Weighing of Different Impact Factors on Wet Web Strength by Full-Factorial Design of Experiments

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The wet web strength is one of the most important parameters for effective paper machine performance. A huge variety of parameters is known from many studies to have an impact on the wet web strength (WWS). In this study, a full factorial design of experiments (DOE) was used to determine the effect of different factors on the WWS. The goal was to use a DOE method within the field of paper strength research to acquire advanced information on the mechanisms of strength development at different dryness levels. The study was carried out with laboratory handsheets made of commercial unbleached softwood pulp, which was refined in a laboratory Hollander beater. The WWS was measured according to the German standard DIN 54514. The analysis of the data showed that weighing of the influencing factors on WWS is possible, which may lead to a better understanding of paper strength development mechanisms at low dryness levels. The applied method was proven to be reliable for the determination of the impact of various factors and will therefore be used in future work.

Keywords: Beating; Wet web strength; Method; pH value; Design of experiments (DOE)

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INTRODUCTION

For high paper machine effectiveness, wet web strength is one of the most important parameters. This is not only implied by the literature (Guldenberg *et al.* 2004; Edvardsson and Uesaka 2009; Miettinen *et al.* 2009; Lindqvist *et al.* 2012), but can also be seen in the current problems in paper production plants.

The WWS is defined as the magnitude of force that is required to break a wet paper web. In general, the term “initial wetness” comprises solid contents from about 10%, in or after the forming section, to 60%, when passing the first drying cylinder. Dry contents of about 10% are mentioned in the scientific literature until about 1960. In this case, these were values from the forming section or laboratory sheet formers. Today, the lower limit of the “initial wetness range” ranges from 18% up to a maximum of 25%, as these levels are achieved in modern forming sections. Initial wetness depends on the fabric used, the forming section design, the use of additives, and the raw materials (Strauß 2008). The upper limit at dry contents of about 60% is based on the strength level of the paper. The paper is strong enough to pass the machine draws at this dry content without breaking.

The strength properties are highly influenced by the dry content of the paper. The wet web strength is no exception. As already indicated by Brecht (Brecht and Erfurt 1959a), with respect to the work of Lyne and Gallay (1954a), different forces are responsible for the strength development of a paper sheet at various dry contents. In recent times, different authors (Wågberg and Annergren 1997; Hubbe 2006; Edvardsson and Uesaka 2009; Tejado and van de Ven 2010; Wågberg 2010) pointed out these dependencies. Single phenomena are able to superimpose their effects. Normally, at a certain dry content with defined solid composition, several forces with different impacts are involved in the strength development. Based on this fact, it is in each case necessary to consider the measured strength in relation to the dry content. According to the paper composition, concerning raw materials and additives, the dry content will be influenced – even if all parameters for sheet forming and pressing are constant. An increase of the WWS due to an increase of the dry content could be mistakenly attributed to the fibre and filler composition or to additives' effects.

In 1959, Brecht defined four stages of strength development, all of which greatly depend on the dry content of the paper web (Brecht and Erfurt 1959a; Brecht and Erfurt 1959b). One outcome of the analysis is that for some pulp mixtures at special boundary conditions, the second stage vanishes and only three stages are recognisable. Page developed an equation to calculate the WWS of paper on the basis of capillary forces according to Young-Laplace (Page 1993). Tejado and van de Ven considered this equation insufficient because surface tension and wetting forces are not included (Tejado and van de Ven 2010). Through further experimental work, the "Entanglement Friction Forces Theory" was developed (de Oliveira *et al.* 2008; Tejado and van de Ven 2010). This theory postulates that entanglement and matting between the fibres keeps the wet paper web together and that these forces are primarily responsible for the WWS. These forces are much stronger than the existing capillary forces. With further pressing, hydrogen bonds (H-bonds) are gradually formed and are reinforced by the collapse of the fibre lumen. Because of this collapse, the fibres change from round shapes to flatter ones, which results in a larger contact area between the fibres. This paper omits some aspects, such as the fibre-water gel structure (Wågberg and Annergren 1997), the interactions due to the surface charge of the fibres, and acid-base interactions (Barzyk *et al.* 1997).

