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Discussion

Mr M. I. MacLaurin Before I open discussion, I am going to ask Mr Howarth to follow on because I know we would like to expand on this question of handsheet work and its validity or otherwise and also to examine the whole question of whether the laboratory work one does on this kind of thing sensibly simulates the real world. Mr Howarth will now present some new work.

Prepared Discussion Contribution

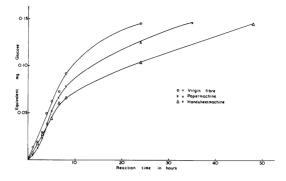
A METHOD OF MEASURING FIBRE CHARACTERISTICS BY ENZYME REACTION

P. HOWARTH and C. J. H. PYCRAFT

In the paper submitted to the Symposium, attention was drawn to the different results obtained by recycling handsheet paper and Fourdrinier machine-made paper. A method has been sought to give some indication of whether this is due to differences in the fibres themselves.

A promising technique, we have found, is to measure the rate of reaction of fibres with an enzyme. Fibres, in suspension in water containing an enzyme, are agitated in a constant temperature bath. We find the enzyme cellulysin and temperature 39° C to be effective. After a measured time, the liquid is separated from the remaining fibres by filtration. The liquid, of course, contains glucose in solution, the product of the cellulose broken down by enzyme action. The amount of glucose in solution may be measured by various techniques; we use the GOD assay. This consists of adding to the solution known amounts of glucose, the intensity of the resulting colour being a measure of the amount of glucose present. Colour intensity is measured by a spectro-photometer at 560 nm wavelength.

Under the chairmanship of M. I. MacLaurin



Thus by varying the time for which the enzyme cellulose reaction is allowed to proceed, a curve may be obtained relating the proportion of cellulose broken down to time. The figure shows such plots for pulp, disintegrated handsheets and disintegrated Fourdrinier paper. The fibres for this experiment were obtained in the following way. The pulp, a semi-bleached kraft, was disintegrated in the UMIST pilot hydrapulper and beaten to 42° SR. From this beaten stock a sample was taken and the remainder went forward to the UMIST Fourdrinier paper machine. Part of the sample was kept and gave the 'pulp' curve in the figure. The rest of the sample was made into handsheets (by B.S. method with recycled backwater) and these handsheets disintegrated, gave the 'handsheet' curve in the figure. Disintegrated paper from the machine run gave the 'paper machine' curve in the figure.

The important feature of these curves is that they are all different and the 'paper machine' curve is closer to the 'pulp' curve than is the 'handsheet' curve. It is tempting to assume that the fast, initial part of the curve relates to the amorphous cellulose in the fibre and the slower part to the crystalline component. If this is so, then the proportions of these components appear to be about the same for the three sets of fibres.

The technique seems to give us a method of measuring changes occurring in fibres as a result of their being made into paper. We propose to exploit it in the following ways:

- a. Examination of fibres by electron microscopy at various stages of enzyme degradation.
- b. Comparison of enzyme reaction rates using fibres from various positions in the paper machine, e.g. flow-box, couch, press etc.
- c. More detailed analysis of reaction curves to obtain characteristic figures.

We are grateful for the help we have received from Dr K. Indge of the department of Biochemistry in UMIST in developing this technique.

 $Mr \ D. \ D. \ Hulit$ I might suggest that there is a considerable difference between the fines present in a recycled sheet and the ordinary fines which are produced in the beating process. I think that what you are seeing as you make the two sheets, first on the machine, and then on the laboratory handsheets, is the retention of these fines. These fines—we call them hornified fines—following some of your earlier work, do not seem to have the same surface activity, or surface reactivity as ordinary fines produced in the beating process. I think what is happening when you make your paper machine paper is that you are not retaining those fibres and they are not reactive. Therefore the strength increases. If you take your disintegrated raw stock, remove the fines from it, beat it and make both handsheets and paper machine paper, you will find that the curves almost coincide. If you take the fines from the paper machine white water and recombine them, in other words, force the fines back into the system, then the paper machine paper comes very close indeed to the strength of the handsheets.

Howarth Thank you very much for that contribution. Of course one of the other things we have to do is to investigate the effect of the enzyme on various fibre fractions. In fact, in the experiment we did, our handsheet machine has recycling backwater and, of course, we recycle the backwater on the paper machine. In our earlier work, where we first saw this difference between handsheets and paper machine sheets, we looked into this very carefully and also did fibre fraction measurements which we reported at Ellenville. The results indicated that there is not really any significant difference in our system between the fibre length distribution obtained when we disintegrate handsheets and that obtained when we disintegrate paper machine paper. However, there may still be something in what you say and it is something we must not lose sight of.

Hulit Yes, it's the minus 200 mesh fines which are the important fines as far as the non-surface active ones that tend to act much like filler clay in the system in my own personal model. Those are the important ones and those are the ones most easily lost. I think in your preprint you mentioned the fact that less of them were retained in your machine made paper.

Prof. L. Göttsching I have a question related to the statement that there is a significant difference between handsheet results and machine made paper results. Do I understand correctly that those research workers at technical universities or institutes where no paper machine is available, are perhaps restricted in going ahead to do research in the waste paper field? If this is so,

wouldn't it be worthwhile to ask those responsible for financing research and development, both in industry and in government-financed establishments, to support those who are active in waste paper research; not only by financing the staff, but also by providing better technical equipment?

Mr MacLaurin I would like to ask Professor Szwarcsztajn to comment on this, partly from the fines point of view, but also because in a minute or two he will present his work which is mostly based on handsheets and he may wish to defend its validity.

