Using Statistical Experimental Design Techniques to Determine the Most Effective Variables for the Control of the Flotation Deinking of Mixed Recycled Paper Grades

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Abstract

Waste paper recycling in South Africa has grown to a respectable recovery rate of 43% in 2008. Pending legislation will further boost the recovery rate of recycled paper. Domestic household waste represents the major remaining source of recycled paper. This source will introduce greater variability into the waste streams entering the recycling mills, which will result in greater process variability, lower quality and operating difficulties.

In this study, a typical deinking process was investigated using the techniques of experimental design to determine the relative effects of process chemical additions, pH, pulping and flotation times, pulping and flotation consistencies and pulping and flotation temperatures on the final deinked pulp properties.

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Samples of newsprint, magazines, and two grades of mixed office waste were pulped and deinked by flotation and washing in the laboratory. Handsheets were formed and measured for brightness, residual ink concentration and yield. A Plackett-Burman experimental design was applied and the net effects of the various process parameters were determined. The net effects for all grades and process parameters were ranked in order of magnitude. It was determined that the grade of recycled paper had by far the greatest influence on final brightness, followed by the effect of the deinking process and then by the effect of the individual process variables.

Within the process variables, the order of influence on brightness was flotation conditions, alkalinity and addition of bleach. The residual ink concentration was largely determined by flotation conditions and alkalinity. The yield was determined only by flotation conditions. Pulping time and pulping temperature were involved in many interactions between variables.

The results are expected to be useful in developing a neural network control strategy to deal effectively with the challenges associated with a variable waste paper raw material stream.

Introduction

Until recently there has been a lack of legislation in South Africa pertaining to waste paper recycling. Despite this, the recovery rate of waste paper has grown from 29% in 1984 to 43% in 2008 (PRASA, 2009a). Waste paper collections have been driven by the major paper manufacturers by aggressive promotion of the recycling ethic and the paying of good prices for waste paper.

However, the National Environmental Management: Waste Act, 2008 has recently been put into effect. This bill stipulates that any material that can be recycled will not be classified as waste, and will thus not be allowed to be dumped in landfill sites. This bill will thus boost the supply of waste paper in South Africa (PAMSA, 2007).

According to the Paper Recycling Association of South Africa (PRASA), the recovery of waste paper for recycling in South Africa was projected to be *ca*. 60% of the total available for recovery (PRASA, 2009a). Most of this paper originates from domestic households, and represents the last available source of waste paper for recycling. Besides the difficulties associated with the collection of this waste, there is a need for extensive sorting of the waste into useable fractions. Even with sorting processes, the resultant grades of waste are not uniform and will present challenges to the waste recycling plants in terms of ever increasing variability of incoming raw material. These challenges are by no means unique to South Africa. Globally, recyclers are looking to lower and lower quality recycled paper streams to satisfy their fibre needs.

Waste paper originating from household waste is a mixture of newsprint, magazines, office papers and packaging papers including additional contaminants such as plastics. These papers are collected and sorted into the main recycled paper grades designated as ONP (old newsprint), OMG (old magazines), HL1 (Heavy Letter 1: white office papers) and HL2 (Heavy Letter 2: grey and pastel coloured office papers). Thus, the grades of sorted waste available to recyclers in the future will be more variable in terms of paper grade and more complex to process because of the additional contaminants.

This communication is part of a larger project that will investigate the factors affecting the deinking processes at South African plants with a range of waste materials, and design an Artificial Intelligence based control methodology for feed-forward control of such processes to allow for optimal response of deinking processes to changing incoming waste paper conditions.

Background

Process chemistry of deinking plants

Deinking plants typically utilise the following main unit operations, in a single-loop process:

- batch pulping under alkaline (pH of 9 - 10.5) conditions and medium consistency (10-15%, 4 to 60 mins, 45 ºC to 60 ºC, (Ali *et. al.,* 1994). However, deinking systems for tissue use more neutral pulping conditions with low or no addition of sodium hydroxide (Goettsching & Pakarinen, 2000: 219)
- centrifugal cleaning and screening to remove light, dense and large contaminants;
- ink removal by froth flotation (0.8-1.5% consistency, pH 8-9);
- washing by means of filtration or dewatering to remove very fine and colloidal contaminants;

- dispersion at high consistency to break up remaining dirt into sub-visible particles and
- finally oxidative and/or reductive bleaching to improve the optical properties.

In more advanced processing plants producing higher quality recycled pulps, second stages of flotation, washing, dispersion and bleaching can be included. These are referred to as double-loop processes.

The alkaline conditions are achieved by the addition of sodium hydroxide and/or sodium silicate. Chelants such as ethylene diamine tetraacetic acid salts (EDTA) are added to control heavy metal ions which would otherwise disturb the bleaching chemistry. Surfactants such as fatty acid soaps, ethoxylated fatty acids or ethoxylated fatty alcohols are added to assist in the wetting of the fibres, the dispersion of hydrophobic ink particles and froth generation for flotation. (Goettsching & Pakarinen, 2000: 241-258).

The final outcome of the deinking process is measured most often by the brightness of the pulp. Alternate measures of ink removal, such as residual ink concentration (ERIC) or dirt count are sometime performed. In addition, the yield is often monitored, as it impacts on the economics of the process.

Process control of deinking plants

Typical deinking processes offer few opportunities to make adjustments to the process if the quality of the output changes. In particular the setup of the flotation cells offers no flexibility. The aeration air is aspirated into the cell through a fixed aperture, so the air flow can’t be independently adjusted. The level in the float cell is typically maintained at a set point and not varied in response to changing conditions. It is of course possible to adjust the level of addition of process chemicals, but with the exception of the final bleaching chemicals, this is not routinely done. The general strategy is to check the quality of the incoming recycled paper as a first step, and vary the addition levels accordingly. Thereafter, adjustment in process chemicals might be attempted. Such adjustments are in the main ad-hoc and based on the experience of the operating staff (Crosby, 2008).

Thus, the main driver of quality in a paper recycling plant is the variability of the incoming recycled paper, with little possibility to remedy poor quality in the process. In extreme cases, when the final properties cannot be achieved, the product is reprocessed by blending back into the pulper feed. Grades of paper with a high intrinsic brightness (for example magazines in newsprint deinking and Heavy Letter 1 in recycled office paper deinking) are employed to control the final brightness. However, this approach is becoming less viable due to constrained availability of such high quality feedstocks. The deinking mills are having to make use of lower grade and mixed waste.

Whilst there is a need to develop process control strategies, it is unclear which variables are most effective to manipulate the final deinked pulp properties.

Potential control parameters

A good *control variable* is one that can be easily changed or adjusted without throwing the process out of balance, reducing the capacity of the plant or requiring considerable operating or capital expenditure. On the other hand a variable which has a significant effect on the process but is not a practical control variable might still need to be optimized. Such a variable would be considered to be an *optimisation variable*.

High consistency batch pulpers can control deflaking and ink fragmentation through variations in consistency and pulping time (Merza & Haynes, 2001). However, a high consistency pulper cannot be effectively operated at low consistency, and *visa versa*. Hence pulping consistency and pulping time were considered redundant control variables, and pulping time was chosen as the preferred control variable, keeping pulping consistency constant.

Based on these criteria, the following variables were considered as possible control variables: pulping time, pulping temperature, addition levels of hydrogen peroxide, sodium hydroxide and sodium silicate, level of surfactant addition in the pulper and flotation cell, flotation temperature, flotation consistency, flotation pH and flotation time.

Finally, whilst still having an effect on the process, chelating agent addition and calcium hardness were considered unsuitable as control variables.

Purpose and limitations of this study

This work will investigate the factors affecting the deinking processes, by considering individually the main recycled paper grades together with the above selected process parameters in a simulated laboratory-scale process consisting of batch pulping, a single batch flotation stage and simulated washing.

Much has been reported in the literature on the individual effects of the parameters mentioned above. However, in a complex deinking system some parameters will interact, some will dominate and others will exert little influence. This work is an attempt to consider the deinking system as a whole and to look at the relative effects of all the parameters on the outcome of the deinking process, with a view to identifying the most influential control parameters.

Experimental

Experimental design

In South Africa, mixed recycled paper originating from households and destined for deinking is sorted into the following relevant grades, termed and defined as follows (PRASA, 2009b):

- HL1: white, woodfree, printed or written office papers.
- HL2: a blend of white and pastel coloured printing and writing papers.
- ONP: old newsprint, including advertisement inserts, no flexographic prints.
- OMG: all types of books and magazines, including ledgers.

