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Enzymatic Deinking of Flexographic Water-Based Inks

Somporn Chaiarrekij

ENZYMATIC DEINKING OF FLEXOGRAPHIC WATER-BASED INKS

by

Somporn Chaiarrekij

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Somporn Chaiarrekij

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CHAPTER I

INTRODUCTION

In recent times, environmental concerns have led to the development of water-based flexographic and rotogravure inks. One of the major concerns for water-based inks is their deinkability. Wash deinking of flexographic water-based ink is much more effective than flotation deinking; however, it has two major disadvantages. It consumes large amounts of water and gives a lower yield than flotation due to the loss of fines and fillers in the effluent (1).

Current flotation deinking provides a poor brightness of deinked pulp due to binders used in flexographic water-based inks. These binders are resolubilized when the printed paper is repulped under alkaline conditions. This contributes to both an increase in the released ink particles from fibers and the subsequent dispersion of them into very small particles. These small particles being hydrophilic have few chances to collide and attach to air bubbles and float to the surface of the flotation cell. On the other hand, they tend to deposit back onto paper fibers.

However, it has been found in some recent research (2-4) that deinking of flexographic water-based inks can be improved by using neutral or acidic condition in repulping and flotation processes. It may also be possible to use enzymatic deinking with flexographic water-based inks. Some research works (5-10) show that enzyme can

be used to improve deinking efficiency considerably, especially with offset, letterpress, and xerographic printed paper. In order to promote enzyme activity, repulping under acidic condition is required and this condition may be adequate for flexographic water-based inks.

In this study, the enzymatic repulping conditions (pulping time, displector concentration, and dosage of enzyme) will be varied in order to determine the optimum condition that provides the highest deinking efficiency. The criteria used to estimate the deinking efficiency will be brightness, freeness, yield, pulp viscosity and water retention value.

CHAPTER II

LITERATURE REVIEW

Deinking Process

Deinking process can be divided into ten basic steps. These steps are pulping, pre-wash (heat and chemical loop), screening (coarse and fine screening), through flow cleaning or reverse cleaning, forward cleaning, washing, flotation, dispersion, bleaching, and water recirculation and makeup (11). Depending upon the initial furnish and the final product requirements of the deinking system, all or some of these ten basic steps may be required and depending upon the final requirements of the system, these process steps can be put in various sequences. Out of all these steps, the following four steps are very important.

Pulping or Repulping

Pulping is a critical operation in deinking because in this stage ink is removed from the fiber and the particle size is most efficiently controlled (12). Pulping in deinking plant may be batch or continuous. However, the batch pulping method is more common since it gives better control of the process. Pulping is also the most common point of chemical addition. Chemicals that are normally added to the pulper just prior to the furnish addition may consist of sodium hydroxide, sodium silicate, hydrogen

peroxide, a surfactant, and a chelating agent. Consistency in the pulper is usually between 4 and 6%. However, higher consistency (12-15%) is preferred today because of improved fiber-to-fiber interaction and savings in chemicals, heating costs, and operating personnel.

Washing

Washing is a series of dilution and thickening steps repeated enough times to produce a clean pulp (12). Dispersants are usually added in order to keep the particles small enough to be removed through washer screen wire and hydrophilic enough so that they will drain readily. Ink particle sizes approximately below 10 microns are necessary in order to achieve effective washing system.

Flotation

Flotation deinking is now becoming much more common in North America. The prime reasons for the growing popularity of the flotation process are its relatively low process water consumption and the concentrated, easily handled ink sludge it produces. Also, inks such as UV cured, heatset and non-impact printing inks cannot be deinked by washing.

In contrast to washing, flotation involves the addition of chemicals which are usually "collectors" in order to make the ink hydrophobic and then agglomerate this hydrophobic ink into larger particles. Then they will attach to air bubbles and float to

the surface for removal from the flotation cell. The optimum particle size for effective ink flotation is generally in the range of 10 to 100 microns.

Dispersion

An alternative method of treating ink particles is to disperse these particles in small enough size so that they cannot be visible to the naked eye. This residual ink may dull the overall brightness of the sheet slightly, but no ink specks would be seen (13). This dispersion process has been successfully utilized on inks such as ultraviolet inks, xerographic inks, and laser print type inks which are normally difficult to remove.

Deinking Chemicals

Deinking chemicals are selected based on wastepaper, ink types, design of the deinking system (washing and/or flotation), and the desired quality of deinked stock going to the paper machine. Table 1 shows the principal deinking chemicals and the potential places of application. Out of all these chemicals, the most common ones such as surfactants, dispersants, collectors, and displectors will be discussed in more details.

Surfactants

Surfactant is a catch-all term that covers dispersants, collectors, wetting agents, displectors, anti-redeposition aids and the like (14). Surfactants have two principal portions: one, hydrophilic (water-loving) and the other, hydrophobic (water-hating) portion. The hydrophobic portion consists of carbon and hydrogen atoms and may be

linear or branched, saturated or unsaturated. Depending on the hydrophilic portion, surfactants can also be divided into ionic and nonionic surfactants.

Table 1

Principal Deinking Chemicals (14)

Chemical	Application
Sodium hydroxide	pulper, bleaching
Sodium silicate	pulper, bleaching
Chelating agents	pulper, bleaching
Hydrogen peroxide	pulper, bleaching
Surfactants	pulper, flotation, washing
Collector chemicals	pulper, flotation
Agglomeration chemicals	pulper, cleaners
Calcium chloride	flotation
Dispersants	washing, stock prep
Sodium hypochlorite	bleaching
Sodium hydrosulfite	bleaching
Formamidine sulphinic acid	bleaching
Contaminant control	pulper, storage, stock prep
Clarification polymers	clarification

Ionic surfactants can be cationic (positive charge), anionic (negative charge), and amphoteric (both positive and negative charges) (Table 2).

Nonionic surfactants having no charge are most frequently used since they function independently of pH and water hardness. Two of the more common nonionic surfactants used for deinking are the ethoxylated alkyl phenols and ethoxylated linear alcohols. These two surfactants are used as dispersants in washing process.

Table 2

Table of Surfactant Ionicity (17)

Anionic (Negative)	Cationic (Positive)	Amphoteric (Neg + Pos)	Nonionic (No charge)
Sulphonates Sulphates Carboxylates Phosphates	Ammonium- Pyridinium- Imidazolinium- Piperidinium- Sulphoxonium- compounds etc.	Aminocarbon acid etc.	Akyl- Alkylaryl- Acyl- Acylamid- Acylamin- Polyglycol ethers Polyolester Alkanolamid Ethyleneoxide Propyleneoxide

When a surfactant is added into the pulper, or just prior to flotation, the hydrophilic portion remains in the water while the hydrophobic portion will associate with the ink oil and dirt. This surfactant action is the formation of "micelles" and is shown in Figure 1.

Surfactants function in deinking systems by decreasing the surface tension of water to provide more effective wetting, adsorbing on surfaces to help in ink removal and dispersion, and by solubilization and emulsification. To get the best performance of a surfactant system, the formulation is frequently a blend of many components, often upto 4 different chemicals. It is occasionally mentioned that blends of surfactants will give synergistic performance (14).

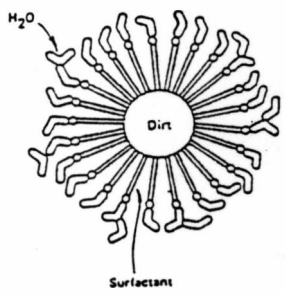


Figure 1. Formation of Surfactant Micelle (14).

Dosage levels of surfactants can vary significantly depending on the chemical type and deinking condition. However, levels usually fall between 0.2% and 2.0% based on O.D. fiber.

Dispersants

Dispersants are primarily found in wash deinking mills. The most commonly used wash deinking dispersants are surfactants such as nonylphenol ethoxylate, alkylphenol ethoxylate, and linear alcohol ethoxylate. Figure 2 shows the structures of dispersants commonly used in washing process.

Dispersants decrease the surface tension to increase contact area between the ink and the fiber. They also not only break ink particles down and keep them small enough to be removed by washing, but provide ink particles hydrophilic character through micelle formation in order to drain easily as well. The optimal level of dispersant addition is about 0.2-2.0%.

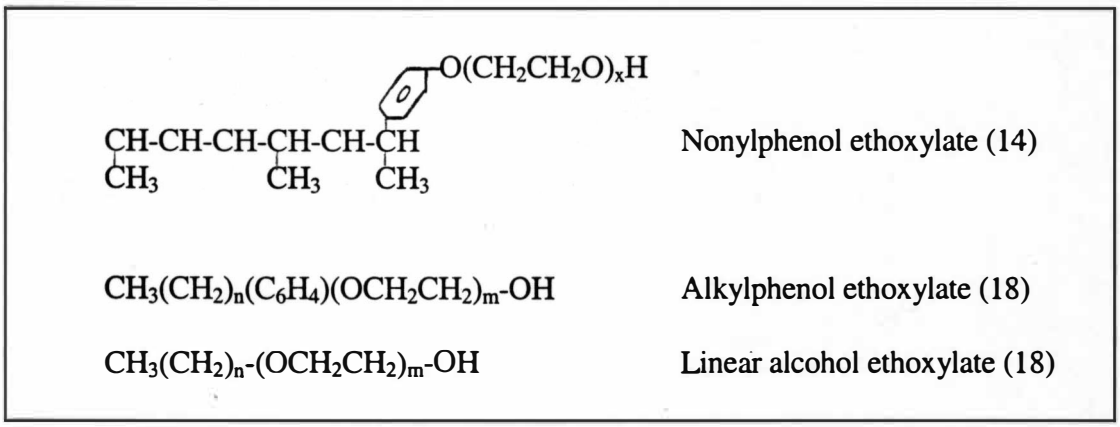


Figure 2. Dispersants Used in Washing Process.

Some hydrophilic polymers are good dispersants but they are not surfactants. They are water soluble and multi-functional polyelctrolytes. Besides particle dispersion, these polymeric dispersants sequester cations, inhibit scale, and exhibit some detergent properties (12). Polyacrylates and diisobutylene maleic anhydride copolymer are two common examples of polymeric dispersants. The optimal dosage of these polymeric dispersants is 0.1-0.5%.

Collector Chemicals

Collector chemicals are commonly used in flotation deinking. They can be added either at the pulper or just prior to the flotation cell. For ink removal to occur, the ink particle must come into contact with the collector chemicals, which in turn must come into contact with the air bubbles so that the ink agglomerates can be removed at the surface of the flotation cell.

The formulation of the collector chemicals helps to adjust the surface tension of the air bubbles so that the bubbles will have sufficient surface tension strength to carry the ink particles through the surface of the flotation cell.

The most common type of collector chemicals is a fatty acid soap with the presence of calcium ions. However, there are synthetic collectors such as Eo/Po (Ethylene oxide/Propylene Oxide copolymers) and blends such as ethoxylated fatty acids.

Fatty Acid Soap / Calcium Ions

Calcium ions can be introduced to the deinking system from the hard water, from the waste paper itself in the form of calcium carbonate from the coatings or fillers, and from some calcium rich chemicals such as calcium chloride, calcium formate, etc. To maximize the use of source of calcium ions in the paper, it is essential to maintain the correct pH level about 8.5 at the flotation stage in the system (19). For good flotation, the level of calcium ions needed is 200 ppm measured as calcium carbonate equivalent or the water hardness must be at least 12 degree German hardness (dH).