Almost parallel to the work of Tejado and van de Ven (2010), Erhard *et al.* published a three-stage theory on strength development (Erhard *et al.* 2010). In the first stage up to a dry content of about 25%, capillary forces and mechanical entanglement of the fibrils are primarily responsible for the approach of the fibres and hence for the resulting strength (Kendall 2001). The morphology of the collapsed fibres enables an approach and initial contact points. The fibre characteristics orthogonal to the fibre longitudinal axis (*e.g.* coarseness) has an effect on fibre collapse and flexibility (Paavilainen 1993a; Paavilainen 1993b; Weise *et al.* 1996). In the second stage, up to 50% dry content, the van der Waals forces of attraction and repulsion begin to act according to the DLVO theory (Derjaguin and Landau 1941; Derjaguin 1954; Pelton 1993; Wågberg and Annergren 1997). In addition, a flexible, visco-elastic and soft fibre surface is needed for the formation of larger contact areas between the fibres (Pelton 1993; Nilsson *et al.* 2000). These properties promote the diffusion of polymer chains and polyelectrolytes from wood polysaccharides, especially from xylan (McKenzie 1984; Pelton 1993).

The third stage is the development of dry strength, which is not relevant for the field of WWS research.

Extensive research has already been carried out to increase the wet web strength of paper, considering that fibre characteristics play a major role in strengthening paper at low dryness levels. These studies can be divided into three different areas. The first class of studies investigates the properties resulting from wood type and pulp processing. The second class of studies examines the composition of different pulp types, while the main topic of the third class of studies is pulp refining to improve the WWS.

Early work was carried out with ground wood and sulphite pulp mixtures (Brecht and Klemm 1952; Brecht and Langer 1953; Brecht and Erfurt 1961). It was determined that a pulp mixture gives, in all instances, higher WWS compared to the values of its single components. This was also confirmed in some newer publications with actual pulp qualities (Corson 1979; Seth *et al.* 1984; Luukko 1999; Schwarz and Bechtel 2003; Klein 2007).

An improvement of the WWS can be generated by refining pulp to a certain extent (da Silva *et al.* 1982; Szeiffova and Alince 2003; Klein 2007; Lindqvist *et al.* 2011).

In recent years, much effort has been expended in testing and developing additives based on conventional and renewable resources. For example, cationic aldehyde starch (Laleg and Pikulik 1991; Laleg and Pikulik 1993; Retulainen and Salminen 2009), guar gum (Weigl *et al.* 2004; Oksanen *et al.* 2011b; 2012b), CMC (Klein 2007), and chitosan (Laleg and Pikulik 1992; Borchers 2004) are able to affect the WWS positively when added to the approach flow or as a sprayed wet end application. Another research group developed vinylformamide copolymers to increase the WWS with good results (Esser *et al.* 2008; Gels *et al.* 2012). In addition, retention aids and surface-active chemicals have an impact on paper sheet dewatering and thus also on the WWS (Alince *et al.* 2006; Lindqvist *et al.* 2012). On the other hand, additives that influence the fibre swelling are also options for obtaining better WWS (Laine *et al.* 2000; Laine *et al.* 2002; Koljonen 2004; Horvath 2006).

This short review demonstrates that the task of influencing the WWS is multi-dimensional. In the cited articles, the relationship between parameters is mostly tested and evaluated in a two-dimensional manner. The influence of interactions is neglected. The knowledge on the interactions and the effects of the above-mentioned parameters and additives is still limited — especially the factors that depend on paper dryness. In this paper, a method based on design of experiments (DOE) is presented to answer the questions of multi-dimensional evaluation and of weighing the influencing parameters on WWS. To show the performance of the method, this paper focuses on the parameters' dryness, pH, and dewatering resistance, measured as Schopper Riegler (SR). Besides the dryness and the degree of refining especially the parameter pH was selected to study the different mechanism between dry and wet web strength. For dry strength it is already known that increasing pH leads to increased dry strength because of better flexibility of the fibres showed via the water retention value (Lindström 1980; Lindström and Kolman 1982). The question rises whether this could be also the case for wet paper web or is it possible to see a different mechanism of strength development in the paper at low dryness? This paper will give researchers, as well as practical paper workers, an awareness of multi-dimensional evaluation of wet web strength.