Plackett-Burman experimental designs have proven to be efficient ways of screening large numbers of variables in a relatively small number of experimental runs (Barrentine, 1999). In particular, a 2-level, 11-factor, 12-run reflected Plackett-Burman design was chosen to screen the potential control variables. In this design each factor is used at a “high” level and a “low” level. The high and low levels were chosen relative to local levels of usage. The complete design is depicted in Table 1. The high and low levels for each factor are arranged in a geometric pattern with an equal number of high’s and low’s for each factor. Each run is performed and the output is measured. The outputs at the high values and low values for each factor across all the runs are averaged and subtracted to produce the ‘net effect’. An example of the calculation of the net effect of brightness is given in Table 1 for HL1.

Runs 13 to 24 are termed the reflected design, as the levels are the mirror image of the levels in runs 1 to 12. This has the effect of eliminating any 2-factor interactions. Thus the final net effect of the reflected design indicates the effect of the factor alone (Barrentine, 1999: 46).

For each run the outputs were the final brightness, residual ink concentration (ERIC, see under “Laboratory methods”) and yield. Ideally, a number of replicates of a design should be carried out to determine the variance (S^2) for each run and each variable. This makes it possible to calculate the statistical significance of the differences observed. However, because of the large number of runs involved in this project, replicates of the screening designs were not performed. Instead, a number of *midpoints* were run, using the average of the low and high values of the factors. The variance for the midpoint runs gave an indication of the variability of the runs. The midpoints were also run at intervals through each screening run, to detect any significant drift in the processes with respect to time (refer to “Variability and drift”.).

Laboratory methods

In order to minimise the variability induced by recycled paper composition, the composition of the four main grades of recycled paper was standardised as follows:

- ONP: A random selection of South African newspapers less than 6 months old with all inserts removed. Printed by web-offset.
- OMG: A blend of *ca.* 33% heavy weight coated glossy magazines, *ca.* 33% light weight coated and *ca.* 33% uncoated magazine grades, all less than 6 months old. Printed by web-offset and rotogravure.
- HL1: A blend of 80% Xerographic printed paper (laser printer/photocopier) and 20% inkjet printed paper.
- HL2: Mixed office papers comprising 44% white or grey papers and the balance a blend of yellow, green, blue and red pastel shades of paper. Printed by offset, Xerographic and inkjet.

Waste paper sufficient for one experimental design (24 runs) was torn into strips, mixed well and stored under standard conditions of 22 ºC and 50% relative humidity.

For each grade of recycled paper, water at 200 ppm calcium hardness, pulping chemicals according to addition levels in Table 1 and paper were charged to a laboratory pulper (Laboratory Hydra Pulper model UEC 2020, Universal Engineering Corporation, India) and allowed to soak for 10 minutes. Hydrogen peroxide (Table 1) was added and the mix was then pulped at 8-10% consistency at the specified temperature and time (Table 1). A sample was taken and 200g/m^2 pulp pads were formed on a Rapid-Koethen sheet former with reduced dilution (2l instead of 7l), based on the method Tappi 218 om-91: Forming handsheets for the reflectance testing of pulp. The pulp pads were measured for brightness (GE brightness, UV included, D65, 10º) and Effective Residual Ink Concentration (ERIC, C, 2º) on a Technidyne ColorTouch PC Spectrophotometer. In all cases each quadrant of the pad was measured and both sides of the pad were measured and the total average taken as the brightness of the pad.

The pulped mass was allowed to stand for 1 hour, and then a sample was withdrawn, transferred to the 15 litre flotation cell (Flotation Cell model UEC 2026, Universal Engineering Corporation, India), made up to the required consistency with 200 ppm calcium hardness water and floated at 1550 to 1600 rpm at the conditions specified in Table 1. At the end of the float, the contents of the cell were transferred quantitatively to a bucket and weighed to determine the yield (dry mass of fibre out/dry mass of fibre in). A sample of the floated pulp was formed into 200gm^{-2} pads and measured for brightness (UV included) and ERIC, as above. In addition, 60 gm^{-2} handsheets were formed on the Rapid Koethen former, according to a method based on Tappi 205 sp-95: Formation of handsheets for physical testing of pulp. The process of dilution and filtering allowed considerable quantities of fine material, including ink particles, to be washed through the screen. This resulted in different reflectance measurements to the pulp pad method of forming sheets and also between the top and wire sides of the handsheet. To accommodate the two-sidedness, the brightness and ERIC of both sides of the pads or sheets were measured and the average was taken.

Thus, for the pulping and flotation stages of the process, where it was important to measure all the ink in the pulp, the pad process was used. The washing out effect that had been observed in the preparation of the handsheets was actually used in this study to simulate the washing process that occurs in a deinking plant. These final samples were thus designated as *washed* pulp.

Office papers typically contain large quantities of Xerographic (photocopy) inks, which do not deink well by flotation (Goettsching and Pakarinen, 2000: 277). The inks tend to agglomerate into particles too large for effective flotation. This was evident in the pulp pads formed, which exhibited high levels of dirt spots, with bright deinked pulp between the fairly widely scattered dirt spots. The brightness of the handsheets was measured with the dirt spots, whose area relative to the total area measured was relatively small and thus considered to have a negligible effect on the brightness.

The amount of surfactant required to achieve frothing, and hence flotation in the cell was 0.25% to 0.75%, which is in excess of that normally added to commercial deinking plants (ca. 0.1%). This is probably due to the fact that commercial plants use back-water, which contains a certain quantity of recirculating surfactant.

Results and Discussion

Results of Plackett-Burman screening runs

General comments

The results of the screening runs with the associated calculation of the net effect of the washed brightness for HL1 is shown in Table 1 at the end of the document, as an example. Similarly, the net effects for the remaining outputs (ERIC and yield) and paper grades (ONP, OMG and HL2) are summarised in Table 2.

Pareto analyses of the net effects of the variables on the final washed brightness, washed ERIC and yield for ONP, OMG, HL1 and HL2 are depicted in Figures 1 to 12. The Pareto diagrams depict the magnitude of the effects on the brightness, ERIC or yield. Those variables that have an *adverse* effect are shown in square brackets *eg.* [%H_2O_2]. An adverse effect would be a lower brightness, a higher ERIC or a lower yield.

It can be observed (Figures 1-12) that the net effects of the 12-run and 24-run (*viz.* reflected) designs are different for some or all of the variables. The reflected design eliminates the influence of the confounded second-order or two-factor interactions from the net effect. Significant differences between the base design (*viz.* the 12-run design) and the reflected (24-run) design indicate the presence of interactions. The so-called *hereditary rule* in experimental design states that the significant main effects often have significant interactions (Barrentine, 1999: 40).

Newsprint

With reference to Figure 1, hydrogen peroxide addition, flotation time (t_f) and pulping time (t_p) were the most influential variables. Because of the inherently low base brightness of the recycled paper, the large influence of hydrogen peroxide is not surprising. The large differences in pulping time (t_p) and pulping temperature (T_p) are probably a result of interactions with hydrogen peroxide in the bleaching reaction, *viz.* the longer the time (t_p) and the higher the temperature in the pulper (T_p) the higher the brightening effect.

Studies by Azevedo *et. al.* (1999), Dionne (1994) and Renders (1993) have shown that hydrogen peroxide requires a certain optimum ratio of sodium hydroxide and sodium silicate to be effective. The results in Figure 1 suggest that each component of the bleaching liquor has an independent effect. Sodium silicate has also been shown to have an independent effect on final brightness (Pauck, 2003; Mahagaonkar *et. al.,* 1997; Santos *et. al.,* 1996; Renders *et. al.,* 1996; Ali *et. al., 1994;* McCormick, 1990).

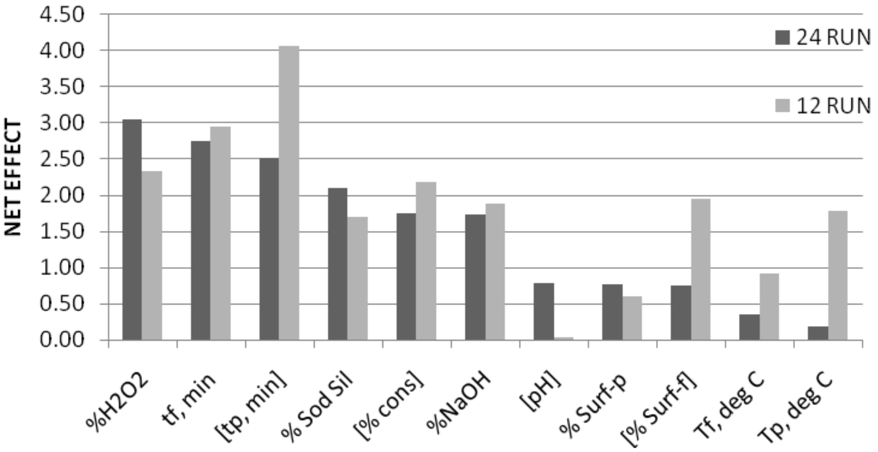

Figure 1: Net effect of variables on the washed brightness of newsprint. Variables with an adverse effect are indicated with brackets.