Major concerns when using calcium ions are not only that the calcium ions are believed to cause scaling and other deposits on a paper machine and deinking plant but that high calcium ion levels contribute to stock loss as well. Schwinger and Dobias (20) studied the effect of calcium ion concentration on stock loss and found that fiber loss increased from 1.5% to 9.0% as the calcium ion concentration increased from 0 to 60 degree dH. They concluded from their work that calcium ions adsorbed on the fiber

surface and the adsorption of calcium reduced the negative charge of the fiber considerably. This enhanced the flotation of the fiber only if a few hydrophobic groups were present on the fiber.

Synthetic Collectors

Synthetic collectors are easier to control and do not require calcium ions for ink collection. They perform at much lower dosage and the usual dosage is 0.10-0.35%. Synthetic collectors may be added to the pulper or just before the flotation depending upon the design of the deinking system.

The study of Jarrehult, Horacek and Lindquist (3) showed that the flotation deinking of water-based flexographic newsprint inks was more effective when synthetic collectors were used, especially at a lower pH of 7.

Displectors

Displectors are surfactants that are a combination of Dispersants and collectors and have been developed in order to obtain the maximum benefit of the combination of flotation and washing system. Displectors are synthetic, 100% active liquids and usually proprietary formulations of alkoxylated fatty acid derivatives. They have the physical properties of both dispersants and collectors. Thus, they provide good adhesion to air bubbles in flotation and are so hydrophilic that fine particles do not reprecipitate onto the fiber surface (21).

Horacek and Jarrehult (21) studied the effect of displectors on brightness in combined flotation/washing system. The results indicated that the brightness gained across flotation with displectors was lower than with conventional fatty acid soaps and the brightness gained across washing was less than with dispersants; however, when the two were combined in the same mill then the brightness gained was better than conventional chemistry.

Displectors also offer additional process advantages. They are insensitive to water hardness and will not contribute to scaling on equipment. They also result in lower fiber loss and very low chemical carryover.

Flotation Chemistry

The removal of ink particles in flotation deinking consists of these processes: (a) the detachment of ink particles from the fibers, (b) the agglomeration of detached ink particles by collector chemicals, and (c) the attachment between ink particles and air bubbles and ascent to the surface of the flotation cell where they are removed.

For a successful flotation to take place in any system, several criteria must be fulfilled (22-25). First, the particles must collide with the air bubbles. This step is controlled by hydrodynamic forces in such a way that small particles have a tendency to follow the streamlines around the bubbles rather than really colliding with them. Next, the collision must induce a rupture of the thin liquid film between the particle surface and the air in the bubble. This can happen when the particle and the bubble do not repulse each other due to colloidal forces. In addition, the thin liquid film must not have

too high an elasticity resulting from adsorption of surfactants. The particle surface must also be adequately hydrophobic to affix to the bubble.

When the bubble with the attached particle is rising through the liquid, it must be stable enough to sustain both the gravity force and the viscous drag which tend to pull the aggregate apart. The force keeping the aggregate together is the surface tension of the liquid surrounding the air bubble, multiplied by the cosine of the air-liquid-particle surface contact angle. Consequently, flotation will be favored by a relatively high surface tension of the liquid giving a theoretical optimum at 90 ^{o} contact angle (22). Because of the wide variation in size, shape and roughness of particles, no single value of the contact angle can be cited that satisfies the requirements for flotation; however, a value of 50-75 ^{o} is a typical minimum requirement (26,27).

The Importance of Ink Particle Size

Figure 3 illustrates the particle size distribution against removal efficiency. As shown in Figure 3, the optimum size range for flotation is between 10 microns and 100 microns. Figure 4 indicates the reason why the particle removal is effective for the size range of 10-100 microns. If the particle is too large compared to the size of the bubble, the high turbulence in the flotation cell will dislodge the particle from the surface of the bubble before it can be carried to the surface of the flotation cell. If the particle is too small compared to the bubble size, then the surface tension and the electrostatic double layer forces come into effect and the particle cannot adhere to or penetrate the surface of the bubble.

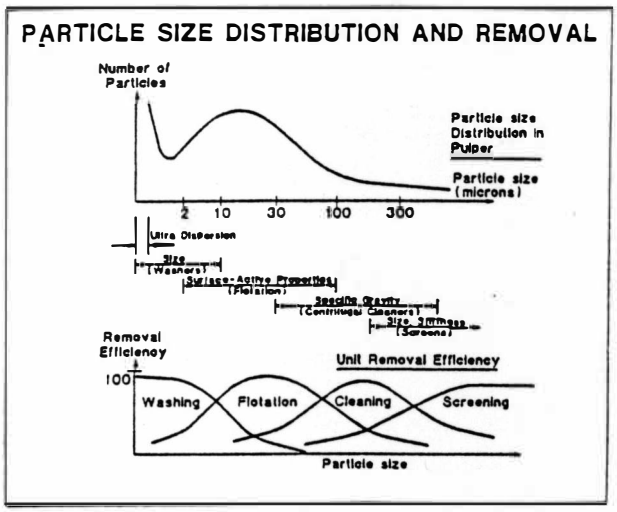


Figure 3. Particle Size Distribution and Removal (28-30).

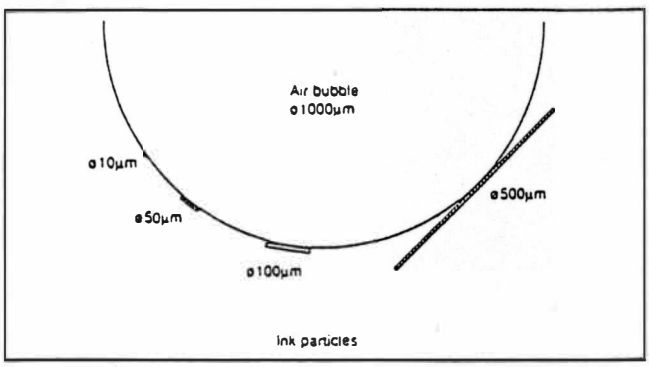


Figure 4. Model of Contact Surfaces (28,29,31).

Small particles also tend to follow the streamlines around the bubble rather than actually colliding with air bubbles. Therefore, the probability of collision between small particles and air bubbles decreases while the probability of collision is higher if the particles are larger.

The Importance of Bubble Size

Controlling bubble size is a major key for flotation cell performance. Bubbles with a diameter larger than 0.3 mm. have sufficient buoyancy to push through the elastic network formed by the fibers in the suspension (29,31). On the contrary, bubbles with a diameter smaller than 0.1 mm. have a tendency to stick to the fibers, causing fiber removal during flotation (29,32).

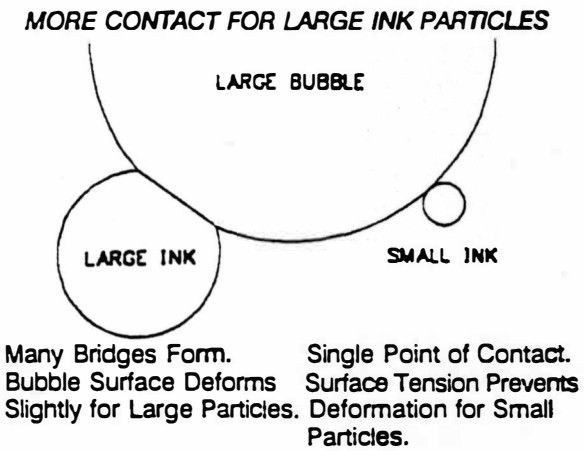


Figure 5. The Effects of the Large Bubble (33).

Figure 5 illustrates the effects of large bubbles. Large bubbles rise quickly and take with them a few large ink particles. Large bubbles take large ink particles because the large bubble surface allows sufficient contact area for large ink particles to adhere and the contact angle between the bubble and ink particle allows many bridges to form. The bubble shape will alter slightly to accommodate large ink particles. Surface tension prevents such accommodation for small ink particles.

If bubbles are too small, they are trapped among the network of fibers and do not rise due to their low buoyancy. However, if they do rise, they will take suspended solids like fibers and fines with them to the surface of the flotation cell. It has been theorized that an air bubble/ink particle size ratio of 5:1 is optimum for efficient removal (28,29).

Role of Mixing

There is no driving force, no attraction between the particles and air bubbles. Thus, if the two are to come into close contact, this must be achieved through mixing. Mixing takes place in the injector area where high stock velocities and turbulence compel air bubbles and aggregated ink particles together. Good mixing also ensures a maximum rate of collision between the bubbles and the particles.

Stabilization of Bubble-Particle Aggregates

To ensure a successful flotation deinking, the ink particle-bubble aggregates must be stable enough to sustain the external stress forces on their long journey to the surface of the flotation cell. Forces exerted on the ink particle-bubble aggregates include the gravity force, buoyancy force, capillary force on the three-phase-contact line, capillary pressure in the air bubble exerted on contact area, and detaching force resulting from the turbulence of flow field in flotation cell (34).

Hou and Hui (34) studied the stabilization of bubble-particle aggregates and concluded that the capillary force is the most dominant force. Contact angle and the penetration distance of the particles into the bubble strongly influence capillary force. Particles with higher contact angle will cause deep penetration and stronger attachment

whereas particles with low contact angle only allow very shallow penetration. Stability of the particle-bubble aggregates decreases rapidly with increasing particle size and lower surface tension will destabilize the particle-bubble aggregates.

Consequently, liquid surface tension, surface energy of ink particle, ink particle size, air bubble size and the external turbulence are important factors to determine the stability of the particle-bubble aggregate.

Flexographic Water-Based Ink

Flexography or flexo is a specified form of rotary letterpress. The use of flexographic printing becomes more attractive because it can print not only on impermeable substrates like metal or film, but also on porous substrates like paper or board as well. It can use either water-based or solvent-based inks. However, water-based inks have gained more popularity for the main reason of reduced volatile organic compounds (VOC) emission.

Generally, printing inks have the following ingredients:

1. Pigments - Pigments provide color and opacity to the ink. They also affect the flow properties of the ink.

2. Binders - Resin is the main component of binders. Resin is used to disperse the pigments and then to bind them on the surface of the paper after printing.

3. Solvents - Solvents maintain the ink fluidity and ensure the ink transfer from the inking system to the paper.

4. Additives - Additives consist of waxes, drying agents, anti-oxidizing agents, surfactants, etc.

Table 3 illustrates a typical flexographic water-based ink composition (35).

Table 3

Typical Flexo Water-Based Ink Composition (35)

Components	Black	Color
Water	65-77 %	65-80 %
Binder		
Resins	9-12 %	5-8 %
Organic amines	0.1-0.3 %	0.1-0.3 %
Pigment	12-16 %	12-20 %
Additives	2-10 %	2-10 %

Amines are added to neutralize the acid elements of the resin and then form soluble salts in the aqueous media. Waxes and silicones help to increase the surface resistance and the friction coefficient whereas antifoams and surfactants increase the wetting of the ink by decreasing the surface tension. Biosides and fungicides are added to inhibit the growth of microorganism (36,37).

