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THE EFFECT OF BEATING ON THE SOLUBILITY OF PULPS IN SODIUM IRON TARTRATE

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Introduction

IT has long been standard practice to evaluate and assess the usefulness of a papermaking pulp by beating it in a laboratory beater for increasing periods of time and examining the pulp and sheets of paper made from it as the beating process continues. The examination of the pulp can include the inspection under a microscope, fibre fractionation, the Schopper–Riegler evaluation or equivalent, and the water retention value using a standardised centrifuging technique. The examination of the sheets can include any of the physical tests that are of interest for the particular use to which the pulp is to be put. In particular, the rate at which the wetness (in °SR) increases with the beating time is called beatability of the pulp. For example, sulphate pulp is generally more difficult to beat than sulphite pulp, and cotton linters are more difficult still.

This method of pulp evaluation is not only very time consuming, but also does not tell the papermaker much about how best to treat his pulp in the stock preparation. There is a need for a simple method to examine an unbeaten pulp and to assess its behaviour in a beating process without actually carrying out the beating. Such a method can be based on the solubility of the pulp in a suitable solvent. The condition for the success of such a method is that it is sensitive to those structural features of the fibre which affect the beating and to the changes which they suffer during this treatment.

The structural features of the fibre most sensitive to the beating action whilst contributing to the paper properties, are the elements which influence the swelling phenomena in water. Moreover, they are responsible for the solubilisation of low polymers (hemicelluloses) in NaOH-solutions.

Under the chairmanship of J. Mardon

Beating and solubility of pulps

Jörgensen's work[1] on the 'chemistry of pulp fibres' and Stephansen's[2] on 'analytical beating of pulp' have guided our experiments.[3, 4, 5]

It is well-known that the WRV (water retention value) suggested by Jayme[6] in 1944, i.e. the ability of pulps to retain, under dynamic conditions (e.g., using a standardised centrifuging technique) part of the swelling water, is a significant indication of the interaction of water with cellulose fibres, mainly depending on the chemical composition (hemiculluloses). The significance of this test for the characterisation of unbeaten and beaten pulps and for the papermaking process were carefully assessed at the Cambridge symposium 1965.[7, 8] The classification of different pulps, even if produced from the same wood, presents certain difficulties, probably because we do not know the structural features of the fibre material that are sensitive to the beating action.

Aqueous NaOH-solution not only cause pulp fibres to swell but also dissolve some of the material. In the chemical testing of both dissolving and papermaking pulps this effect is used to determine the amounts of cellulosic and noncellulosic low polymers (hemicelluloses). The dissolving power of NaOH-solution is limited, but an 18 per cent solution dissolves all the hemicelluloses, i.e. the practically amorphous fibre constituents.

Both the WRV and the solubility in 18 per cent NaOH increase with beating. A plot of these values against beating time would produce different curves for different pulps which do not lend themselves to a meaningful characterisation of the pulps.

On the basis of Malm, Glegg and Luce's work,[9] we have taken the FeTNa (iron–sodium tartrate) into consideration. FeTNa is a cellulose-dissolving complex introduced by Jayme and Bergmann[10] and subsequently studied by Valtasaari.[11]

Sodium hydroxide, sodium tartrate and ferric nitrate are used to prepare the solvent. In our preparation (see Appendix) 1·025 moles of sodium hydroxide per litre are used: 0·9 mole is required to react with 0·3 mole of iron, thus leaving 0·125 moles per litre (0·5 per cent by volume) of free sodium hydroxide in the iron–sodium tartrate solution. This standard solvent can be stored at 25° C for months without special protection from air or ordinary room lighting. Neither does its use require special precautions.

The dissolving power of FeTNa with 0·5 per cent free NaOH is very low but can be increased by increasing the amount of NaOH. Thus the solubility of a pulp can be determined as a function of free NaOH, that is, of the dissolving power of the solvent. The simple preparation, storage, use and dissolving power adjustment of this solvent permit the evaluation and

assessment of the structural features of papermaking fibres for quality and process control purposes.

The swelling and solubility of a pulp in a given FeTNa solution, like the swelling and solubility in water and in NaOH solutions, increase as beating time increases. A plot of the solubility in a given FeTNa solution against the beating time would produce different curves for different pulps which still would not lend themselves to a meaningful characterisation of the pulps.

The situation can be improved by adjusting the dissolving power of the FeTNa solution with added NaOH so that always a certain fraction, say 20 per cent of the unbeaten pulp, is dissolved.

$$\text{S FeTNa} = 20 \text{ per cent of the unbeaten pulp}$$

This causes the solubility-beating time curves of different pulps to coincide at zero beating time but still produces different curves for different pulps, which give a certain amount of information about the beatability of papermaking pulps.