Flotation consistency, together with agitation speed and air flow rates have been shown to greatly affect the flotation process (Carrasco *et. al.,* 1999). The results confirm that flotation consistency (% cons) has a large adverse effect. Agitation conditions and air flow rates were not varied on the laboratory flotation cell, but were kept constant. However, if flotation time (t_f) is considered a summation of the exposure of ink particles to flotation conditions, its importance and effect is well demonstrated in Figure 1.

Pulping time has also been studied by a variety of researchers. Bennington *et al.* (1998) found that pulping time was not a major influencing factor, but Ali *et al.* (1988) reported that pulping time interacted with pulping temperature and the level of addition of bleaching chemicals to influence the final bleaching, and hence brightness. Long pulping times favour the complete disintegration of the waste paper, but contribute to excessive ink fragmentation which leads to *lumen loading*. Lumen loading is a process whereby tiny ink particles enter the fibre cells through the pits, and reside permanently in the lumen, making it impossible to remove the ink particle.

It is generally recommended to minimize the pulping time, consistent with efficient deflaking. (Körkkö & Laitinen, 2008; Merza & Haynes, 2001)

With reference to Figure 2, the five most significant effects on the washed ERIC all seem to interact. Since the ERIC does not depend on the bleaching chemistry so much as the ink removal efficiency, the low influence of hydrogen peroxide is not surprising. Variables such as time (t_p and t_f), alkalinity (NaOH and sodium silicate concentrations) and flotation consistency all appear to have a greater effect on ink removal, and hence ERIC.

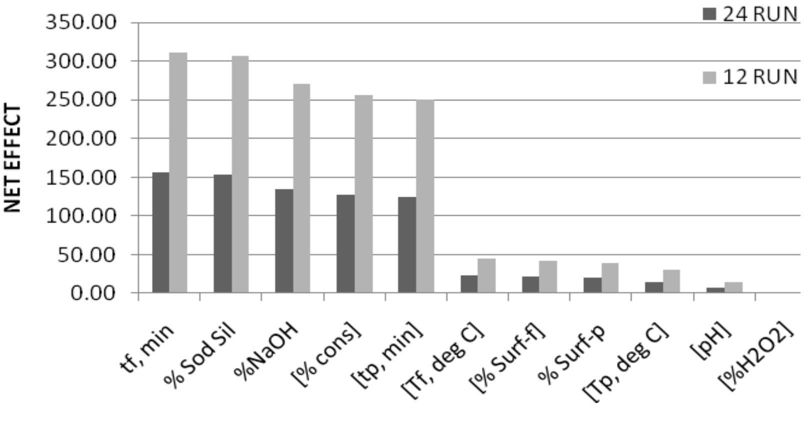

Figure 2: Net effect of variables on washed ERIC of newsprint. Variables with an adverse effect are indicated with brackets.

Factors which affect the flotation of fibres rather than ink seem to play a role in determining the yield. Figure 3 indicates that the main variables influencing flotation yield were flotation conditions (t_f and consistency) and to a lesser extent sodium hydroxide concentration and surfactant concentrations ($Surf_f$ and $Surf_p$).

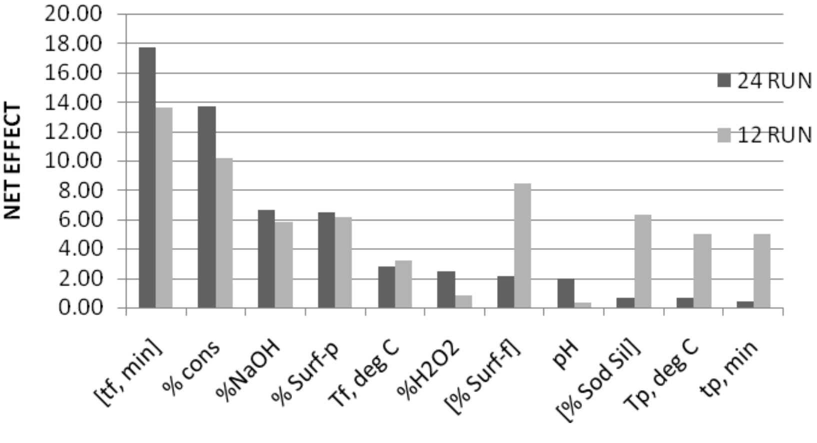

Figure 3: Net effect of variables on the yield of newsprint. Variables with an adverse effect are indicated with brackets.

Magazines

Pareto analyses of the main variables affecting the deinking of magazine papers are shown in Figures 4 to 6. In general terms, the variables had similar effects for both recycled newsprint and magazine. The ERIC was not influenced by the concentration of hydrogen peroxide or bleaching chemistry, because ERIC is a direct measure of ink, irrespective of the underlying brightness of the paper substrate.

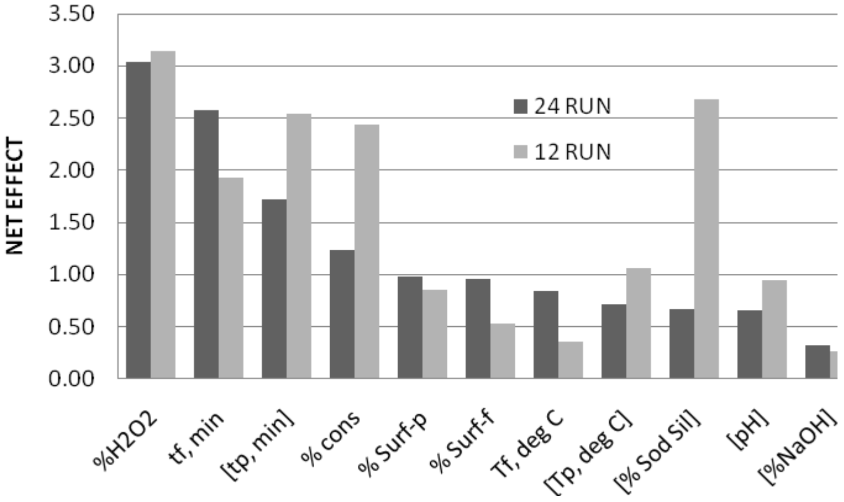

Figure 4: Net effect of variables on the washed brightness of magazine papers. Variables with an adverse effect are indicated with brackets.

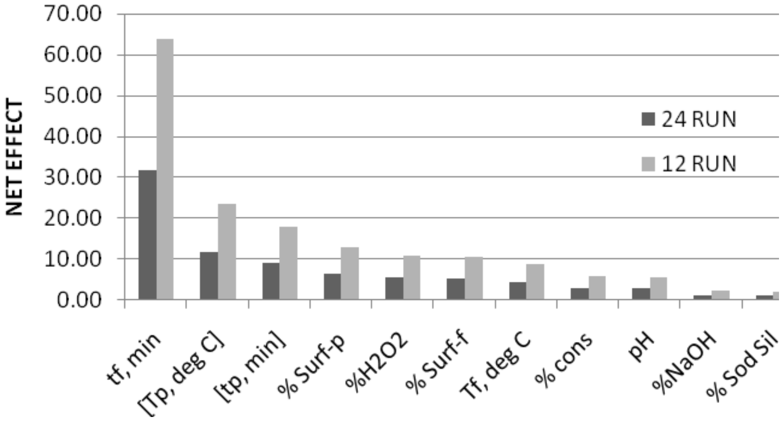

Figure 5: Net effect of variables on the washed ERIC of magazine papers. Variables with an adverse effect are indicated with brackets.

