methods and different retention chemicals, so I don't see anv in these papers and contradiction between what we found what you say. We have not measured point by point. It is only a way of comparing two spectra and they can be representative of a percentage increase in grammage by the However, statistically, there could be other filler. distributions of filler to give the same spectrum, but we do not think it is very likely that this is so in our case.

A. Ibrahim Papyrus Inc., Westerville, U.S.A.

I would like to learn more about the chemistry you used in Figure 3. Could you elaborate on what kind of chemicals you use, how much, where and so on.

Norman I cannot comment on this, perhaps you could ask Dr. Lindström after the next paper.

R.W. Dent Albany International, Dedham, U.S.A.

The calculation of formation value implicitly assumes that the standard deviation is proportional to the basis weight. If I have two papers of different basis weights, is the formation comparable if the formation value is the same?

Norman You cannot say that changing the grammage will affect the formation, because many other things could happen, such as changes in consistency or dewatering characteristics so you cannot simplify things in the way you just did.

Dent So how would you compare the formation values of papers of different weights?

Norman If you want to compare them at one reference weight, you multiply both formation values by the square root of the grammage. The higher value will be the floccier sheet. This is a statistical rule, if the sheets are the same, they should end up with the same number. It is just a statistical comparison.

V.W. Punton Wiggins Teape R & D, Beaconsfield, England

have been making direct measurements of filler We distribution in paper using Beta and Alpha radiography. The total mass distribution in the sheet is determined by Beta radiography. The mass distribution of the filler -Titanium dioxide, chalk or clay - is measured directly from radiography of the sample. fibre Alpha The mass distribution is then found from the difference between these two distributions.

We have examined a variety of production and experimental paper samples.

Our results have indicated that the correlation between fibre and filler distribution is dependent on such factors as;

prevailing wet-end chemistry conditions, state of fibre flocculation and the drainage characteristics of the paper machine concerned.

Depending on those factors, one can have fibre/filler distribution correlation co-efficients ranging from significantly negative, through zero, to strongly positive.

Our experience with samples, in which cationic starch and anionic polyacrylamide are used, is that the filler/fibre distributions show a strongly positive correlation.

That was certainly not the case in many other production samples we have examined where negative or zero correlations have been seen.