EXPERIMENTAL

Pulp Preparation

Unbleached softwood kraft pulp (Monopol V, Mondi Frantschach, Austria) was used. It was pulped with Munich tap water and refined with a Voith laboratory valley beater according to TAPPI T200 SP01 to 20 SR and 31 SR.

pH Adjustment

The pH values were adjusted with 1 M hydrochloric acid (HCl) to pH 1 or pH 4.5, or with 1 M sodium hydroxide (NaOH) to pH 8.

Sheet Preparation

Sheets were formed with the Retention and Drainage Analyzer (RDA) from Company Frank PTI (Lee *et al.* 2010; Ryu and Bong-Keun 2011). This special sheet forming device was chosen to enable proper and reliable sheet forming with only 1 L of stock solution. This is advantageous for working with adjusted pH values and avoiding a washing effect, which could occur if the sheets were prepared with conventional sheet formers.

Dry Content Adjustment

To adjust the sample dryness to 20% and 35%, filter paper and a Rapid-Koethen couch roll were used. For preparing samples with 55% dry content, a laboratory roll press by Sumet-Messtechnik was used. To obtain reproducible and solid values, the samples were pressed within a defined sandwich on a support plate (Fig. 1).

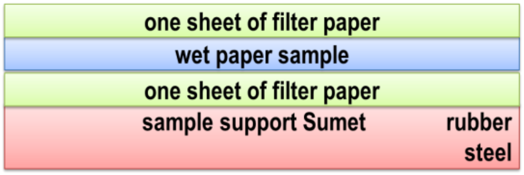

Fig. 1. Defined pressing sandwich and support plate

Measurements

The wet web strength measurement was conducted on the basis of the German standard “Prüfung der initialen Nassfestigkeit” (DIN 54514 2008).

For the measurement, a vertical tensile testing machine from Zwick was used. Immediately before each measurement, the test strip was weighed to determine the real dryness of the paper for the evaluation. A special clamp with a grooved role was designed to ensure that the strip is not wedged in direct above or below the free clamping length. The clamping force was distributed via a 90° redirection on the grooved role, ensuring that no water was pressed in the span. The tensile strain measurement was performed with a speed of 1.5 mm/s until the strip ruptured (Schwarz and Bechtel 2003).

The results of the WWS measurements were used to calculate the value of F_{index} (see Eq.1):

$$F_{index} = \frac{\frac{Fmax[N]}{test\ strip\ width[mm]} \times 10^3}{basis\ weight\ of\ test\ strip[\frac{g}{m^2}]}\ [\text{Nm/g}] \tag{1}$$

Process of Sheet Preparation and Measurement

At first, 360 g of oven-dry pulp was suspended in 23 L of tap water and beaten. Afterwards, the pulp was diluted and stored in batches in 15-L dispensers. There, the pH value was adjusted to the required level. Then, the sheets were formed. According to DIN 54514, the handsheets were stored for a minimum of 12 h in plastic bags at 7 °C. This provides moisture profile equalisation over the whole sheet structure. After moisture content adjustment, the sheets were stored for at least 12 h at 7 °C. Afterwards, test strips were prepared and the dry content was adjusted precisely. Tensile measurements were then carried out.

Design of Experiments

To obtain a valid procedure for the laboratory trials, a full factorial design of experiment was developed with the software Modde 9.1 from Umetrics. This software was also used to evaluate the generated data. Figure 2 shows the experiment trial area.

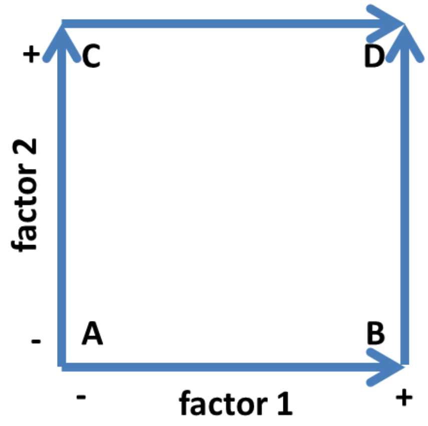

Fig. 2. Trial system

The following equation (Kleppmann 2011) was used to calculate the factor weighing of the two changed parameters.

$$effect_{factor\,2}[\frac{Nm}{g}] = \frac{C+D}{2} - \frac{A+B}{2} \tag{2}$$

It is understood that:

$\frac{C+D}{2}$ is the mean value of results at factor 2 level + (high).