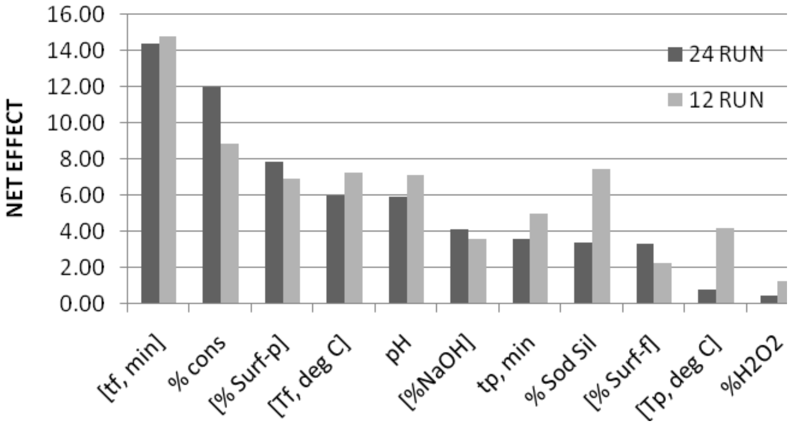

Figure 6: Net effect of variables on the yield of magazine papers. Variables with an adverse effect are indicated with brackets.

Heavy Letter 1

Pareto analyses of the main variables affecting the deinking of HL1 are shown in Figures 7 to 9.

With reference to Figure 7, the process residence time (as given by pulping time t_p and flotation time t_f) plays a large role in determining the final brightness, as does the alkalinity (combination of sodium silicate and sodium hydroxide). Hydrogen peroxide seems to play a lesser role, probable due to the already very high base brightness of Xerographic papers.

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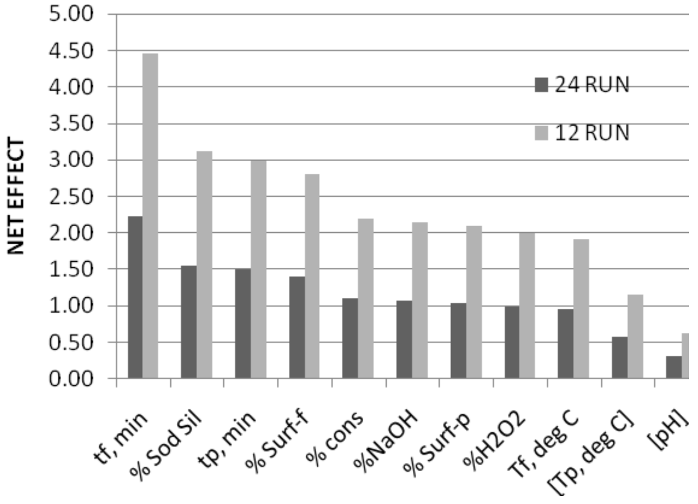

Figure 7: Net effect of variables on the washed brightness of HL1. Variables with an adverse effect are indicated with brackets.

The alkalinity (NaOH) and pulping temperature (T_p) have the most significant effects on the washed ERIC (Figure 8), with variables such as time (t_p and t_f), and flotation consistency still playing a role.

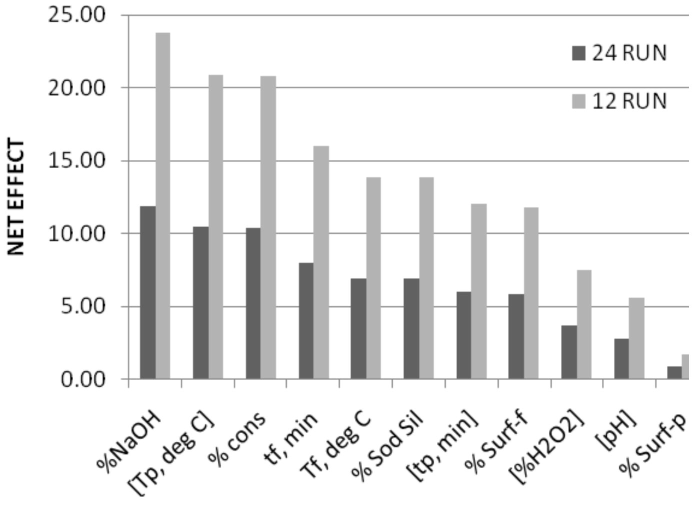

Figure 8: Net effect of variables on washed ERIC of HL1. Variables with an adverse effect are indicated with brackets.

Not surprisingly, the hydrogen peroxide concentration is not a significant factor. In fact the data suggests that it plays a small negative role on the ERIC, which is not easy to explain. Also, the pulping temperature exerts a large influence on the deinking of toner inks. Fabry, Carré & Crémon (2001) found that temperature, amongst other variables, affected ink fragmentation, particularly with the thermoplastic binders found in toner inks. Thus, pulping temperature is a variable that would have to be optimised for these systems.

Factors which affect the flotation of fibres rather than ink again seem to play a role in determining the yield. Figure 9 indicates that the main variables influencing fibre yield were alkalinity (NaOH and sodium silicate) and flotation conditions (t_f and consistency). As expected, hydrogen peroxide played a minimal role.

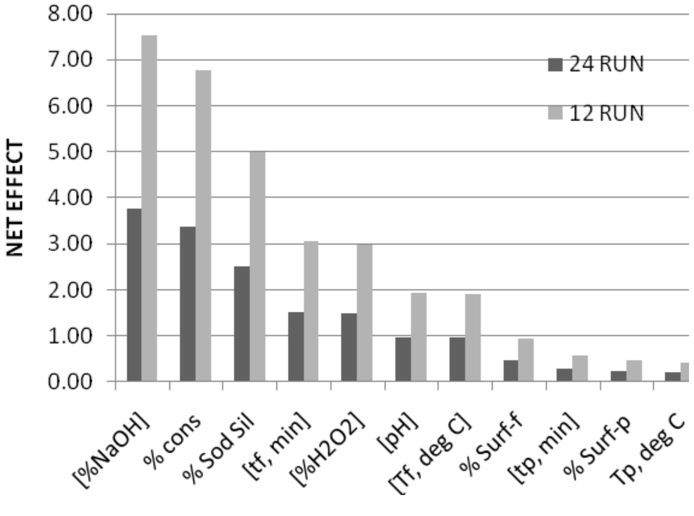

Figure 9: Net effect of variables on the yield of HL1. Variables with an adverse effect are indicated with brackets.

Heavy Letter 2

The Pareto analyses of the main variables affecting the deinking of HL2 are shown in Figures 10 to 12. The effect of pulping temperature (T_p) on brightness was prominent for this grade. As for HL1, temperature plays a role in the fragmentation of the inks found on this type of paper. A higher pulping temperature could also benefit the bleaching reaction. HL2 has a lower base brightness than HL1, and thus hydrogen peroxide can be seen to have a greater effect.

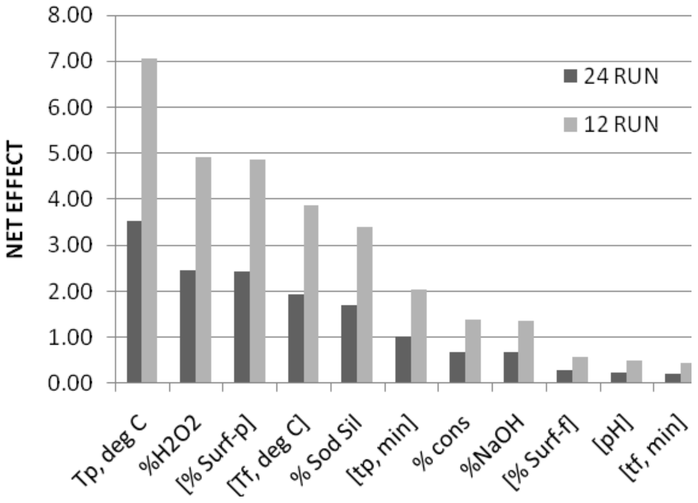

Figure 10: Net effect of variables on the washed brightness of HL2 papers.

With respect to the ERIC of HL2 (Figure 11), hydrogen peroxide continues to play a minor role, with flotation conditions (consistency and time t_f) dominating.

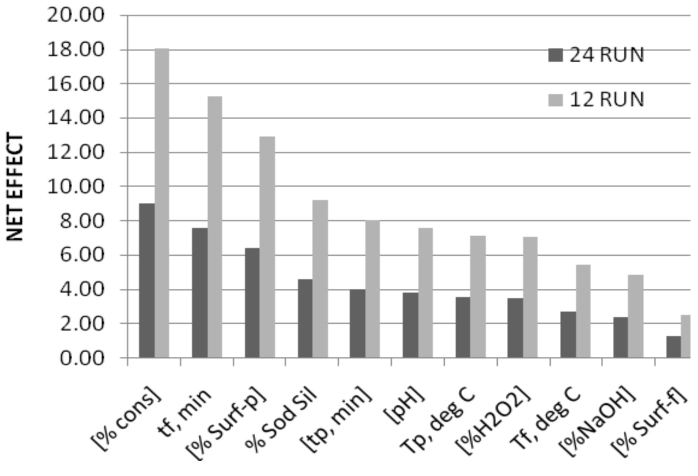

Figure 11: Net effect of variables on the washed ERIC of HL2.