Flexo water-based inks contain acrylic resins that become soluble when neutralized by alkalies such as amines. During the printing process, the amines evaporate along with water and/or are neutralized by the acidity of the paper. Instantly,

the carboxylate groups of the resin are converted into carboxylic acids. The acid groups are much less polar which helps to release the water from the ink film and precipitate the resin with the pigments to set the printing image. Figure 6 illustrates this relationship.

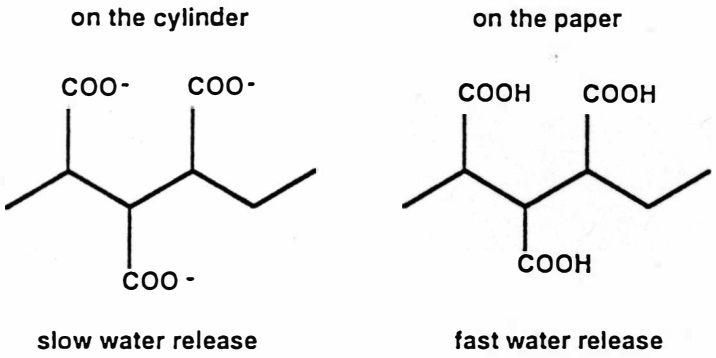


Figure 6. Ink Drying (2).

The conventional deinking process in alkaline medium gives the best conditions for dislodging and dispersing printing inks for removal. However, for flexo water-based inks, this alkaline medium produces over-dispersion to very small ink particles, about 0.3-2.0 micron in size (29). This is because when the paper is repulped under alkaline condition, the acrylic resins are resolubilized by forming soluble anionic carboxylates. This increases the release of ink particles from fibers and overdisperses them in the water phase.

These small particles are also hydrophilic due to the presence of carboxylate group which favor dispersion in the aqueous phase. This makes flexographic water-based inks incompatible with flotation deinking but removable by wash deinking.

Deinking of Flexographic Water-based Inks

Difficulty in Deinking Process

Ackermann et al. (38) studied the deinkability of flexographic water-based inks and found that flexo water-based inks were less easily removed by flotation process when compared to offset and rotogravure inks (Figure 7).

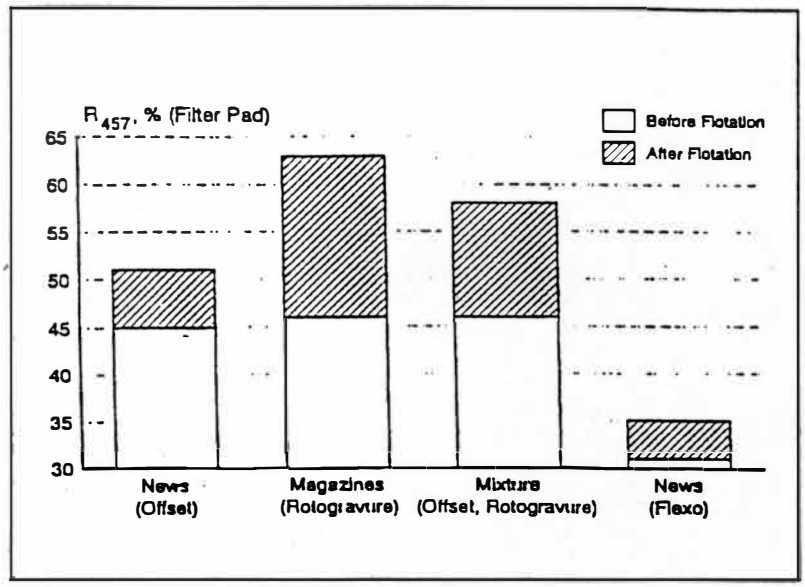


Figure 7. Effect of Classic and Water-Based Inks on Brightness of Filter Pad (38).

They also examined the effect of flexo water-based print content on mixed wastepaper using both single and double flotation. The brightness of the deinked pulp without flexo water-based portions which reached 66% after double flotation was set to 100% relative brightness.

As shown in Figure 8, a 10% flexo water-based print content in mixed wastepaper was adequate to destroy all advantages of the second flotation stage because

the brightness of 62% was certainly the same as the single flotation stage deinked flexo-free furnish.

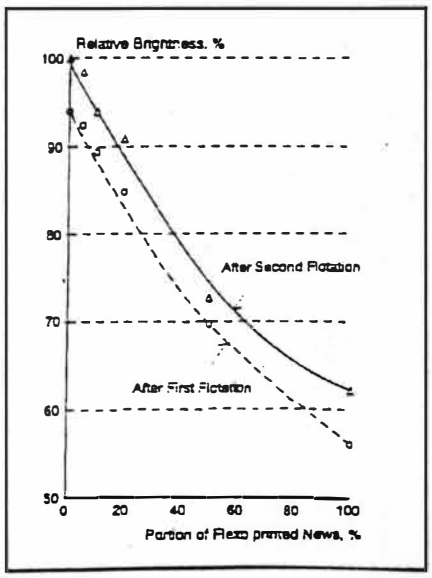


Figure 8. Relative Brightness After First and Second Flotation Depending on Proportion of Flexo Printed News (38).

Rangamannar et al. (39,40) compared 100% flexo water-based newsprint and 100% offset newsprint for their deinkability. Figure 9 clarifies that starting brightness at the pulper with 100% flexo water-based printed paper was considerably lower than with 100% offset printed paper although both furnishes contained approximately the same amount of ink.

It can be noticed from Figure 9 that flotation significantly contributed to flexo water-based ink removal but could not remove them completely. Therefore, washing was required to achieve removal of flexo water-based inks.

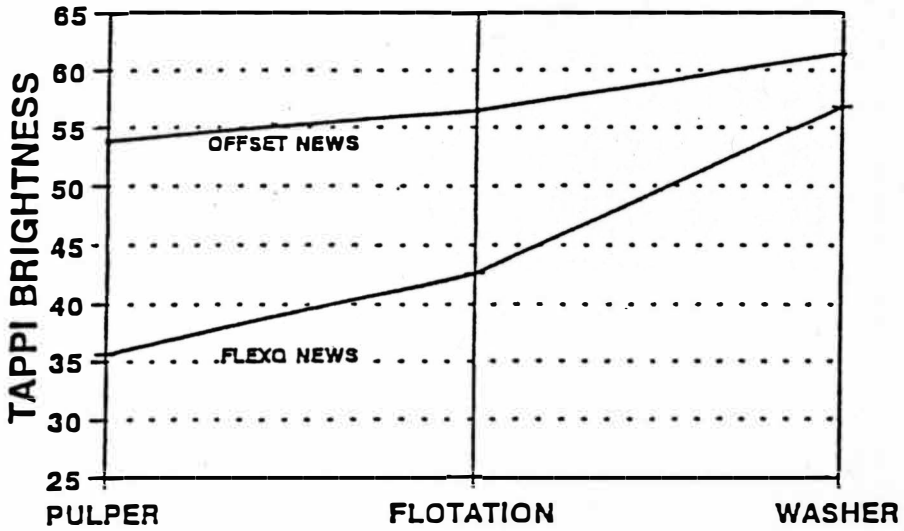


Figure 9. Brightness Across System (39,40).

Image analysis was performed on the pulped samples before flotation in order to determine the particle size distribution. The results indicated that the size range for the flexo water-based ink was much smaller than the offset ink and the majority of the particles were below 6 microns. It is evident that during pulping flexo water-based inks are dispersed into very fine particles due to the solubilization of their acrylic resins in alkaline medium.

After running the 100% furnishes, trials were run with furnish mixtures from 100% offset newsprint to 100% flexo water-based newsprint in order to study the effect that different addition levels of flexo water-based inks would have on the end product. It is shown in Figure 10 that up to a level of 10% flexo water-based inks, the brightness of the final product was not significantly reduced. However, at about 20% and above there was a definite reduction in the brightness of the final product. It is also clear that washing is more efficient than flotation for removing the extremely small ink particles.

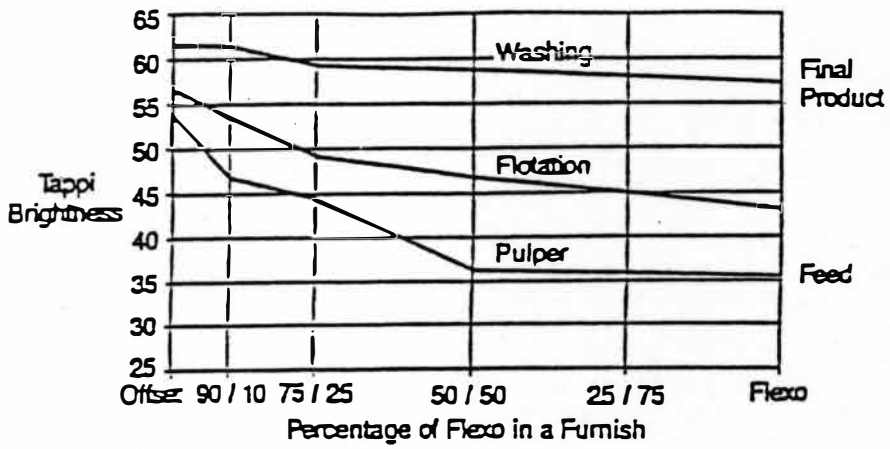


Figure 10. Influence of Quality of Flexo in a Furnish (39,40).

The effect of the amount of flexo water-based printed newsprint on the flotation deinking of mixed wastepaper (old newsprint and magazine) was also examined by Tremont (41). The results as presented in Figure 11 illustrate that the efficiency of flotation deinking with fatty acid soap was reduced by 50% when the flexo content of the old newsprint furnish reached 20% because the brightness increased from pulping to flotation dropped from 12 points with no flexo to 6 points with 20% flexo.

The study of Mah, Reid, and You (42) also indicated that deinked pulp brightness was affected by various quantities of flexo water-based printed materials. The brightness continuously dropped when increasing the amount of flexo water-based printed substrates up to 60 %. They also examined the aging effect of the prints and concluded that a maximum deinking response was obtained after the prints had been aged for approximately one month. Finally, they pointed out that there are different

formulations in flexo water-based inks and all of them do not respond in the same manner in the flotation deinking process.

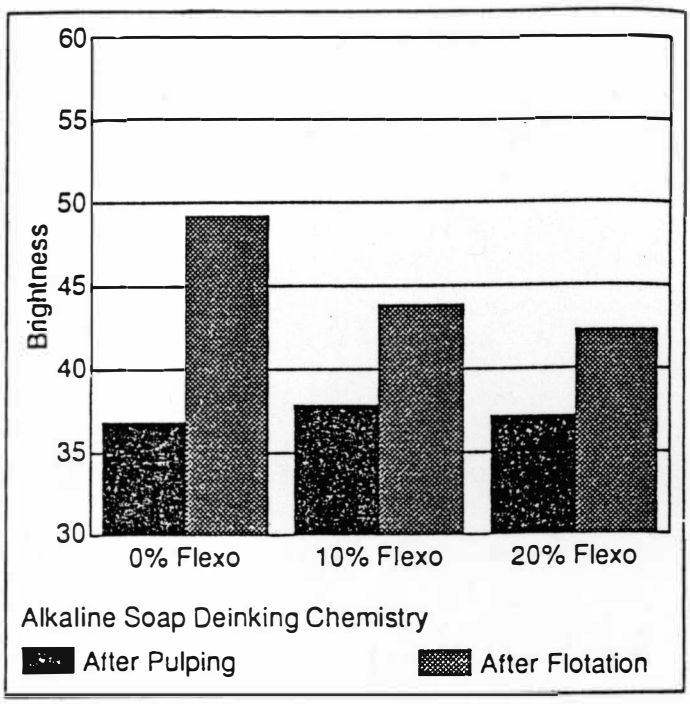


Figure 11. Effect of Flexo Printed News on Flotation Deinking (41).