$\frac{A+B}{2}$ is the mean value of results at factor 2 level – (low).

For the evaluation of a DOE, the following parameters are important (Eriksson *et al.* 2008):

Condition number: The condition number represents a measure of the sphericity of the design. All values below 3 are a measure of a good design for screening of robustness tests like those used in this work.

- Variation R^2: Shows the variation of the measured values (goodness of fit).
- Variation Q^2: Expresses the variation of the response predicted by the model according to cross validation (goodness of prediction).

The closer the values of R^2 and Q^2 are to 1, the better the fit of the measured values and the resulting model.

- Model validity: This value is a measure of the validity of the model. When the model validity is larger than 0.25, there is no lack of fit of the model, so the model is valid.
- Reproducibility: This number shows the variation of the responses. A value greater than 0.5 shows a small pure error and a good control of the experimental procedure.

Approach

It is well known that the WWS is influenced by surface phenomena like capillary forces, acid-base interactions (Lindström 1980; Lindström and Kolman 1982), polarity surface energy (Lyne and Gallay 1954a), and surface roughness of single fibres (Leporatti *et al.* 2005; Persson *et al.* 2013). In addition, the morphology of the fibres affects the WWS (Brecht and Klemm 1952; Lyne and Gallay 1954b), as does fibre swelling (Brecht and Erfurt 1959a). Some authors state that the fibre morphology influences the strength much more than the surface phenomena do (van de Ven 2008; Tejado and van de Ven 2010). However, it is not clearly stated to which extent these forces influence the WWS at certain moisture contents.

To get an idea of how this mechanism operates, the degree of refining and the pH were varied. These parameters worked as sum parameters. The degree of refining is responsible for several fibre morphological changes, such as fibre length, fines, broken fibres, and fibril content. A change in the pH value affects the fibre surface charge, the degree of swelling, and the conductivity in water. It is obvious that there is an additional intersection if the pH-value and the beating degree are changed (Fig. 3).

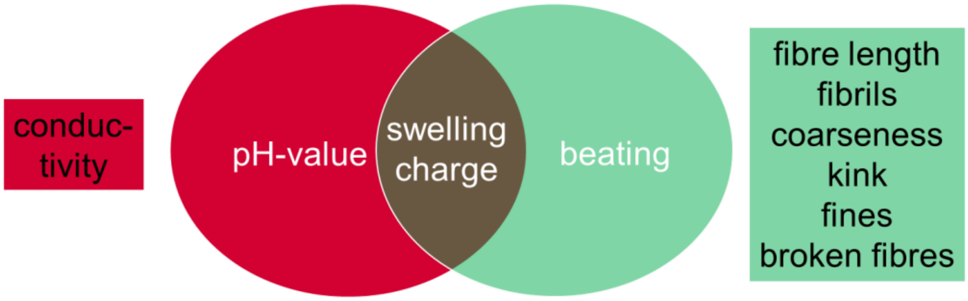

Fig. 3. Intersection model of refining and pH value

With this background information, the design of experiments leads to the design region in Figure 4.

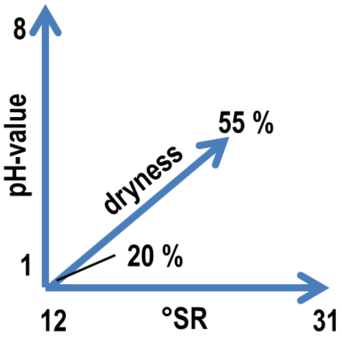

Fig. 4. Trial design region

The order of measurements and adjustments was as follows: At first the value of SR was determined immediately after the valley beater to indicate the extent of refining. The pH was then adjusted, shortly before sheet forming. Thirdly, the dryness was modified directly before measuring the paper strength.