A different set of variables affect the yield of HL2 (Figure 12). Flotation conditions (consistency and time) now dominate and alkalinity was less influential.

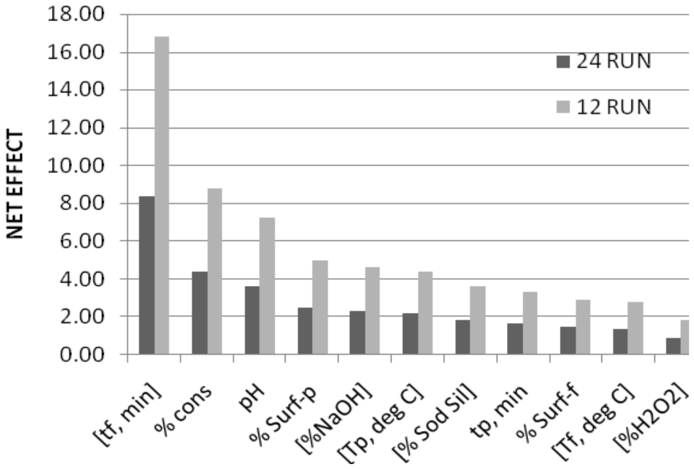

Figure 12: Net effect of variables on the yield of HL2.

Overall Performance

Dependence of brightness on flotation time

Flotation time (t_f) emerged from the Plackett-Burman screening trials (Figures 1-12) as one of the consistent determinants of overall performance. The brightnesses as a function of flotation time are shown in Figure 13. A flotation time of zero was assigned to the brightnesses of the pulper sample. The average brightnesses at 5 minutes and 20 minutes of the Plackett-Burman runs are reported and the variability is depicted by error bars showing plus and minus one standard deviation. The variability in the brightness results at each flotation time is due to the high-low pattern in the experimental design. The large variability, although a result of the high-low nature of the experimental design, also serves to demonstrate the magnitude of the variability induced by the process conditions as opposed to that induced by paper grade, flotation time or processing stage.

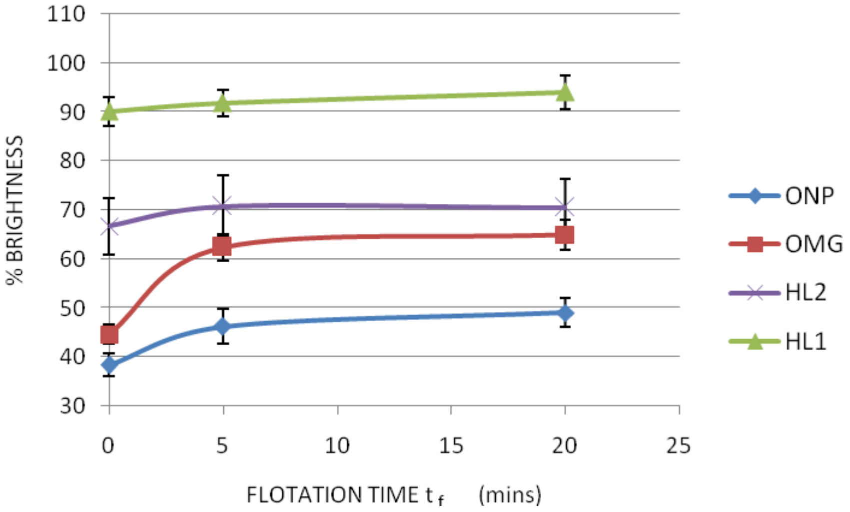

Figure 13: Brightness as a function of flotation time.

For all recycled paper grades, the brightness increased with flotation time (Figure 13), with the major proportion of the ink being removed in the first 5 minutes. For the office paper grades, the average overall increase in brightness was only about 5 points. This was much less than the 10 to 20 point increases noted for newsprint or magazines. This is probably because office papers are printed lightly compared to newsprint, and start out at much higher brightness levels. In addition, the fragmentation or dispersion of ONP inks lowers the brightness, whereas the toner inks fragment to a lesser extent.

Dependence of ERIC on flotation time

The dependence of the ERIC values (plotted on a logarithmic scale for clarity) on flotation times are shown in Figure 14. These changes are essentially the reverse of those observed for brightness. Again, the greatest changes and variability were observed with newsprint and magazines.

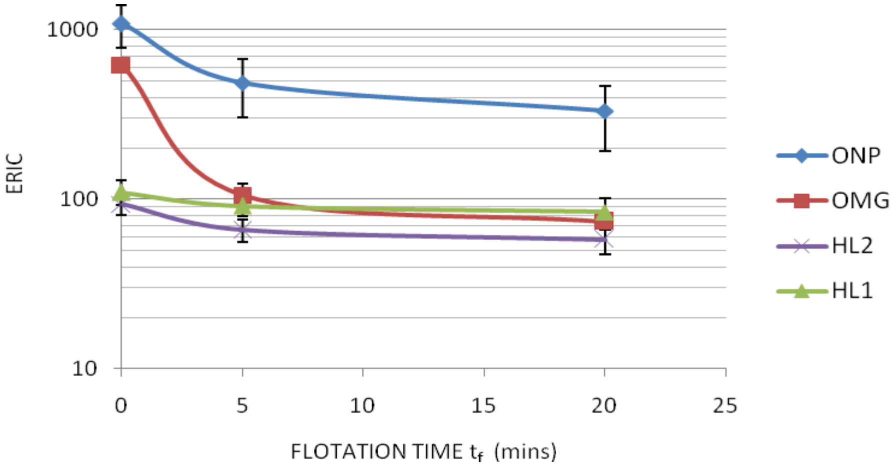

Figure 14: ERIC as a function of flotation time.

Dependence of yield on flotation time

The yield initially decreased rapidly with flotation time (Figure 15), and continued to decrease after 5 minutes flotation time with no great improvement in brightness (Figure 13), for all grades. Yield and optical properties represent the trade-off between economy and quality that has to take place in every deinking operation. The yield loss was greatest for OMG, probably due to the greater losses of filler and coating material.

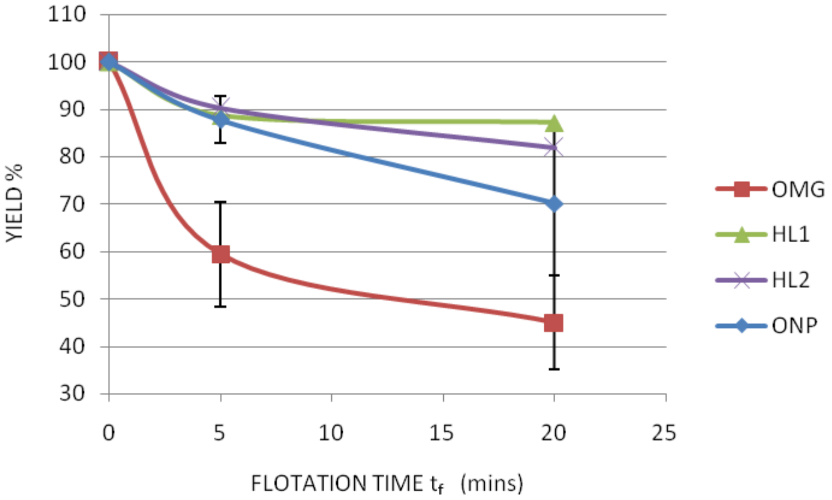

Figure 15: Yield as a function of flotation time.

Variation of brightness and ERIC with processing stage.

As outlined in the experimental procedure, the recycled paper was first pulped, then floated and washed, in sequence. The brightness of all grades increased from pulping through flotation to washing as shown in Figure 16. The greatest increases were noted for magazines and newsprint. Also, the variability induced by the process variables (as indicated by the error bars) was considerable in the case of HL2, compared to the changes caused by processing (flotation time in Figure 13, and processing stage in Figure 16).

The error bars depict the span of influence of the process variables whereas the y-axis differences between the grades represent the influence that the different grades would have on the final brightness of a mixture of paper grades. These differences can often be more significant than the effect of the deinking process. This points to the significant potential of furnish composition as a strong control variable.