The decisive improvement is achieved when in addition the solubility of the unbeaten pulp in 18 per cent NaOH is determined and the FeTNa solution is so adjusted (by adding NaOH) that it dissolves 10 per cent more. Since the amount of fibre material soluble in 18 per cent NaOH is different for different pulps the characterisation of the fibres' structural features by the dissolving power of the solvent is also changed, and always

$$\text{S FeTNa} - \text{S NaOH 18 per cent of the unbeaten pulp} = 10 \text{ per cent}$$

The amount of free sodium hydroxide in the FeTNa solution, which produces this solubility value is called relative solvent power (PSr). The difference between the solubility of a pulp beaten to different degrees in the so adjusted FeTNa solution and the solubility of the unbeaten pulp in 18 per cent NaOH is called the solubility difference SD.

Various unbleached and bleached pulps were beaten in a Valley beater, under predetermined conditions, to wetness values (as °SR) as normally used in laboratory tests. Fig. 1 shows the solubility difference of different pulps as a function of the beating time index. The beating time index is the percentage of the maximum beating time used. For the sake of clarity only four pulps are shown in the figure. It is seen that the curves not only intersect the ordinate at a solubility difference value of 10 per cent as they must do, but are also coinciding straight lines.

Since the action of solvents is sensitive to structural changes of the fibres and since mechanical beating causes such structural changes, the coincidence of the SD-time curves for all pulps means that the SD-value is a true indication of the structural changes which the pulp (any pulp) undergoes during beating.

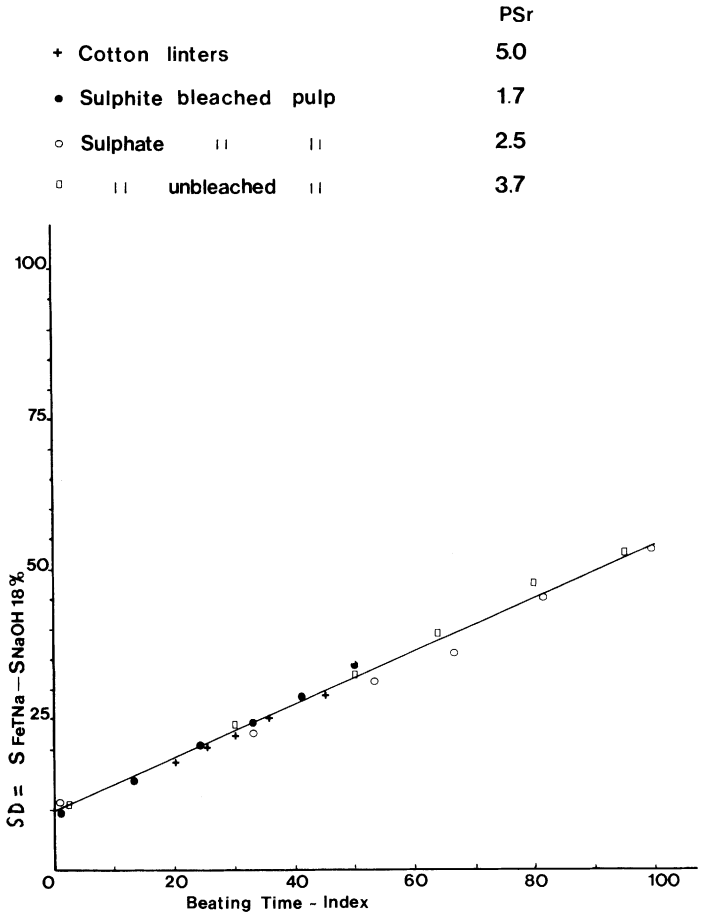

Fig. 1—Solubility increase *vs.* beating

The PSr-value necessary to make (a) SD = 10 for unbeaten pulp and (b) the SD-time curves of all pulps coincide, is thus the easily determinable pulp characteristic aimed for. It is a measure of the beatability of the pulp in the sense that a low PSr-value indicates high (easy) beatability and a high PSr-value low (difficult) beatability, see Fig. 1. Its determination presents no difficulties with bleached and unbleached chemical pulps. If necessary, one can verify the PSr after a mild delignification or a peeling action.[12]

Moreover, the beatability is an indication of how easily the mechanical action of the beater produces structural breakdowns. On the other hand, if a