Improvement of Deinking Process

Ackermann et al. (38) studied the effect of pulping consistency on the flotation deinking using 100% flexo water-based furnish. It was revealed from their studies that pulping consistency had too small an effect on particle size to sufficiently improve flotation deinking (Figure 12).

However, it is evident from Figure 13 that the results of wash deinking was strongly dependent on pulping consistency. The highest brightness of 50 % was

obtained at low pulping consistency (4%) but the brightness reduced to 40% when the pulping consistency was increased to 18%. They made a conclusion that at higher consistency, higher shear forces decreased the ink particle size and led to lower brightness after pulping.

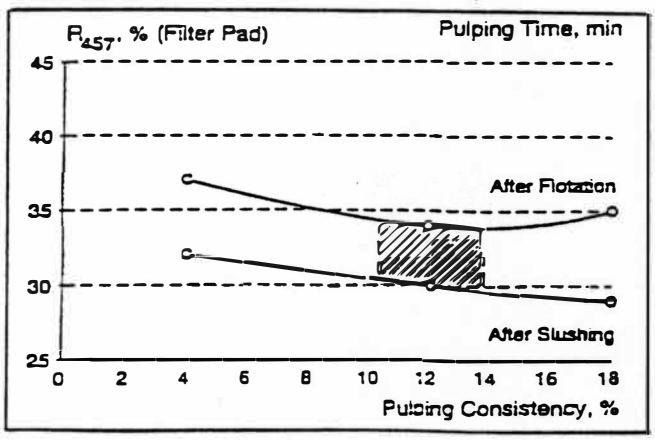


Figure 12. Effect of Pulping Consistency on Brightness Gain by Flotation of Flexo Printed News (38).

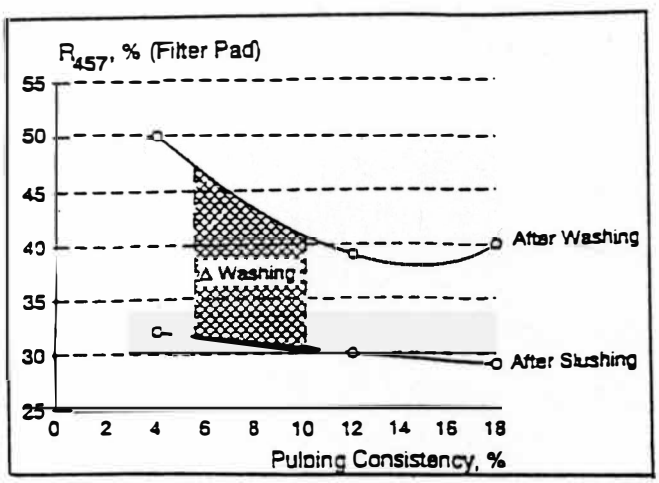


Figure 13. Effect of Pulping Consistency on Brightness Gain by Washing of Flexo Printed News (38).

Galland and Vernac (4) stressed that causes of deinking difficulties with flexo water-based inks are the small size of ink particles, the lack of hydrophobic character of the inks , and the tendency of the ink to redeposit on fibers. They also proposed several methods to improve the deinkability of flexo water-based inks:

1. Changing pulping condition - A reduction of pulping time from 15 to 5 min. with 100% flexo water-based newsprint could increase the brightness of the floated pulp from 32% up to 37%. This effect was certainly due to a reduction of ink fragmentation. However, this change is not practical for industrial mixed wastepaper supplies.

2. Preventing ink redeposition – Some appropriate chemicals such as, CMC (Carboxymethylcellulose) could decrease the ink redeposition on fibers. However, their experimental results did not show any improvement in ink removal during the flotation process. The brightness was increased after thickening and hyperwashing and this confirmed the reduction of ink redeposition.

3. Reducing pH - Figure 14 indicates that a reduction of pH could improve deinking efficiency of flexo-water based inks. As illustrated in Figure 14, without the surfactant (Berocell 213) the brightness increased with decreasing pH. However, when the surfactant was used, the brightness was at a maximum when the pH was close to 7.

4. Two-stage process - This process was designed to improve the deinkability of wastepaper mixture including various amounts of flexo water-based printed substrates. It consists of a first non-alkaline stage for removing flexo water-based inks before they are dispersed. A second alkaline stage including peroxide bleaching is for the detachment and removal of conventional inks. This second stage occurs after the removal of flexo

water-based inks. Their reasoning was that acrylic resins used in flexo water-based ink are neutralized and form larger ink particles at acidic or neutral conditions. These agglomerated ink particles can then be floated in the presence of a nonionic or cationic surfactant. The results also showed that acidic condition is better for nondispersion of flexo water-based inks but less efficient for the detachment of offset printed paper.

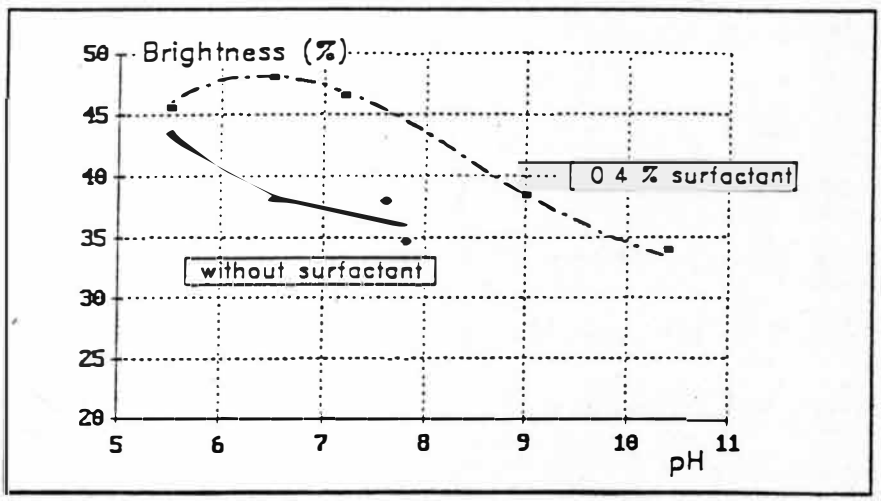


Figure 14. Effect of pH on Flotation Deinking of Flexo Printed Newspaper (4).

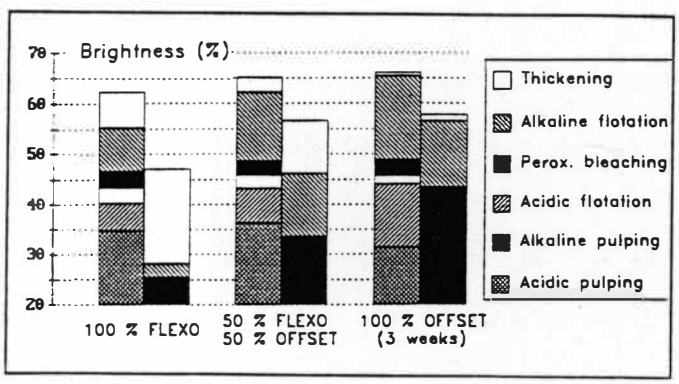


Figure 15. Comparison of Results Between Conventional Deinking and the Proposed Two-Stage Deinking Process (4).

From Figure 15, it can be concluded that with this two-stage process high brightness of deinked pulp could be obtained from all mixed wastepaper containing flexo water-based printed materials. However, the two-stage process could induce higher fiber loss and more stages were necessarily performed.

Jarrehult, Lindquist and Horacek (3) carried out the deinking experiments using flexo water-based printed materials at different amounts. The results showed that when using waste furnishes having up to 70% flexo printed substrates, the effectiveness of flotation could be significantly improved by Berocell 210, a flexo deinking additive developed by Nobel industries (NPC) compared with conventional calcium chloride/fatty acid mixtures (Figure 16). Use of this product also resulted in a higher flotation efficiency which was comparable to two stage washing efficiency. The improvement was likely due to better collection of dispersed ink particles.

In addition to the use of the Berocell 210, flotation efficiency with flexo water-based inks could also be improved by reducing the pH at the pulper to 7-8. Figure 17 illustrates that reduced pH from 9 to 7 was slightly beneficial with 0% flexo newsprint; however, the effect of reducing pH increased considerably with increased amount of flexo newsprint. This was because the resins which disperse flexo water-based inks at pH 9-10 were protonated at neutral pH and therefore would not have hydrophilic characteristics.

They also suggested that the best results can be achieved when flotation is followed by one or two washing stages. This combination of washing and flotation not

only improves deinking efficiency but also reduces the effluent load and simplifies effluent clarification as well.

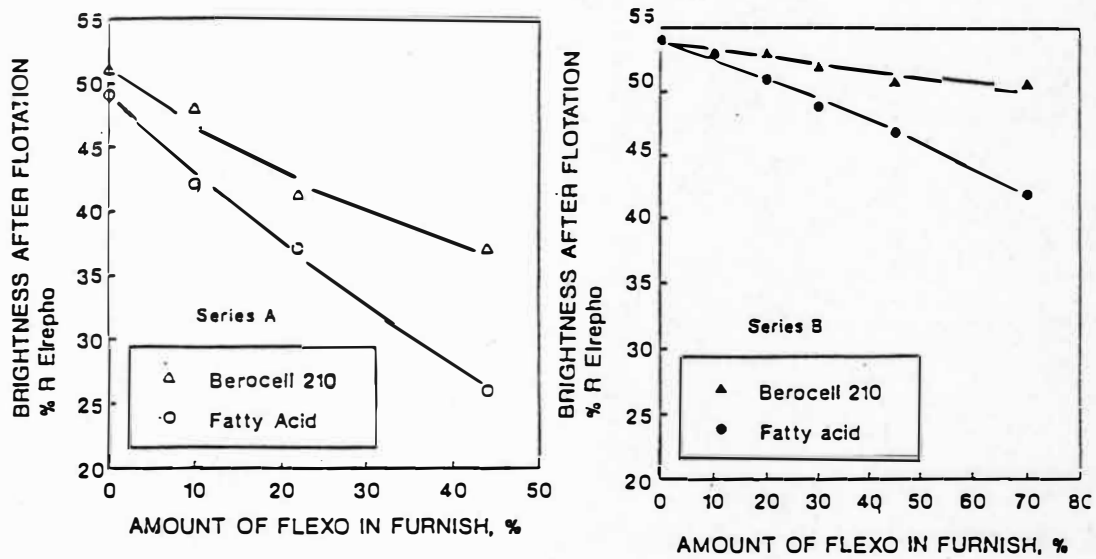


Figure 16. Improvement of Flexo Flotation by Berocell 210 (3).