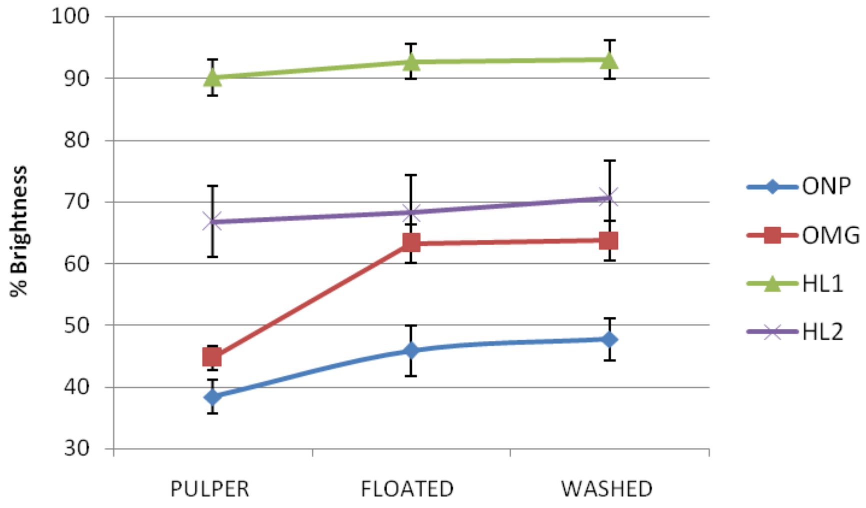

Figure 16: Effect of processing stage on brightness.

Again, similar but inverse trends to those obtained with brightness were observed with the ERIC results (Figure 17). The relative magnitudes of the effect of different grades and processing is evident from Figures 14 and 17.

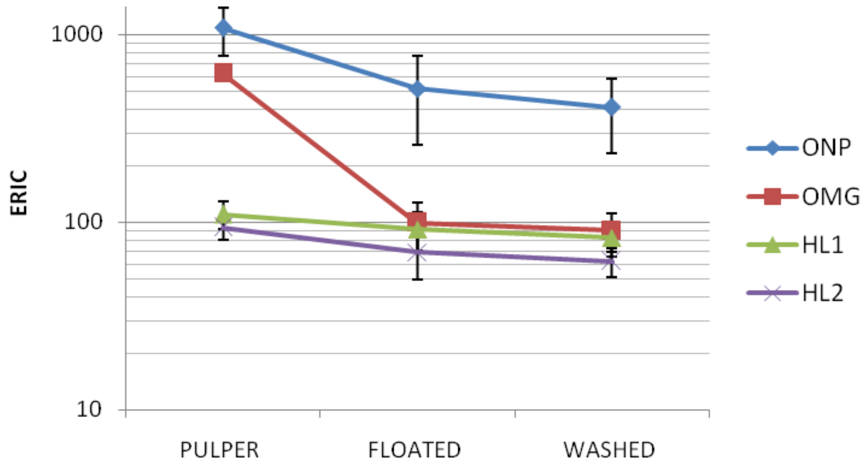

Figure 17: Effect of processing stage on ERIC.

It is also apparent from Figures 16 and 17 that the washing stage improves the final optical properties by only a small amount relative to pulping and flotation alone.

The effect of flotation on the average yield loss is shown in Figure 18. The yield lost in washing was not determined in this work. The yield after pulping was assumed to be 100%.

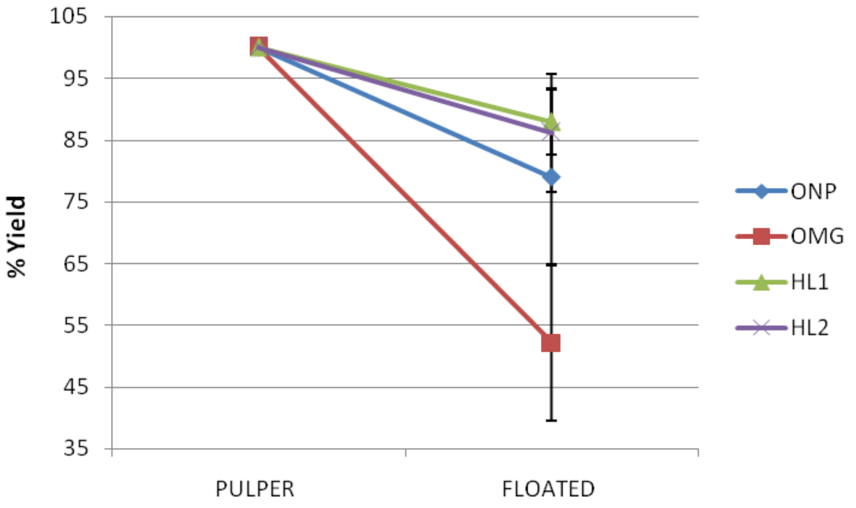

Figure 18: Effect of processing stage on Yield.

The greatest yield loss and variability in yield was observed for OMG, followed by ONP and the recycled office grades. This is probably due to the high levels of filler and coating colours found in high quality magazine grades.

The relative effects of paper grade and processing parameters

The relative effects of recycled paper grade and processing variables on the outcome of the deinking process, as depicted in Figures 16, 17 and 18, are quantified and summarised in Table 3. To determine the effect of paper grade, ONP was compared to OMG and HL1 was compared to HL2, since these would be the likely substiututions to be made if brightness were to be controlled by furnish change.

Table 3: Relative effects of paper grade and processing variables on deinking outcome.

Variables	% Brightness points		ERIC ppm		% Yield	
	ONP & OMG	HL1 & HL2	ONP & OMG	HL1 & HL2	ONP & OMG	HL1 & HL2
Effect of paper grade	16	22	319	22	26.7	1.8
Effect of processing: pulping – flotation - washing	10 - 19	3 - 4	528 - 674	27 – 32	21 – 47.8	12.1 – 13.9
Effect of process variables: standard deviation	3.15 – 3.45	3.16 – 5.9	21 - 175	10.8 – 16.9	12.6 – 14.2	5.4 – 9.6

It can be seen from Table 3 that with respect to brightness, the relative effects are: paper grade ≈ processing > process variables for ONP/OMG and paper grade > process variables > processing for office grades. On the other hand, for ink removal (ERIC), the relative effects are: processing > paper grade > process variables for all paper grades. For the yield of the ONP/OMG system, the relative effects are processing > paper grade > process variables, and the HL1/HL2 system ranked processing > process variables > paper grade. (Note: ≈ means approximately the same or almost equal to). To summarise this complex picture, it can be said that either processing or paper grade always dominate the effect of process variables.

The effects of process variable interactions

The differences between the 12-run and 24-run designs (reflected design) suggested interactions between variables. For Figures 1 to 12, the five largest interactions (difference between Plackett-Burman 12-run and 24-run net effects) were tallied. The variables which most often showed interactions were, in order of frequency: flotation time > sodium silicate ≈ flotation consistency > pulping time ≈ pulping temperature. This indicates that, in addition to the main factors already identified, pulping time and pulping temperature were active in interactions.

Variability and drift

During a screening design of 24 runs, replicates of midpoint runs were performed at random intervals, to determine the stability of the pulping and flotation procedures. The results for the brightness trends in the midpoint runs of the four screening runs are shown in Figure 19. The coefficients of variation (CV = 100 x standard deviation/mean) were the highest in the HL2 runs at 4.3%, and trended upwards slightly in the HL1 run. This is most likely due to the raw material changing slightly through the run.

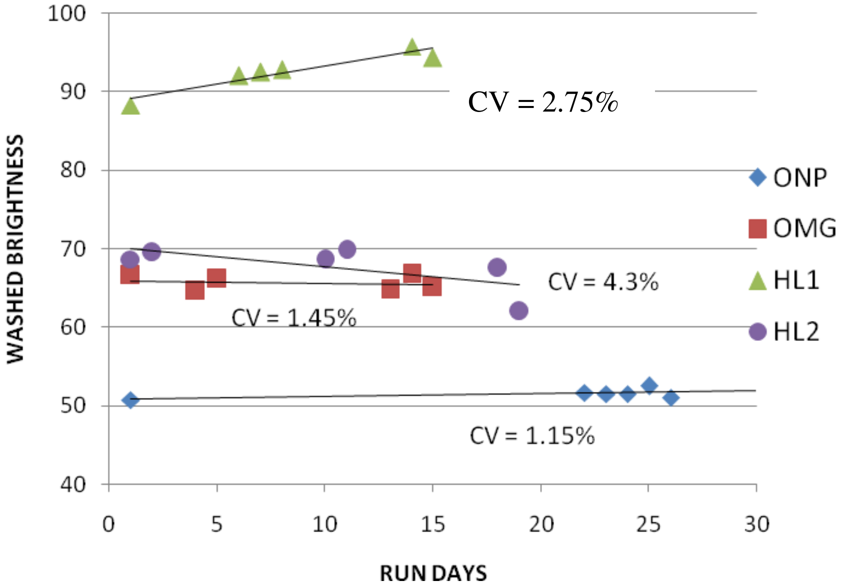

Figure 19: Variability (indicated by the CV) and drift of final brightness.