RESULTS AND DISCUSSION

The evaluation of the measured values resulted in the following key outcomes:

Condition number of the model:	1.27719
Number of experiments:	250
Explained variation R^2:	0.98
Predicted variation Q^2:	0.91
Model validity:	0.533
Reproducibility:	0.998

WWS Affected by pH and SR Variation

As mentioned above, changing a single parameter affects at least two others. For further interpretation, the following interactions should be considered:

- increasing pH increases the degree of swelling and the negative zeta potential value of the fibre,
- increasing degree of beating increases the degree of swelling and the surface area of the fibrous material

With respect to existing knowledge, these results can be interpreted as follows: the results of the DOE evaluation show in Fig. 5 that pulp refining up to a certain degree has a positive effect on WWS, as had been established in earlier investigations (da Silva *et al.* 1982; Retulainen and Salminen 2009). There is no change in WWS due to the pH change at a low dryness level. This is different to what is known from dry paper.

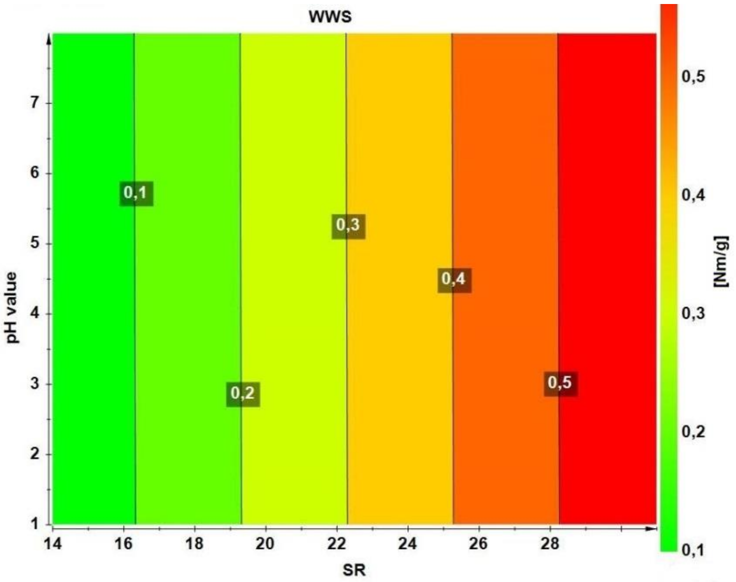

Fig. 5. Development of WWS [Nm/g] depending on pH/SR value at 20% sheet dryness

The strength of dry paper increases with increasing pH value (Lindström 1980) because of increasing fibre swelling. This leads to a better contact between fibres and enhanced hydrogen bonding between fibres. This effect cannot be seen with high water content in the sheet. At this dryness level, only one kind of frictional connection is acting (de Oliveira *et al.* 2008).

Figure 6 shows the responses of the WWS at a dryness level of 55%. These results are in agreement with the model mentioned by Lindström (Lindström 1980): higher pH values cause fibre swelling and softening, resulting in a higher rate of entanglement. That means on the one hand macromolecular and molecular entanglement of cellulose material (Casey 1960; Voyutskij 1963; Kibblewhite 1973; Clark 1978; McKenzie 1984; Neuman 1993; Pelton 1993; Persson *et al.* 2013), and on the other hand that the more flexible fibres will become more entangled with each other during sheet formation (Nanko and Ohsawa 1989; Nilsson *et al.* 2000). In combination with a lower amount of residual water, this results in an increased amount of hydrogen bonding between the fibres and higher WWS.