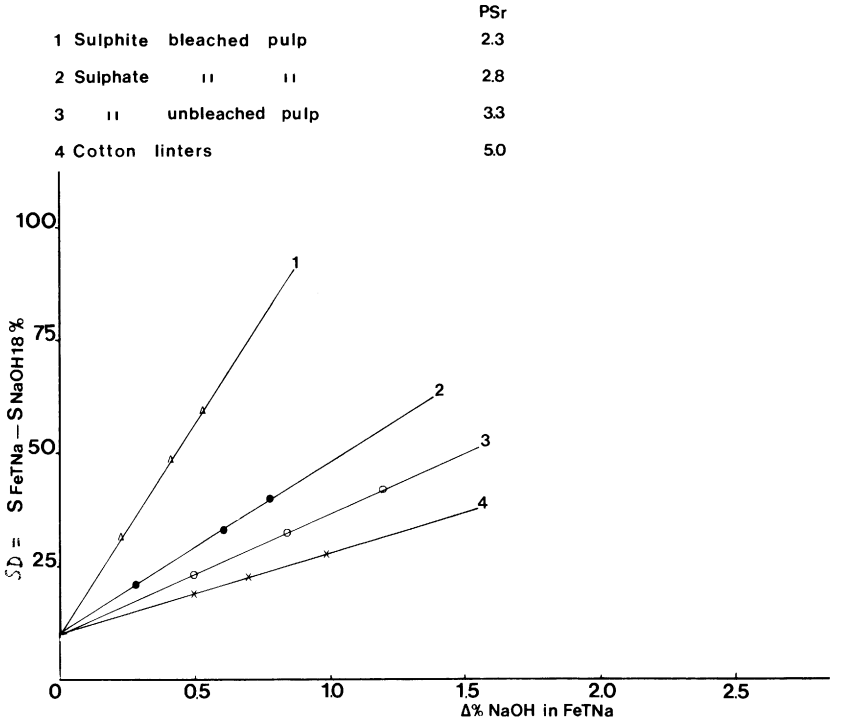

Fig. 2—Solubility increase *vs.* PSr increase

structure is easily broken down mechanically, its solubility in FeTNa should also increase rapidly with increasing dissolving power of the solvent. Conversely, a structure more resistant to mechanical action should also be more resistant to increasing dissolving power. This is shown in Fig. 2 (the pulps are different from those shown in Fig. 1) where the SD-value is seen to increase steeply with the NaOH addition to FeTNa for a pulp of high beatability[1] and much less so for cotton linters[4] with a low beatability. These curves can be said to simulate the beating process, i.e. its structure reducing action, by the action of a solvent, or mechanical power by dissolving power.

The PSr can be used:

for raw material control, i.e. to see whether one batch of pulp is the same as the last batch;

to assess the beating behaviour of pulps from different species or different types of pulp;

to control the beater performance;

to increase more or less the solubility with beating;

to evaluate fibrous, non-fibrous and fine pulp fractions, before and during beating;

to evaluate the effects of the fibre primary wall and the amount of lignin.

Conclusions

THE standard practice to evaluate a papermaking pulp in wet conditions and as sheets of paper is, to say the least, very time consuming. By contrast, the evaluation of the solubility of a pulp in a cellulose-dissolving complex that is easy to prepare and to use (FeTNa) permits (1) the classification of pulps by determining the solvent power (PSr) of the dissolving complex; (2) the assessment of the beating behaviour of a pulp without actually carrying out the beating; (3) the assessment of the pulp solubility increase by beating and thereby helping the papermaker in the selection of raw materials and of beating conditions.

Our investigations have not been continuous or thorough enough. We hope that others will be able to do even more and that the validity of the proposed method will be confirmed.

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Appendix

Preparation and use of the FeTNa *solution*

1. Preparation of the FeTNa solution according to Malm, Glegg and Luce:[9]

41 g of sodium hydroxide
207 g of sodium tartrate ($Na_2C_4H_4O_6.2H_2O$)
121 g of ferric nitrate ($Fe(NO_3)_3.9H_2O$)
and amount of water to bring the volume to 750 ml.

2. 50 per cent NaOH solution to change the dissolving power of FeTNa pulp.
3. Pulp slushing and storing in water for 12 hours.
4. Centrifuge thickening of pulp to 40–50 per cent consistency—determination of moisture content.
5. Test procedure:

(*a*) 0·5 g of o.d. pulp per 100 ml of solvent,
(*b*) water addition,
(*c*) 75 ml of solvent,
(*d*) NaOH volume for solving power control,
(*e*) mild stirring for 15 min at 20°C,
(*f*) 5′ centrifugation,
(*g*) filtration of the liquid using a Gooch crucible (G 2),
(*h*) addition of 10 per cent glycerin solution to the solid residue and mixing,
(*i*) filtration and washing with 10 per cent glycerin solution for complete removal of solvent,
(*j*) washing with water, 1 per cent acetic acid solution, water,
(*k*) drying to constant weight.

Example: FeTNa solving power:

2·5 per cent free NaOH
0·5 g of o.d. pulp
21 ml of water
75 ml of FeTNa solution (0·5 g free NaOH)
4 ml of 50 per cent NaOH

Discussion

Dr J. Mardon Do you think your methods make conventional laboratory beating obsolescent?

Borruso No, it doesn't make beating obsolete because you need a lot of experience to be able to perform this process in the laboratory.

Prof. R. Marton What is the limiting kappa number of the pulp that you can successfully treat in this way?

Borruso You have to use unbleached chemical pulps for this method. However, if you use the technique for high yield and mechanical pulps you can learn a great deal about the action of the pulping method. In other words we can investigate the relative merits of one high yield pulping method to another.

Mr L. A. Gaspar Would you care to comment whether this method can be used to assess the characteristic of waste fibres and could it be used to assess how you should refine waste fibre?

Borruso It is a technique which is very suitable for pulps used for speciality papers, such as condenser paper and filter paper.

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