The relatively flat trends in Figure 19 indicate fairly constant conditions within the different runs.

Ranking of process variables

The objective of this work was not so much to identify and explain all the interactions that take place in a deinking system, but rather to identify the process variables having the most influence on the outcome of the deinking process. In order to select the most influential variables overall, a *factor ranking analysis* was carried out. The variable or factor responsible for the greatest net effect in each case was assigned the rank of 1, intermediate factors were assigned ranks of 2 to 10 and the factor with the least effect was assigned a rank of 11. The outputs for which factors were ranked were washed brightness, washed ERIC and the yield. For each factor the mean, greatest (1) and least (11) rank across all of the waste paper grades and all outputs was calculated and arranged in ascending order. The order of priority for the ranking was taken as greatest rank first followed by mean rank. These calculations are shown in Table 4 and depicted graphically in Figure 20.

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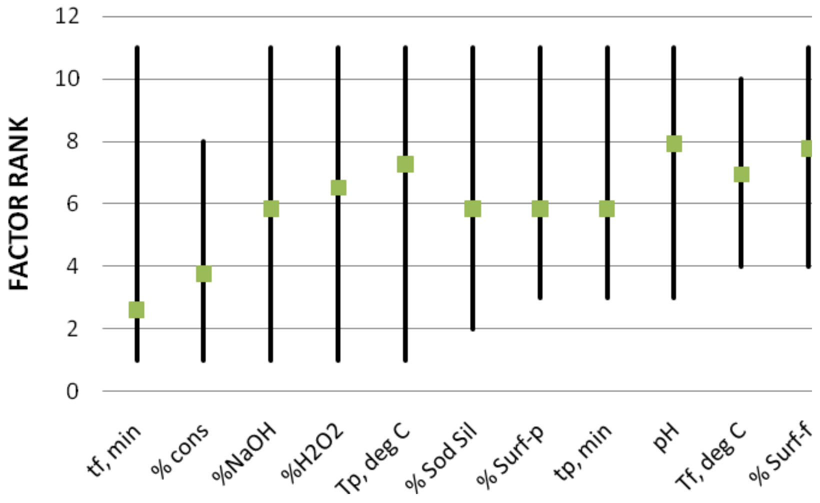

Figure 20: Ranking of control variables – greatest influence-MEAN-least influence.

It can be seen from Figure 20 that the flotation pH, surfactant added to the float cell (%Surf-f), the flotation temperature (T_f) and pulping time (t_p) are the least ranked variables on average. Their greatest ranking was only 3-4.

Energy constraints and the requirement for heat transfer equipment make the temperature an uneconomical control variable. In addition, the effect of temperature in flotation was small. Thus temperature was eliminated as a practical control variable. However, in the case of HL2 brightness, HL1 ERIC and OMG ERIC, the pulping temperature (T_p) was a factor which showed significant influence. In these cases T_p can be considered an important *optimisation* variable, whilst not being practical for process control purposes.

The pulping time (t_p) appeared to have a small influence on the outcome of deinking. This agrees with Körkkö & Laitinen (2008), who suggested that pulping time should be minimised. Flotation pH and surfactant concentrations, particularly in the flotation cell were not particularly influential. Many mills do not even add surfactant into the flotation cell.

The remaining variables *viz.* flotation conditions (time and consistency), alkalinity (sodium hydroxide and sodium silicate) and bleaching agent addition are the most practical and influential variables to control the deinking process. Furthermore, by inspection of the Pareto diagrams (Figures 2, 5, 8, 11), hydrogen peroxide can be eliminated as a determinant of ERIC, and hydrogen peroxide and alkalinity can be eliminated as determinants of flotation yield (Figures 3, 6, 9, 12). These considerations result in a final list of effective control variables for the different deinking plant outputs, listed in Table 5.

Table 5: List of effective control variables for deinking recycled paper mixtures.

Brightness	**ERIC**	**Yield**
Waste grades	Waste grades	Waste grades
Flotation time	Flotation time	Flotation time
Flotation consistency	Flotation consistency	Flotation consistency
% NaOH/sodium silicate	% NaOH/sodium silicate	
% H_2O_2		

Conclusions

Flotation residence time was a consistent determinant of deinking performance. Brightness increased and ERIC decreased with flotation time, with most of the changes occurring in the first few minutes of flotation. Flotation time had the most significant and inverse effect on yield, *viz.* increasing flotation time negatively affected the yield. The improvements in brightness and ERIC were much more pronounced in newsprint/magazine deinking than in office paper deinking.

The processing of recycled paper by pulping, flotation and washing resulted in an increase in brightness and a reduction in residual ink (ERIC). The benefits of processing were more pronounced for newsprint/magazine grades than for office grades. The biggest improvement in brightness and ink removal was achieved with flotation. The washing process after flotation resulted in only minimal improvements.

The statistical design methodology made it possible to screen a large number of process variables and eliminate many as ineffective control variables. A combination of this rigorous screening process and practical considerations has resulted in a consolidated list of most influential process parameters, *viz.* flotation conditions (time and consistency), alkalinity (sodium hydroxide and sodium silicate) and bleaching agent addition are the most practical and influential variables to control the deinking process.

These parameters could be used to control the deinking brightness and ink removal of various blends of recycled papers, ranging from the typical newsprint/magazine and mixed office waste blends to more complex mixtures of three or even four of the recycled paper grades investigated. These same parameters will influence the yield, which will thus require a recycling plant to select the optimum operating point.

The variables which most often showed interactions were, in order of frequency: flotation time > sodium silicate ≈ flotation consistency > pulping time ≈ pulping temperature. (Note: ≈ means approximately the same magnitude or frequency). Thus, pulping time and pulping temperature were involved, albeit less frequently, in interactions and thus need to be well controlled in the deinking process

The magnitude of the effects of the process variables on the deinking outcome is generally much less the the magnitude of the effect of paper grade or paper processing, for all paper grades. Depending on the grade of paper, either grade or processing exercise the most significant effect on the outcome. This confirms that the selection of the grade mix of paper to be deinked will always be the major control variable, and that the adjustment of process variables will only serve to tweak the final outcome.

Further work

The parameters listed in Table 5, in conjunction with different grades of recycled paper in varying proportions will be used in laboratory deinking runs to generate a data base that will be used to train an artificial neural network. This network, once validated by plant data (Pauck, Venditti, Pocock & Andrew, 2012), could then be used by process personnel to predict the effect of changes in waste paper grade on the final brightness produced by a deinking plant. Negative effects of waste paper changes could then be compensated for by the appropriate changes in process parameters, as indicated by the neural network model.

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Table 1 – Plackettt-Burman design and results for HL1