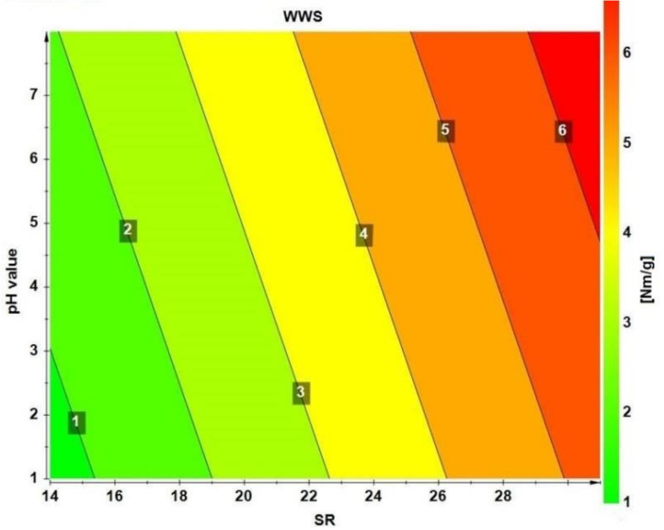

Fig. 6. Development of WWS [Nm/g] depending on pH/SR value at 55 % sheet dryness

Figure 7 shows the development of WWS depending on the pH value and dryness level at a SR value of 31.

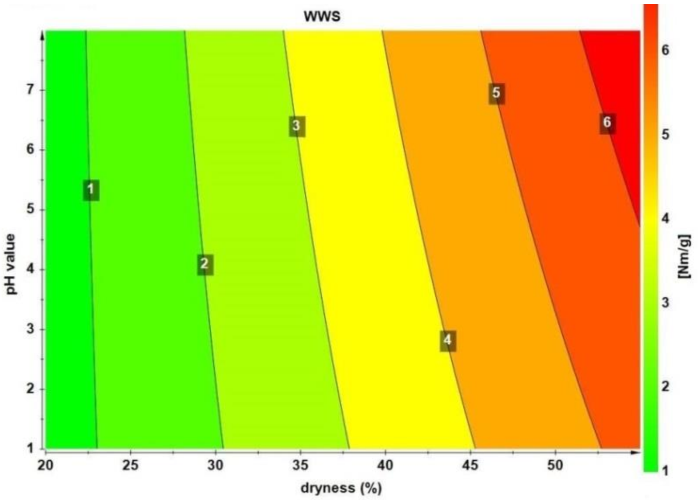

Fig. 7. Development of WWS [Nm/g] depending on pH and dryness at 31 SR

Due to the multi-dimensional evaluation with DOE, it is possible to clearly see the dependency of an increasing pH with increasing dryness. Because the pH value affects the fibre swelling as well as the surface charge and the conductivity, these factors did not affect the WWS at low dryness levels of around 20%. This indicates that only the entanglement between the fibres affected the WWS, as Oliveira *et al.* and Tejado and van de Ven stated earlier (de Oliveira *et al.* 2008; Tejado and van de Ven 2009; Tejado and van de Ven 2010). The WWS at lower SR values showed similar results for increasing pH and dryness.

Weighing of Interactions and Effects

As mentioned above, the weighing of interactions can be calculated throughout the DOE analysis. The results for low and high pH and SR at a dryness level of 55% are:

$A_{(\text{pH 1; SR 14})}$: 0.6 Nm/g
$B_{(\text{pH 1; SR 32})}$: 1.9 Nm/g
$C_{(\text{pH 8; SR 14})}$: 5.3 Nm/g
$D_{(\text{pH 8; SR 32})}$: 6.6 Nm/g

The results were put into Eq. 3 to calculate the effect of a SR change:

$$effect_{°SR}\left[\frac{Nm}{g}\right] = \frac{5.3+6.6}{2} - \frac{0.6+1.9}{2} = 4.6\ Nm/g \tag{3}$$

The complete results for this investigation of the WWS in Nm/g are summarised in Table 1.

Table 1. Summary of Calculated Effects on the WWS [Nm/g]

Dryness	SR	pH	Interaction SR*pH
20%	0.6	0	0.3
35%	2.3	0.6	1.1
55%	4.6	1.3	0.7

Weighing of Interactions and Effects

The calculated values clearly show that the effect of the SR value was much greater than that of the pH value. There was no influence of the pH value at 20% sheet dryness. The interaction effects of SR and pH value were low.

Figure 8 shows the development of the WWS in relation to the interaction effects of pH and SR values at 55 % dryness. The impact of refining is apparent. The interactions between pH and SR can therefore be neglected.