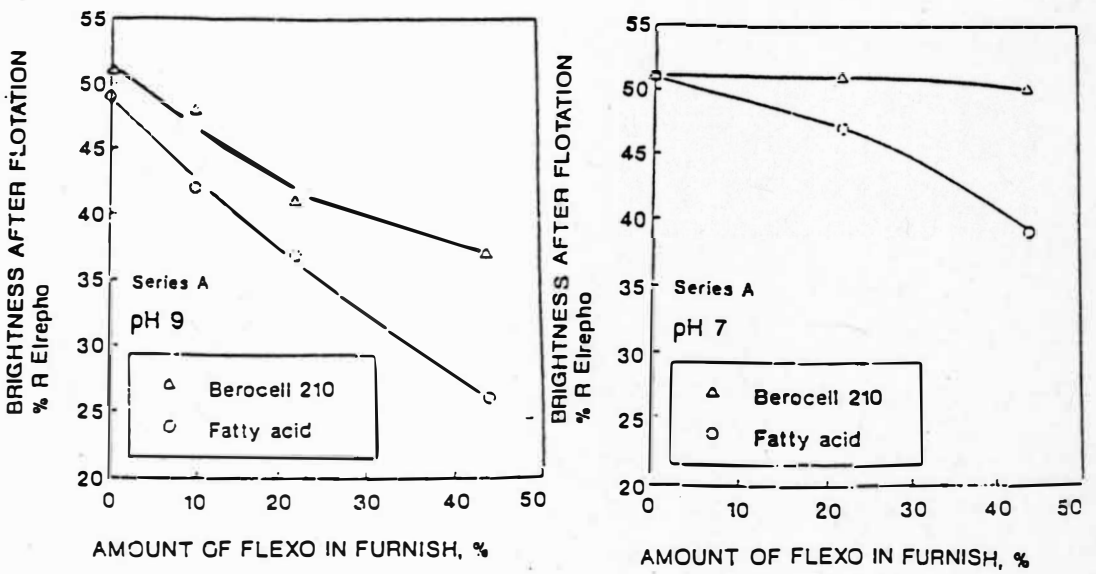


Figure 17. Effect of pH on Flexo Flotation (3).

Ellis et al. (43) examined the deinkability of flexo water-based ink under different amounts of flexo water-based inks in the waste mixture by using flotation followed by washing process. After repulping, the accept was recirculated for prolonged flotation. Samples were then taken for every two minutes of flotation time. They concluded from their studies that deinking typical mixed furnish with low percentage of flexo water-based printed newsprint could be processed with HCC-1, a commercial displector. Figure 18 shows that the pulper sample using HCC-1 had a higher brightness than that of the thrice washed pulp using the soap-based collector. Therefore, it is clear that HCC-1 can deink flexo water-based ink satisfactorily.

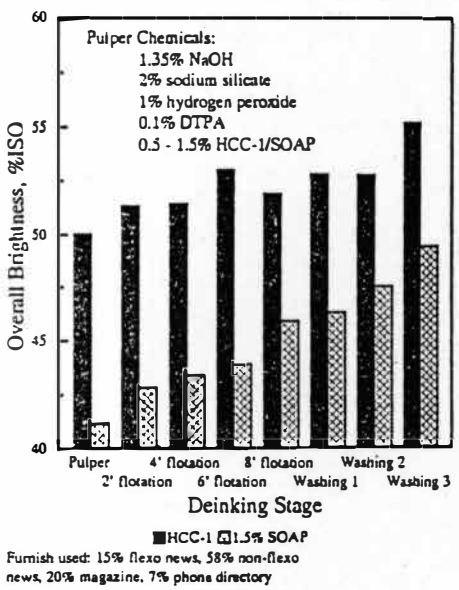


Figure 18. Deinking Wastepaper With HCC-1 and Soap (43).

Ellis, Hou, and Seenivasan (44) performed the deinking experiments using 30% old magazine and 70% old flexo water-based newsprint in a flotation/thickening

process. Only 2% NaOH and 3% product A (D-LINK™ FC 1901) were added to the pulper at a pH of about 8, 12.5% pulp consistency, and temperature of 120 °F. The results showed that good brightness of 50.1% ISO was obtained after 8 min. of flotation and a high brightness of 59.5% ISO was also obtained with only a single stage of sodium hydrosulfite bleaching when brightness after repulping was only 39.6% ISO.

They also conducted other experiments by changing not only the amounts and types of furnishes but also the sequence of deinking process. The results still showed that excellent flotation efficiency was achieved by using the product A, a chemical that agglomerates and imparts hydrophobicity to the ink particles.

Skaar (45) presented a new chemical system that efficiently removes flexo water-based, as well as non-flexo, newsprint and magazine inks in the flotation deinking process. This system is a synergistic combination of surfactant, formulated fatty acid and water soluble polymer for which patents have been filed.

Another way to improve the deinkability of flexographic water based inks is changing the ink formulation so that they are easily deinked. Nevertheless, there are two major disadvantages (46):

1. The introduction of a new property would force a compromise between printability and ink performance.

2. The flocculation and separation of pigments could occur too early in the printing process and create increasing ink instability problems in storage and on the press.

Enzymatic Deinking

The Use of Enzyme in the Pulp and Paper Industry

Enzymes are supercatalysts (47,48). They are natural proteins secreted by living organisms, like fungi, yeasts, or bacteria to catalyze reaction, the products of which will be directly absorbed. Since they are complex molecules resulting from a stringing together of diverse amino acids, they can only join with molecules which correspond to their own structure. This explains the specific qualities of individual enzyme; for example, amylase hydrolyses starch into glucose and cellulase depolymerises cellulose. Enzymes are biodegradable and since they occur in living organism, the conditions in which they can work effectively are very mild.

Cellulose, hemicellulose, and lignin are the major components of wood and are the basic materials for papermaking. For each of them, specific enzymes exist; however, of the three, the enzymes which will degrade cellulose offer the most promise, the hemicellulases less so, and the technology for degrading lignins is still in the realm of dreams (47).

Enzymes used in the pulp and paper industry are relatively large protein molecules with a diameter of around 5 nm or more (49,50). Because of their size, they act mainly on the fiber surface where they hydrolyze or partially depolymerize cellulose and hemicellulose chain structures.

Hemicellulase could be used as a bleaching aid since it can attack hemicellulose bonding with lignin and set the lignin fragments free.

The most promising use of cellulases has been found in deinking, refining, and drainage improvement. In the area of enzymatic refining, various studies have shown that a mixture of cellulases and cellobiohydrolases added during the refining in a Valley beater can decrease the beating time by 20 to 30%

Pommier (48) found that the blend of cellulases and hemicellulases could increase the freeness of the pulp without any loss of the strength. This improvement allows more dilution in the headbox and leads to better formation and improved mechanical properties.

A Japanese paper company (47) had already patented the use of cellulases in deinking, with 1 kg per ton of wastepaper, at 50 °C for two hours, or 5 kg at room temperature for five hours. The brightness could be increased up to eight points.

Enzymatic Deinking of Newsprint Wastepaper

Tae-Jin EOM et al. (5,6) studied the enzymatic deinking of newsprint wastepaper using the cellulose enzyme at a pH of 4.7. The results based on their studies are as follows:

Effect of Enzyme on Pulpimg Time and Ink Particle Size

The addition of 0.5% enzyme could considerably reduce the time required for a complete disintegration of wastepaper when compared to no enzyme treatment. Figure 19 indicates that the enzyme evidently had a strong effect on reducing the ink particle

size. They hypothesized that it may be partly due to a dispersing action of the enzyme protein in the acidic condition.

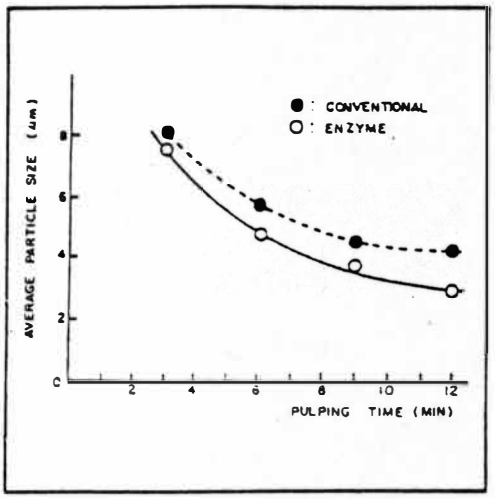


Figure 19. Change in the Size of Ink Particles Pulped in the Low Consistency (4%) Pulper (5,6).

Effect of Enzyme Dosage and pH on Brightness

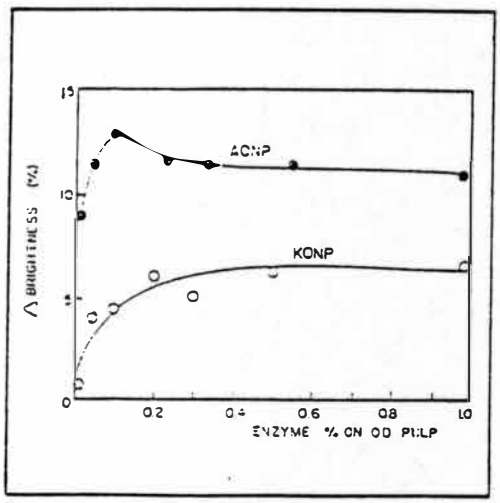


Figure 20. Brightness Gain With Change of Enzyme Addition Level (5,6).

As shown in Figure 20, the required amount of enzyme for the best flotation process appeared to be about 0.1-0.15% at which the highest brightness gain was achieved. Beyond 0.2% enzyme dosage, brightness was not increased. This was probably due to the excessive ink particle size reduction (below 2 microns). It was also found that the brightness of the deinked pulp decreased gradually over a pH range of 4.7 to 8.0 because of decreased enzyme activity.

Effect of Enzyme on Fiber Length

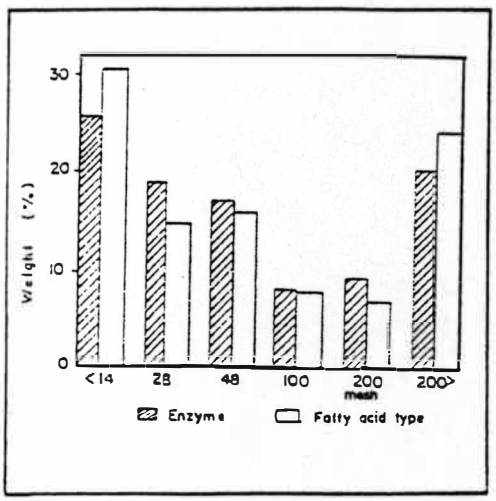


Figure 21. The Effect of Enzyme Treatment on Fiber Length Distribution (5,6).

Figure 21 shows that the apparent reduction in fiber length did not result from the enzyme treatment when compared to the conventional chemical treatment. No increase in the short fiber fraction was observed; instead the short fiber fraction of the enzyme treated pulp was comparatively lower than that of pulp treated with the alkaline

chemicals. However, medium fraction increased since the long fiber bundles were probably disintegrated by enzyme.

Effect of Enzyme on the Physical Properties

The enzyme treated pulp provided higher freeness, better drainage quality and higher mechanical strength properties, particularly burst and tensile (Table 4).