	PULPING						FLOTATION								
	A	B	C	D	E	F	G	H	I	J	K	Y	RESULTS		
NO.	**%NaOH**	**% Sodium Silicate**	**%H_2O_2**	**% Surfactant in pulper (Surf-p)**	**Pulping time, min. (t_p)**	**Pulping tempera-ture, °C (T_p)**	**Flotation tempera-ture, °C (T_f)**	**% Consistency**	**pH**	**% Surfactant in float cell (Surf-f)**	**Flotation time, mins. (t_f)**	**Y**	**WASHED BRIGHTNESS**	**WASHED ERIC**	**YIELD**
1	0.67	0	1	0.25	5	35	45	1.3	10	0	20	93.2	93.2	69.64	87.65
2	0.67	2	0	0.75	5	35	30	1.3	10	0.5	5	91.5	91.5	60.60	94.68
3	0	2	1	0.25	15	35	30	0.8	10	0.5	20	92.8	92.8	101.71	91.35
4	0.67	0	1	0.75	5	50	30	0.8	8	0.5	20	91.3	91.3	89.92	88.35
5	0.67	2	0	0.75	15	35	45	0.8	8	0	20	99.9	99.9	52.53	83.14
6	0.67	2	1	0.25	15	50	30	1.3	8	0	5	95.6	95.6	75.05	93.26
7	0	2	1	0.75	5	50	45	0.8	10	0	5	91.6	91.6	111.02	92.23
8	0	0	1	0.75	15	35	45	1.3	8	0.5	5	94.3	94.3	84.02	95.97
9	0	0	0	0.75	15	50	30	1.3	10	0	20	90.3	90.3	102.93	91.54
10	0.67	0	0	0.25	15	50	45	0.8	10	0.5	5	87.6	87.6	106.74	88.88
11	0	2	0	0.25	5	50	45	1.3	8	0.5	20	96.3	96.3	63.70	86.48
12	0	0	0	0.25	5	35	30	0.8	8	0	5	88.5	88.5	76.89	92.38
13	0	2	0	0.75	15	50	30	0.8	8	0.5	5	92.6	92.6	102.91	90.97
14	0	0	1	0.25	15	50	45	0.8	8	0	20	91.2	91.2	98.86	83.65
15	0.67	0	0	0.75	5	50	45	1.3	8	0	5	89.6	89.6	90.19	86.65
16	0	2	0	0.25	15	35	45	1.3	10	0	5	90.4	90.4	86.73	92.75
17	0	0	1	0.25	5	50	30	1.3	10	0.5	5	94.7	94.7	81.66	83.43
18	0	0	0	0.75	5	35	45	0.8	10	0.5	20	95.8	95.8	64.96	84.85
19	0.67	0	0	0.25	15	35	30	1.3	8	0.5	20	95.7	95.7	78.41	85.86
20	0.67	2	0	0.25	5	50	30	0.8	10	0	20	91.8	91.8	84.66	85.95

21	0.67	2	1	0.25	5	35	45	0.8	8	0.5	5	91.8	91.8	74.91	82.15
22	0	2	1	0.75	5	35	30	1.3	8	0	20	91.0	91.0	89.48	92.16
23	0.67	0	1	0.75	15	35	30	0.8	10	0	5	94.4	94.4	91.08	71.02
24	0.67	2	1	0.75	15	50	45	1.3	10	0.5	20	100.0	100.0	48.79	85.09
ΣY+	1122.35	1125.28	1121.91	1122.22	1124.91	1112.48	1121.69	1122.53	1114.04	1124.36	1129.30				
ΣY-	1109.51	1106.58	1109.95	1109.64	1106.95	1119.38	1110.17	1109.33	1117.82	1107.50	1102.56				
Yavg+	93.53	93.77	93.49	93.52	93.74	92.71	93.47	93.54	92.84	93.70	94.11				
Yavg-	92.46	92.21	92.50	92.47	92.25	93.28	92.51	92.44	93.15	92.29	91.88				
EFFECT	1.07	1.56	1.00	1.05	1.50	-0.58	0.96	1.10	-0.32	1.41	2.23				

Note: Addition levels of chemicals given as % active chemical per dry mass of fibre.

Table 2: Summary of net effects for 12-run and 24-run (bold) reflected designs.

		A	B	C	D	E	F	G	H	I	J	K
		%NaOH	**% Sod Silicate**	**%H_2O_2**	**% $Surf_p$**	**t_p, min**	**T_p, deg C**	**T_f, deg C**	**% flotation consistency**	**pH**	**% $Surf_f$**	**t_f, min**
ONP	**WASHED BRIGHTNESS**	1.89 **1.73**	1.70 **2.10**	2.34 **3.05**	0.60 **0.77**	-4.07 **-2.52**	1.79 **0.18**	0.91 **0.36**	-2.18 **-1.74**	-0.03 **-0.78**	-1.94 **-0.75**	2.96 **2.74**
	WASHED ERIC	-271 **-135**	-307 **-154**	1.22 **0.6**	-39.6 **-19**	-250 **-125**	30 **15**	45 **22**	256 **128**	14 **7**	42 **21**	-311 **-156**
	% YIELD	5.8 **6.6**	6.3 **-0.7**	0.83 **2.5**	6.11 **6.5**	5.0 **0.4**	4.9 **0.7**	3.2 **2.8**	10.1 **13.7**	0.34 **1.96**	-8.5 **-2.1**	-13.6 **-17.7**
OMG	**WASHED BRIGHTNESS**	0.26 **-0.32**	-2.68 **-0.67**	3.14 **3.03**	0.86 **0.98**	-2.54 **-1.72**	-1.06 **-0.71**	0.36 **0.85**	2.44 **1.23**	-0.95 **-0.66**	0.54 **0.96**	1.93 **2.57**
	WASHED ERIC	-2.2 **-1.1**	2.2 **1.1**	-11 **-5.5**	-12.9 **-6.5**	18 **9.0**	24 **12**	-8.9 **-4.4**	-5.7 **-2.9**	-5.7 **-2.8**	-10.6 **-5.3**	-64 **-32**
	% YIELD	-3.6 **-4.1**	7.4 **3.4**	1.22 **0.4**	-6.9 **-7.9**	4.9 **3.6**	-4.2 **-0.8**	-7.2 **-5.9**	8.8 **11.9**	7.1 **5.9**	-2.2 **-3.3**	-14.8 **-14.4**
HL1	**WASHED BRIGHTNESS**	2.14 **1.07**	3.12 **1.56**	1.99 **1.00**	2.10 **1.05**	2.99 **1.50**	-1.15 **-0.58**	1.92 **0.96**	2.2 **1.10**	-0.63 **-0.32**	2.81 **1.41**	4.46 **2.23**
	WASHED ERIC	-23.7 **-11.86**	-13.9 **-6.93**	7.5 **3.74**	-1.76 **-0.88**	12.0 **6.01**	20.9 **10.46**	-13.9 **-6.94**	-20.8 **-10.42**	5.6 **2.81**	-11.8 **-5.89**	-16.0 **-8.02**
	% YIELD	-7.52 **-3.76**	5.0 **2.50**	-2.97 **-1.48**	0.48 **0.24**	-0.58 **-0.29**	0.42 **0.21**	-1.91 **-0.95**	6.8 **3.38**	-1.9 **-0.97**	0.95 **0.47**	-3.05 **-1.52**
HL2	**WASHED BRIGHTNESS**	1.36 **0.68**	3.39 **1.69**	4.93 **2.46**	-4.87 **-2.43**	-2.05 **-1.03**	7.06 **3.53**	-3.87 **-1.94**	1.38 **0.69**	-0.48 **-0.24**	-0.56 **-0.28**	-0.44 **-0.22**
	WASHED ERIC	4.9 **2.43**	-9.2 **-4.60**	7.1 **3.53**	12.9 **6.45**	8.1 **4.03**	-7.1 **-3.57**	-5.5 **-2.74**	18.0 **9.02**	7.6 **3.80**	2.5 **1.27**	-15.3 **-7.62**
	% YIELD	-4.6 **-2.30**	-3.6 **-1.82**	-1.8 **-0.91**	5.0 **2.51**	3.4 **1.68**	-4.3 **-2.20**	-2.8 **-1.38**	8.8 **4.39**	7.3 **3.64**	2.9 **1.46**	-16.8 **-8.40**

Table 4: Ranking analysis

		WASHED BRT				WASHED ERIC				YIELD						
No.	FACTOR	ONP	OMG	HL1	HL2	ONP	OMG	HL1	HL2	ONP	OMG	HL1	HL2	Mean	Highest	Lowest
11	**tf, min**	2	2	1	11	1	1	4	2	1	1	4	1	2.58	1	11
8	**% cons**	5	4	5	7	4	8	3	1	2	2	2	2	3.75	1	8
1	**%NaOH**	6	11	6	8	3	10	1	10	3	6	1	5	5.83	1	11
3	**%H2O2**	1	1	8	2	11	5	9	8	6	11	5	11	6.50	1	11
6	**Tp, deg C**	11	8	10	1	9	2	2	7	10	10	11	6	7.25	1	11
2	**% Sod Sil**	4	9	2	5	2	11	6	4	9	8	3	7	5.83	2	11
4	**% Surf-p**	8	5	7	3	8	4	11	3	4	3	10	4	5.83	3	11
5	**tp, min**	3	3	3	6	5	3	7	5	11	7	9	8	5.83	3	11
9	**pH**	7	10	11	10	10	9	10	6	8	5	6	3	7.92	3	11
7	**Tf, deg C**	10	7	9	4	6	7	5	9	5	4	7	10	6.92	4	10
10	**% Surf-f**	9	6	4	9	7	6	8	11	7	9	8	9	7.75	4	11