Prof. Szwarcsztain I want to say something about the sheet information. I agree with some of the points made. Obviously there must be a difference between the properties of a standard handsheet and machine-made paper produced under quite different conditions. The main differences are: The consistency of the stock used, dynamic or static formation, restrained or unrestrained drying and, last but not least, closed or opened water circuit. Generally, also, the stock for handsheets is prepared in laboratory beaters. Unfortunately it is too expensive and time consuming to make extensive investigations with many variables exclusively on a paper machine. We therefore, to check our results from time to time, did our beating in a small Escher Wyss conical refiner, formed the sheet in a French dynamic former, recirculated the waste water and dried the sheet on a drying cylinder. In some cases we formed paper on a small pilot paper machine. According to paper forming conditions we naturally found some differences as were also mentioned in the papers by Howarth and by Eastwood & Clarke. But these differences, in our experience, were only quantitative and not qualitative in character. I think that we should not give too much attention to the scale of these differences. If we at the same time analyse, not only the strength properties of paper or hand sheets, but also such properties as freeness, specific surface, water retention value and fines content of the recycled stock and of its fibrous fraction, we then are able to better understand the roles of the pulp fractions and of the individual processing variables in the mechanism of the degradation of a pulp during recycling. I think that it is not the problem of the formation of the sheet on a laboratory sheet former or on the paper machine that is important, but the conditions for sheet formation, which can be varied very widely, both in a sheet former as well as on a paper machine. These conditions have a major affect on the quantitative, but not qualitative, changes in the strength properties of a recycled pulp. From all the investigations which have been made world-wide on the changes in physico-chemical and physical properties of a recycled stock, it emerges that there must be a

reduction in its strength properties with recycling. Therefore, when I look at some of the results recorded in the preprints, for paper made on a paper machine, which show that recycling does not produce any fall in strength and sometimes even a slight rise; I wonder what can we say as a panel to the audience. Is there a fundamental problem in the recycling at all?

Mr S. F. Brailsford This difference between the results from handsheets and machine made papers interests me. I wonder if the academics have asked themselves why there is this difference. It seems to me that there is one major difference in the method of manufacture and that is concerned with pressing. I think Mr Howarth's results might well be explained in terms of wet pressing. It is a well-known fact that people, using recycled fibre in production, do work or try to work at the maximum line pressures possible. The recycled fibres are of course bulkier than virgin fibres and it seems to me that you therefore ought to press them harder if you want to achieve equal bonding. Certainly it is the practice in our own mills to work at the maximum possible line pressure.

Howarth That is why we have developed this enzyme technique to try to see the difference. However, I think one point is being missed. We are not talking about the difference in the properties of the paper made by handsheet and by paper machine. We are talking about the difference between the fibres obtained when you disintegrate handsheet paper and those obtained when you disintegrate machine made paper. This is the difference we are interested in-the difference that the different making processes have on the nature of the fibres obtained from the paper when recycled. Your point about pressing may be correct. Mr Brailsford, and it is obviously something which we must look at. At the moment we say that, if you make paper on the British Standard handsheet former by the standard method, the fibres you obtain by disintegrating those handsheets are different from the fibres obtained if you had formed the same stock into paper on the UMIST Fourdrinier machine. We have no experience on other paper machines, but we suspect that this difference between the fibres occurs because of something in the actual making process, perhaps the drying under tension or the forming on the wire.

Brailsford Are you suggesting that a major factor in this difference could be wet pressing?

Howarth Yes.

Dr A. de Ruvo I fully agree with Hulit that the difference that exists between the different kinds of recycling equipment is the difference of retention of the minus 200 mesh fines and, of course, this is difficult to control. You have to be careful with your fractionation procedure in order to be accurate. The second thing with regard to the paper making process, is that we believe that the process of closing the pores is very important. The process during the drying of paper has a definite influence on the strength potential of the paper.

Our work has shown that, for papers which have only been defibrated, there is a clear difference associated with the drying temperature. We also see a difference in strength potential in fibres from papers dried freely and under restraint. The freely dried paper has a high strength of failure at the same tensile index as the paper dried under restraint.

It appears then that paper, when recycled, 'remembers' how it was dried. The more we beat the recycled fibres, the less this effect shows, but it is apparent in paper made from recycled fibres which have been merely dispersed and not beaten.

As you have already said, wet pressing will induce an increased drying tension in the dryer section if the web is being cooled. This will also be 'remembered' in the properties of the final sheet.

Göttsching We are talking about the difference between handsheets and machine-made sheets, but there are also differences between the standard procedures in different countries for making handsheets. The German standard method is different from the TAPPI method and the British and the Scandinavian. The forming procedures and retention, and the drying conditions, are all different and therefore the results are different even for different kinds of handsheets. It is not surprising, therefore, to find differences between handsheets and machine-made sheets.

What is important is the fact that the differences are quantitative rather than qualitative, as Professor Szwarcsztajn pointed out, so that we will find the same rank order when we study the effects of different influencing variables, such as the type of pulp, additives, the method of drying, and others.

 $Dr \ K. \ Ebeling$ To me the difference in the tendency to release glucose into the solution between fibres obtained from disintegrated handsheets and from disintegrated machine-made paper is not very peculiar. It is a fact that in slushed pulp there is already some glucose in the solution as Mr Levlin pointed out earlier. The fibres have a surface rich in colloidal hemmicellulose. There are more fines, that is, a larger surface area, in a suspension of fibres from disintegrated machine made paper than in a suspension of fibres from

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disintegrated handsheets. I believe the enzyme reaction is a surface phenomenon, and when the surface area increases the enzyme will release more glucose into the solution.