Table 4

Comparative Physical Properties of Bleached Enzyme Deinked Pulp (5,6)

Property	Enzyme	Fatty acid/soap	Syntheic displector
Freeness (CSF)	176	156	156
Bulk	2.57	2.69	2.70
Breaking length (km)	3.24	3.00	3.02
Burst Index ($kPam^2/g$)	1.60	1.33	1.48
Tear Index ($mN.m^2/g$)	2.06	2.04	2.03
Opacity	88.9	90.3	91.6
Air Resistency (sec/50 mL)	9.5	9.7	10.1

The enzymatic deinking on a mill scale led to 2 or 3 points higher deinked pulp brightness after the flotation and final dewatering stages. Besides, the enzyme treatment increased freeness and substantially reduced the effluent load of BOD and COD.

Enzymatic Deinking of Colored Offset Newsprint

Prasad et al. (7) studied the enzymatic deinking of colored offset newsprint using different mixtures of cellulases and hemicellulases. They operated each experiment at a pH of 5.5 with the addition of enzyme, surfactant and calcium chloride. In their work, no enzyme was added to the pulping stage in order to avoid confounding the effect of pulping in the enzyme treatment.

Brightness and Ink Removal

They discovered that pulp brightness was increased for all samples treated with enzyme preparations compared with the control and blank (a blank was a reslushed pulp without enzyme treatment or flotation while a control was the reslushed pulp with no enzyme treatment but with flotation). The enzyme treatment significantly improved ink removal and decreased residual ink area in all cases.

Opacity and Light Scattering

No difference was found in opacity between the control and enzyme treated pulps, but there were large differences in light scattering power. Scattering coefficient of all enzyme treated pulps was much higher than the control and blank. Brightness also increased in the same order: enzyme treatment > control > blank.

Pulp Freeness and Fiber Length Distribution

Table 5 indicates that the freeness increase in the enzyme treated samples varied from 20 to 28% when compared with the control. The control pulp had a lower freeness and higher fines. The enzyme treatment decreased the quantity of fines and slightly boosted the second coarse fraction.

Table 5

Effect of Enzyme Treatment on Offset Newsprint Pulp (7)

Property	Pulp from					
	Blank	Control	Enzyme deinking with			
			Prep.I	Prep.II	Prep.III	Prep.IV
Freeness (mL)	265	250	300	300	315	320
Fiber length distribution (fraction retained)						
+28	30	30	31	30	30	30
+48	25	26	28	29	29	27
+65	7	7	7	7	7	8
+150	7	5	6	6	7	7
- 150	30	32	27	27	27	28
Tensile index (Nm/g)	32.7	31.6	36.0	37.0	39.2	35.6
Burst index ($kPam^2/g$)	1.6	1.5	1.7	1.7	1.9	1.6
Tear index ($mN.m^2/g$)	7.8	7.5	7.9	8.3	8.7	7.8

Strength Properties

Enzyme treatment provided significant improvement in tensile, burst, and tear indexes when compared to the control pulp (Table 5).

Enzymatic Deinking of Black and White Letterpress Newsprint

Parsad et al. (8) also reported their studies in enzymatic deinking of black and white letterpress old newsprint furnish. They used different mixtures of cellulases and hemicellulases in order to better understand the effects of enzyme types and concentrations. The various amounts of enzymes added per g O.D. pulp are reproduced in Table 6.

Table 6

Application of Enzyme on Black and White Letterpress Newsprint Pulp (8)

Preparation	Dosage (U/g O.D. pulp)	Activity basis
I	0.2	CMCase
II	0.2	CMCase
III	100	Xylanase
IV	19.34	Xylanase

U: enzyme activity unit equivalent to 1 micromole of reducing sugar released per minute under the assay conditions.
CMCase: carboxymethylcellulase

Figure 22 shows that all of the enzyme treated samples had higher brightness values than control. The maximum brightness gain of 5.5 points ISO (compared to the control) occurred for the pulp treated with enzyme preparation IV. The freeness also increased in the enzyme-treated samples by 14-25% compared to the control.

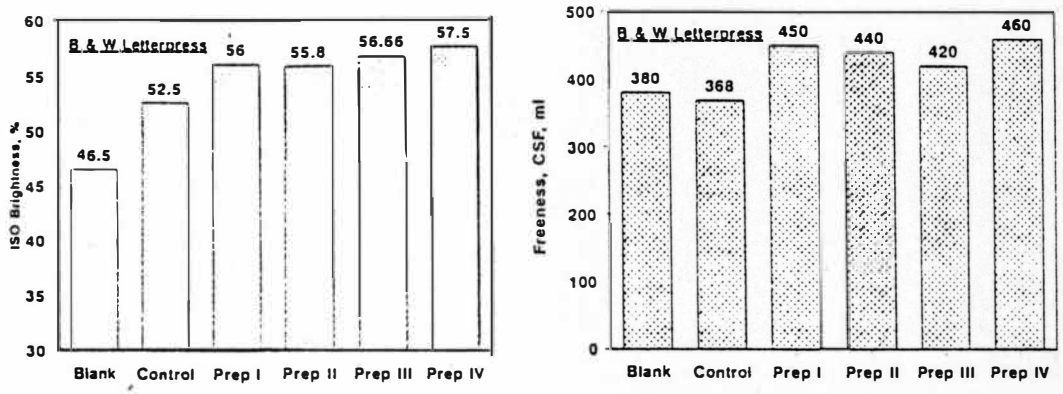


Figure 22. Increased Brightness and Freeness of Enzyme Treated Samples (8).

They also found that the enzyme treatment decreased pulp fines and increased the coarse fraction. However, the reasons for the increase in the coarse fraction of the enzyme treated samples have not been clarified.

The changes in pulp freeness and fiber length distribution after enzyme treatment have been attributed to hydrolysis (8,51,52). This creates a peeling effect that removes small elements or components which have a great affinity for water and contribute only slightly to the overall hydrogen bonding potential of the fibers (8).

The increase in the scattering coefficient of the enzyme treated samples also pointed towards the possible changes in structural parameters such as specific area or crystallinity index (52).

Strength properties such as tear index, breaking length, and burst index were also increased by the enzyme treatment. Clark et al. (53) attributed the improvement in strength properties to the changes in the hemicellulose content and the breakdown of lignin-hemicellulose linkages which facilitates the release of lignin during treatment with hemicellulases.

Enzymatic Deinking of Non-Impact Printed Paper

Jeffries et al. (9) conducted the experiments of enzymatic deinking of xerographic printed paper using a cellulase enzyme. They first optimized the enzyme treatment with respect to temperature, time and enzyme dosage. Then, they compared the optimized enzyme method with a standard chemical method using identical steps of high consistency pulping, flotation and washing.

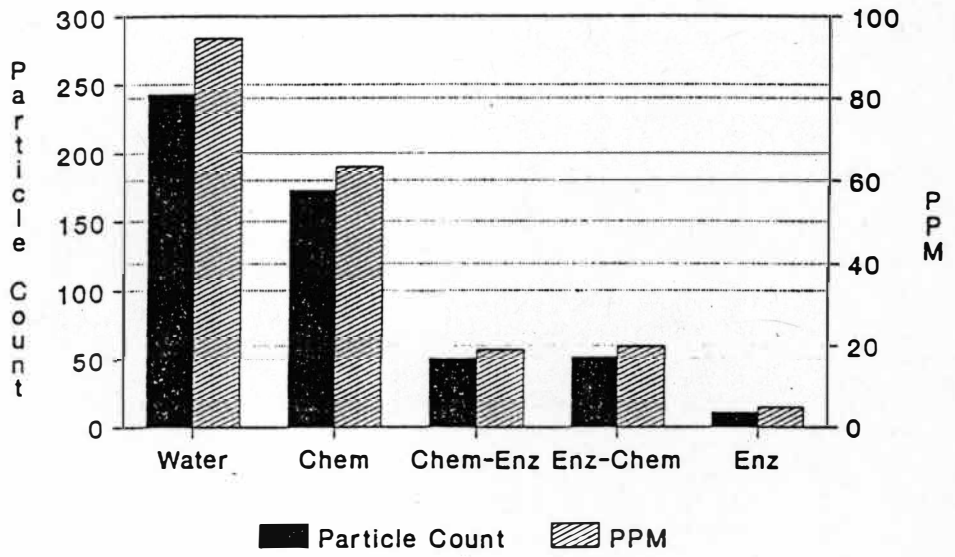


Figure 23. Comparison of Chemical and Enzyme Treatment Method (9).

Image analysis results summarized in Figure 23 show that enzyme deinking provided the most efficient ink removal with a residual count was of only 5 ppm. This is well within less than 10 ppm acceptable dirt count for deinked market pulps. They also noticed that the combination of enzyme and chemicals provided no additional benefit. This might be because residual chemicals deactivated the enzyme in the sequential treatment, or the loosened ink particles might have redeposited onto the fibers with continued pulping.

Jeffries et al. (10) also continued their studies by using seven commercial enzymes with cellulase activity, xylanase activity, or a combination of both at optimum conditions for each enzyme. They used only medium consistency pulping conditions in these experiments. The activities at the optimum recomended pH and temperature for each enzyme are summarized in Table 7.

They found that supplier X's enzymes except enzyme D were most effective at a dosage level of 0.2 mL of enzyme preparation per kg of O.D. pulp. Supplier Y's enzymes were most effective at a dosage level of 1.5 mL per kg of O.D. pulp.

Figure 24 shows the comparison of the residual ink on handsheets prepared from each treatment. The result indicated that all enzyme treatments except enzyme D were more effective than the chemical treatment for ink removal. Enzyme A, B, E, and F provided the most efficient deinking since they all removed about 96% of the ink. Table 8 summarizes the brightness, bursting strength, and freeness values for enzyme treated pulps and controls.

Table 7

Commercial Enzyme Properties (10)

Enzyme	Optimum pH	Optimum temp (C°)	Cellulase activity	Xylanase activity	Measured FPU/mL
Supplier X:					
A	6.5	55	1,500 CMCU/g	*	35
B	7.0	55	800 CMCU/g	*	27
C	5.0	55	1,500 NCU/g	*	90
D	7.5	50	*	600 EXU/g	3
Supplier Y:					
E	5.5	55	2,500 IU/mL	350 DNS/mL	84
F	5.5	55	5,300 IU/mL	1,500 DNS/mL	28
G	5.5	55	50 IU/mL	12,500 DNS/mL	2

* not available
CMCU: carboxymethyl cellulose unit
NCU: cellulose unit
EXU: xylanase unit
DNS: dinitro salicylic acid unit
IU: international unit which is equivalent to 1 micromole of reducing sugar released per minute under the assay condition (8).
FPU: the amount of enzyme necessary to release 1 micromole of reducing sugars per minute from 50 mg of Whiteman No.1 filter paper in a 60 min assay (10).