Figure 9 shows the weighing of effects as absolute values of the WWS change. Refining had the most important impact. A pH adjustment from pH 1 to pH 8 at the dryness level of 20% had no effect, and at 35% and 55%, it led to an increase in the WWS.

A dryness level increase from 20% to 55% caused an approximately ten-fold increase in the WWS.

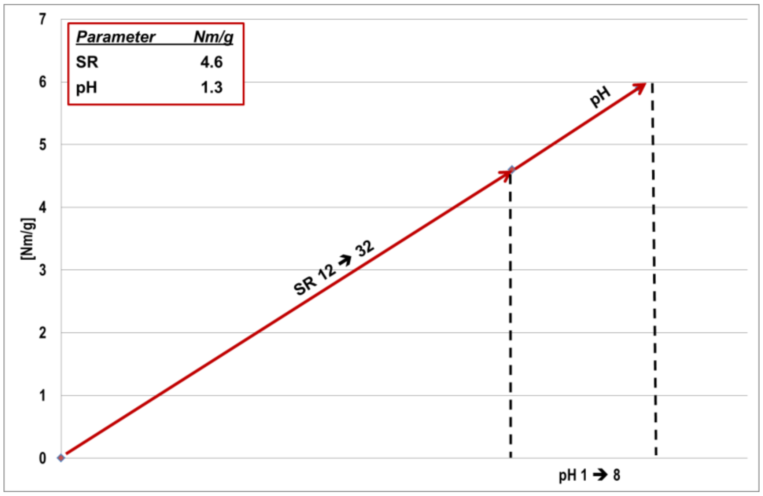

Fig. 8. Development of WWS at 55% dryness related to the interaction effects of pH and SR

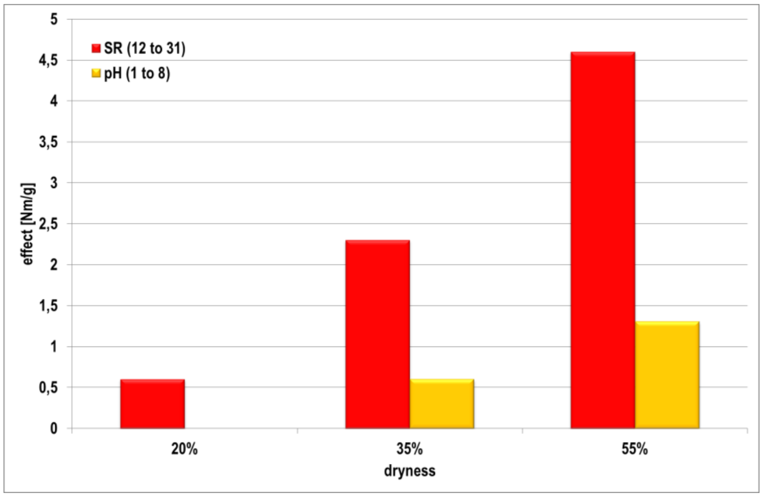

Fig. 9. Weighing of effects of WWS development at certain dryness levels

CONCLUSIONS

1. Using design of experiments (DOE) and its various possibilities to analyse the data is beneficial for evaluating the multi-dimensional reflection of the WWS phenomenon and shows among other things the weighing of the varied factors. This method enables researchers to find the dependencies of different parameters on WWS, including the interactions and the weighing of these factors. With this method, it is possible to ascertain clearly the extent to which a single parameter influences the WWS. In conclusion, it can be stated that DOE is a powerful method not only for product development but also for scientific work.
2. The use of DOE showed, in this experimental design, that pH value, with all of its influence on the fibre chemistry, had no impact on the WWS at a dryness level of 20%, as postulated by Tejado and van de Ven (2010).

3. By increasing the dryness to around 25%, the influence of the pH value was beginning to behave as it is known from the literature for dry paper (Lindström 1980).
4. An increasing dryness level as well as an increasing SR value had a major positive impact. Therefore, it is important to consider that an increasing SR value increases the water retention value, leading to more difficulties attaining a certain dry content.
5. In future work, this method will be used in different investigations to obtain more holistic information on the mechanisms that hold the paper structure together at low dryness levels and to determine how to prepare the stock to obtain higher strength with less effort.

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