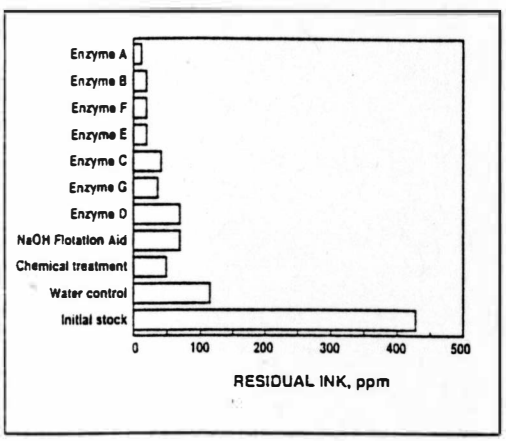


Figure 24. Effect of Enzyme Treatment on Residual Ink Particles (10).

Table 8

Properties of Enzyme Deinked Pulps and Control Pulps (10)

	Burst index (kN/g)	ISO brightness (%)	Freeness (CSF, mL)
Unprinted paper	-	85.0	-
Pulper stock	2.73	73.3	-
Control	2.50	78.3	423
Chemical*	2.78	81.3	445
Enzyme A	2.55	78.3	485
Enzyme B	2.66	78.2	488
Enzyme C	2.49	76.5	539
Enzyme D	2.50	80.2	470
Enzyme E	2.68	76.8	505
Enzyme F	2.46	78.1	500
Enzyme G	2.44	78.1	515

* This preparation used hydrogen peroxide treatment

As shown in Table 8, all enzyme treated pulps had high brightness values of 76-80 %. However, chemically deinked pulps were slightly brighter than the enzyme treated pulps. This might be due to the presence of hydrogen peroxide in the chemical deinking trials.

According to the burst index values, enzyme treatments provided pulps that were as strong as or slightly stronger than the control pulps but weaker than the chemically treated pulp. This might be because sodium hydroxide was present in the chemical deinking mixture and contributed to the higher burst index for this pulp (10). However, the burst indices for enzymes B and E were comparable to the chemically deinked pulp. The freeness values in each enzyme treatment were also higher than the control pulp and chemically treated pulp.

Based on the results from their studies, Jeffries et al. (10) hypothesized that enzymes work by removing cellulosic fines and microfibrils. This increases freeness and reduces the hydrodynamic drag. The enzymes could also work by releasing fibers from the surface of the toner particles.

Factors Influencing Enzymatic Deinking

Zeyer et al. (54,55) studied the enzymatic deinking of paper by using a cellulose fabric as a model in order to determine how enzymes enhance the deinking process and under what conditions enzymes work best when performing this specific task.

Based on their studies, they concluded that:

1. Mechanical action is important to improve enzymatic deinking. They performed experiments by using both AHIBA apparatus which provides a good mixing but very little friction on the fiber surface and LAUNDER-OMETER apparatus which provides much more agitation and fiber surface friction. 20 or 40 beads were also added in LAUNDER-OMETER. Total treatment time for all samples was 3 hours and ink

removal was measured by image analysis. They found that ink removal without enzymes was much lower than with increasing enzyme treatment duration (Figure 25).

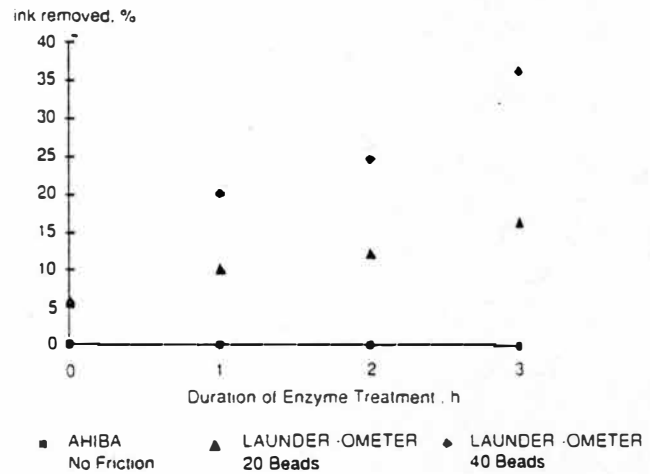


Figure 25. Time Dependence of Deinking Under Different Conditions in AHIBA-MAT and LAUDER-OMETER (54).

Fiber surface friction also plays an important role in the enzymatic deinking since experiments under conditions with very little or no fiber surface friction showed no measurable deinking.

General biochemical considerations suggest that there is an optimum for fiber surface friction because of denaturation of the enzyme due to shear as observed by Reese et al. (54,56).

2. Enzyme treatment and mechanical action have to be performed simultaneously to achieve an effective deinking. This is because the fiber is covered by a layer of ink which prevents the fiber from the enzyme attack. Thus, mechanical action plays a role in allowing the enzyme to attack cellulose chains.

They (54,55) also proposed a model of elastic opening of the contact between the ink and the fiber followed by cutting of the anchoring fibers by enzymes (Figure 26). The mechanical action leads to a distortion of the arrangement of the chains and increases the accessibility of cellulose chains that attach the ink particle to the fiber. At that point, the enzyme is able to cleave the glucosidic bond. This is consistent with the general finding of Fan et al. (57) that only easily accessible cellulose chains can be cleaved by enzymes.

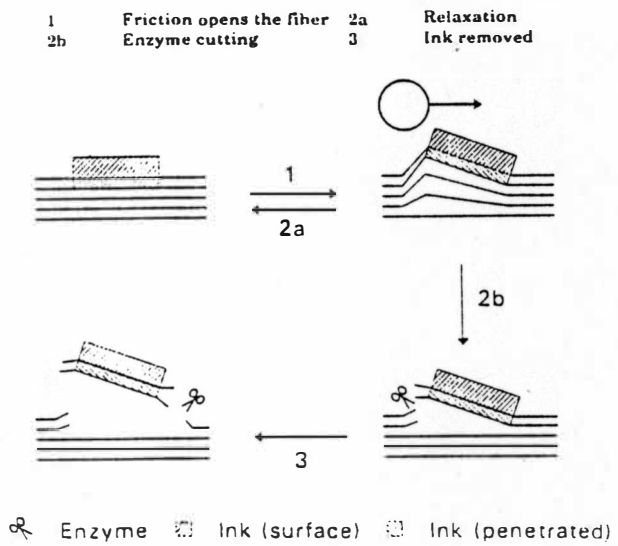


Figure 26. A Model of Enzymatic Deinking (54,55).

Possible Mechanisms of Enzymatic Deinking

Enzymatic approaches (58,59) involve either attacking the ink or fiber surfaces. Esterases and lipases are believed to easily degrade vegetable-oil-based inks.

Hemicellulases, cellulases, pectinases and lignolytic enzymes can modify fiber surfaces or bonds in the vicinity of ink particles in such a way that ink particles are free for removal by washing or flotation.

Welt and Dinus (58) reviewed the enzymatic deinking works and summerized the possible mechanisms of enzymatic deinking as follows:

1. Catalytic hydrolysis may not be necessary since enzymes can remove ink under non optional conditions. Cellulase binding alone may disrupt fiber surfaces enough to release ink during pulping.

2. Enzymes partially hydrolyze or depolymerize cellulose at the fiber surfaces. Fibers are free from one another because of weakened bonds. Ink particles are dislodged when fibers separate during pulping.

3. Enzymes possibly weaken bonds by increasing fibrillation or removing surface layers of individual fibers.

4. Hemicellulases facilitate ink removal by breaking lignin-carbohydrate complexes and releasing lignin from fiber surfaces. Ink particles are then dispersed with lignin.

5. It is also possible that cellulases peel fibrils from fiber surfaces, thereby releasing ink particles for dispersal in suspension.

6. Mechanical action is crucial for enzymatic activity because it distorts cellulose chains at or near fiber surfaces and increases the accessibility of cellulose chains for enzyme attack. However, Putz et al. (60) found that greater shear force during pulping at high consistency did not increase brightness.

7. For non-impact printed paper, enzymes remove fibrous material from ink particles. This increases ink particle hydrophobicity and eases separation during flotatiion.

A particular deinking system would probably involve more than one of these mechanisms. However, the relative importance of each mechanism would depend on fiber substrate, ink composition and enzyme mixture (59).

Until now, most research works have been conducted for conventional inks like offset and letterpress, and non-impact printing inks like xerographic and laser. Only one study has been reported on enzymatic deinking of flexographic water-based inks in which by using cellulases, the brightness from enzyme treated pulp was about 4-5 points higher than the control pulp (58).

Therefore, the objective of this study was to confirm whether such enzymes can be used for deinking of flexographic water-based inks. If so, the next step would be determination of the optimal conditions to reach the high efficiency plateau of this enzymatic deinking.

CHAPTER III

PROBLEM STATEMENT AND OBJECTIVE

Flexographic water-based inks are widely known to be difficult to deink by the flotation process. Unlike oil-based inks, flexographic water-based inks tend to disperse into very small particles during alkaline repulping and thus lower brightness of the deinked pulp. The acrylic resins used as binders in flexographic water-based inks are resolubilized under alkaline repulping. This leads to easy release of ink particles from the fibers and then causes over-dispersion into very fine particles. Also, these fine particles tend to have little hydrophobic characteristics required for the flotation process. The very small particles also have a tendency to redeposit onto the fibers.

However, it has been found that acidic or neutral condition during repulping and flotation process can significantly improve the deinkability of flexographic water-based inks (2-4). Acrylic resins are neutralized and then precipitated to form larger ink particles. These larger ink particles have more chances to collide with air bubbles and then float to the surface of the flotation cell.

Until now, many researchers have shown that enzymatic deinking significantly improves the deinking efficiency of offset, letterpress and non-impact printed paper (5-10). Only one preliminary study has been reported that enzymatic deinking also provides positive results on flexographic water-based inks (58).

In this enzymatic deinking study, pulping under acidic conditions is done to promote cellulase enzyme activity and this condition should enhance deinking of flexographic water-based inks.

The objectives of this study are:

1. To confirm whether or not such enzymes can be used to improve the deinking of flexographic water-based inks.

2. To examine the effects of reaction time, displector concentration and dosage level of enzyme on enzymatic deinking.

3. To find the enzymatic repulping conditions that will lead to an increase in the deinking efficiency.

The criteria used to evaluate the deinking efficiency in this study were brightness, freeness, water retention value (WRV), fiber length, pulp viscosity and yield.

CHAPTER IV

EXPERIMENTAL DESIGN

Design

This enzymatic deinking study was divided into four phases as follows:

First Phase: Preliminary Experiments

The main objective of this phase was to study the effect of each repulping variable on enzymatic deinking of flexographic water-based inks and then establish the conditions for the second phase.

Three variables (repulping time, displector dosage and enzyme dosage), each at two levels (low and high) were used to design various enzymatic repulping conditions. Table 9 indicates the variables and the levels of each variable for the experiments in this phase.

Jeffries et al. (9) studied the enzymatic deinking of xerograhic printed paper and concluded that 30 minutes is the optimum reaction time; however, the enzyme effectively enhances the ink removal at shorter reaction times. The optimum dosage of displector for deinking is 0.3%-0.4% based on oven dry (O.D.) weight of fibers and the optimum dosage of enzymes, Celluclast 1.5 L is between 0.1%-1.0%. Kim et

al. (6) also found that the required amount of enzyme for the best ink removal by flotation is about 0.10%-0.15% at which the maximum brightness gain was obtained.

Table 9

The Variables and Levels of Each Variable (Phase I)

Variable	Low level	High level
Repulping time	15 min.	25 min.
Displector dosage	0 %	0.3%
Enzyme dosage	0.1%	0.3%

Based on these three variables and their two levels, the 2^3 factorial design was used to construct the experiments for this phase as shown in Table 10.

The run number was randomly assigned to each enzymatic repulping condition except the two extreme conditions where all variables were at high or low levels. Two control experiments (repulping time 15 min. and 25 min.) were also performed without adding enzyme and displector, in order to compare and study the effects of enzyme and displector on enzymatic deinking. The experiments were done with two replicates for each enzymatic repulping condition.

The flexographic water-based ink printed newspaper was repulped in the slush-maker at about 6% consistency. At the end of pulping, temperature was raised to 90°C to denature enzyme and then the repulped stock was diluted to about 0.7-0.8% consistency in the dilution tank. The flotation deinking of the diluted stock was

then performed and the floated stock was washed through the sidehill screen. Repulping and deinking of each enzymatic repulping condition was conducted in duplicate in order to obtain the error variance. Figure 27 illustrates the deinking process flow diagram.

Table 10

The 2^3 Factorial Design for Enzymatic Repulping (Phase I)

Treatment combination	Levels of the variables			Randomized run number
	Repulping time	Displector dosage	Enzyme dosage	
1	-	-	-	1
2	+	-	-	6
3	-	+	-	8
4	+	+	-	3
5	-	-	+	5
6	+	-	+	4
7	-	+	+	7
8	+	+	+	2

"+" sign = high level and "- "sign = low level

Pulp was sampled at three different locations (after repulping, floated accept and sidehill screen washing accept) shown as P_1, P_2 and P_3 in Figure 27. Pulp viscosity and water retention values were measured for only repulped stock (P_1). Freeness testing was performed for repulped stock (P_1), floated accept (P_2) and screen washing accept (P_3). Three handsheets were then made from repulped stock (P_1), floated accept (P_2) and screen washing accept (P_3) in order to measure brightness.

Yields after flotation and sidehill screen washing as well as overall yield were also determined.

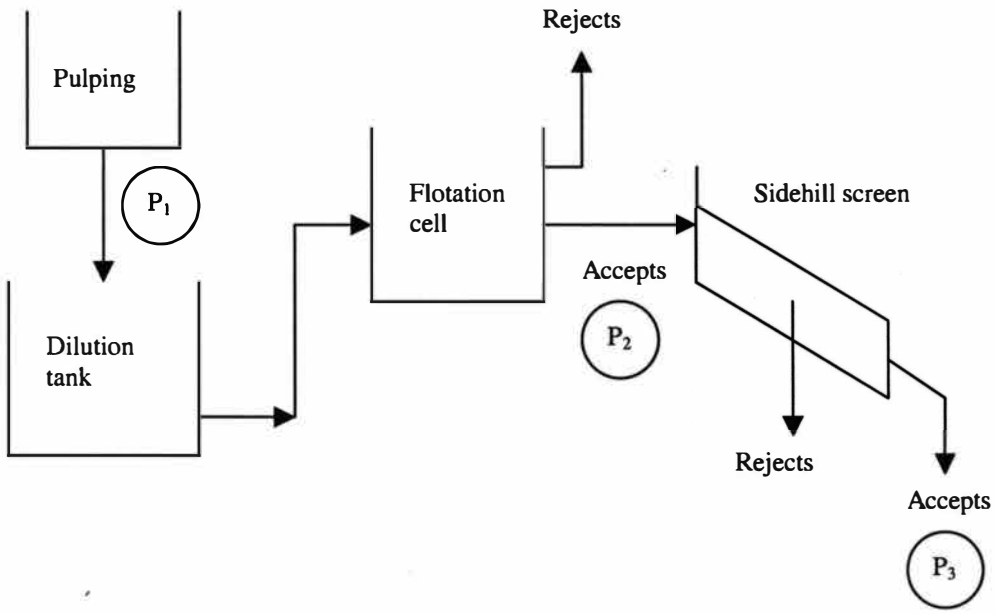


Figure 27. Deinking Process Flow Diagram.

The results from first phase were used to modify the variables and enzymatic repulping conditions in second phase.

Second Phase: Optimal Conditions for Enzymatic Deinking

The objective of this phase was to determine the optimal enzymatic repulping conditions in order to maximize the deinking efficiency. The experiment in this phase was designed and executed based on the results from first phase.

The results from first phase showed the trend that enzyme may need more time to react with the pulp. Consequently, the experimental conditions were set in such a way that after repulping time, enzyme was not denatured right away but was

still left in the slush-maker to react with the repulped stock at different time intervals. Then, at the end of each time interval, enzyme was denatured to stop its reaction by increasing temperature in the slush-maker up to 90°C. The slush-maker was insulated to make sure that there was no heat loss during the reaction time.

The repulping time was fixed at 15 min. and displector dosage was also fixed at 0.3% based on oven dry (O.D.) weight of the pulp. Enzyme dosages were 0.1% and 0.3% based on O.D. weight of the pulp. Four different reaction times after 15 min-repulping time were 0, 30, 60, 90 min. Table 11 illustrates the completely randomized two-factor factorial design for this phase.

Table 11

The Completely Randomized Two-Factor Factorial Design (Phase II)

Treatment combination	Enzyme dosage (%)	Reaction time after 15min-repulping time (min.)	Randomized run number
1	0.1	0	4
2	0.1	30	6
3	0.1	60	2
4	0.1	90	3
5	0.3	0	1
6	0.3	30	8
7	0.3	60	7
8	0.3	90	5

The run number was randomly assigned to each enzymatic repulping reaction. The experiments were done with single replicate for each enzymatic reaction. Repulping, flotation and sidehill screen washing processes for each run were

performed as in first phase. Pulps were sampled and all tests were performed as described in first phase.

Third Phase: The Effect of Heat on Pulp Brightness

The objective of this phase was to investigate the effect of heat on brightness of the repulped stock. Heat was introduced into the repulping process by two different ways. The first one was the heat due to the increase in temperature caused by repulping. The other was the heat from hot steam that was injected at the end of the reaction time to kill the enzyme by increasing the temperature to 90°C.

For this phase, flexographic water-based printed newspaper was pulped at 15 min in the slush-maker without adding any enzyme and displector. After repulping, pulp was kept in the slush-maker for 0, 30, 60, 90, 120 min-time intervals. At the end of each time interval, pulp was sampled and heated up to 90°C by injecting hot steam to imitate the situation of denaturing enzyme after repulping. Then, handsheets were made to measure brightness from these heat-treated pulps. In order to study and compare the effect of heat introduced by hot steam to kill enzyme, handsheets were also made from pulp samples taken after 90 and 120 min-time intervals but without heat treatment.

Fourth Phase: Double Repulping and Washing

The objective of this phase was to study the effects of enzyme on pulp brightness on each stage during two staged repulping and washing.

For this phase, flexographic water-based printed paper was repulped in the slush-maker at 6% consistency with 0.3% displector dosage for 15 min. Enzyme dosage level were 0%, 0.1% and 0.3% respectively. At the end of repulping time, temperature was raised to 90°C to denature enzyme even for the case of "0%" enzyme dosage. The repulped stock was then washed by using a "250 count" pillowcase until the filtrate was clear. The washed pulp was then centrifuged to remove the extra water out.

The pulp was next repulped in the slush-maker again for 15 min. with 0.3% displector dosage and enzyme dosage levels were maintained at 0%, 0.1% and 0.3% respectively. Again, at the end of this second repulping time, temperature was increased to 90°C to kill enzyme. Then, about 3 g O.D. weight of the second repulped stock was randomly taken. By using Britt jar, the pulp was then washed with the combination of distilled water and 0.3% displector until the filtrate was clear.

Handsheets were made to measure brightness from pulp taken after 1st repulping, 1st washing, 2nd repulping and 2nd washing. Figure 28 shows the flow diagram of this experiment. In the figure, P_1 to P_4 indicate the points that pulp was sampled to make handsheets.

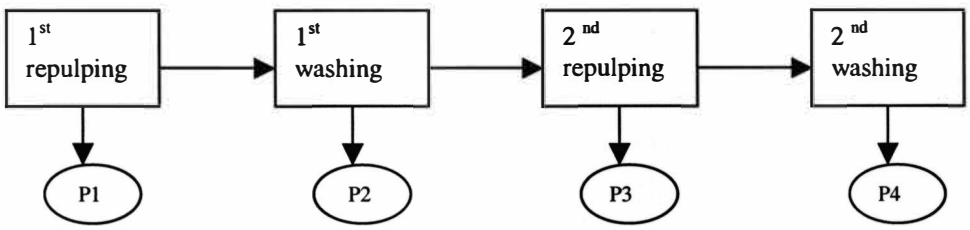


Figure 28. Flow Diagram of Double Pulping and Washing.

Materials

Ink

Black flexographic water-based ink "NEWSAQ-BLACK: GA93-4538" was supplied by Sun Chemical Company. Its composition is shown in Table 12.

Table 12

Composition of the Flexographic Water-Based Ink

Ingredient	Weight (%)
Carbon black	15%
Acrylic resin	15%
Water	70%
Amines	Small amount
Defoamer	Small amount
Derivatives	Small amount

Paper

The newsprint TMP paper was supplied in 17.5" diameter, 23" wide rolls by Department of Paper and Printing Science and Engineering, Western Michigan University. It had a brightness of 59.9 units.

Displector

Displectors are surfactants that combine the action of dispersants and collectors in order to obtain the maximum benefit of the combination of flotation and

washing system. DI-600 which is an alkoxylated fatty acid nonionic deinking surfactant was used as a displector in this study. DI-600 was supplied by Kao Specialties Company (High Point Chemical Corporation)

Enzyme

The enzyme used in this study was Celluclast 1.5L and was supplied by Novo Nordisk Biochem North America, Inc. This enzyme is a liquid cellulase enzyme prepared by a controlled fermentation of the fungus "Trichoderma reesei". This brown liquid enzyme has a density of approximately 1.2g/mL and its activity is 1500 NCU/g. One Novo Cellulase Unit (NCU) is defined as the amount of enzyme which degrades Carboxymethyl cellulose (CMC) to reducing carbohydrates with a reduction power corresponding to 1 μmol glucose per minute. The standard condition for determining the activity of Celluclast 1.5L is given in Appendix A. The optimal working temperature for this enzyme is about 50-60°C and the optimal working pH is 4.5-6.0.

Procedure

Flexographic Printing

The flexographic water-based ink was printed on newsprint paper by using the Zeran "PMR-R&D" flexo press at Western Michigan University. The width of this press is 23 inches. The initial speed was 315 ft/min. and the maximum speed was 600 ft/min. These printed papers were separated into two groups. The first group printed

on the first day had higher ink density than the second group printed on the next day. The amount of ink applied on the first group was about 1.42% of the paper weight whereas the amount of ink applied on the second group was about 1.32%. This ink density was determined by cutting the blank newspaper and printed newspaper into pattern sheets as shown in Figure 29. Each single sheet was then weighed to 0.0001g for the blank newspaper, the first printed group and the second printed group (30 sheets per each group of blank and printed papers). The average weight of single sheet from each group was then determined and the ink density was calculated from the differences of the average weight of single sheet between blank and printed papers.

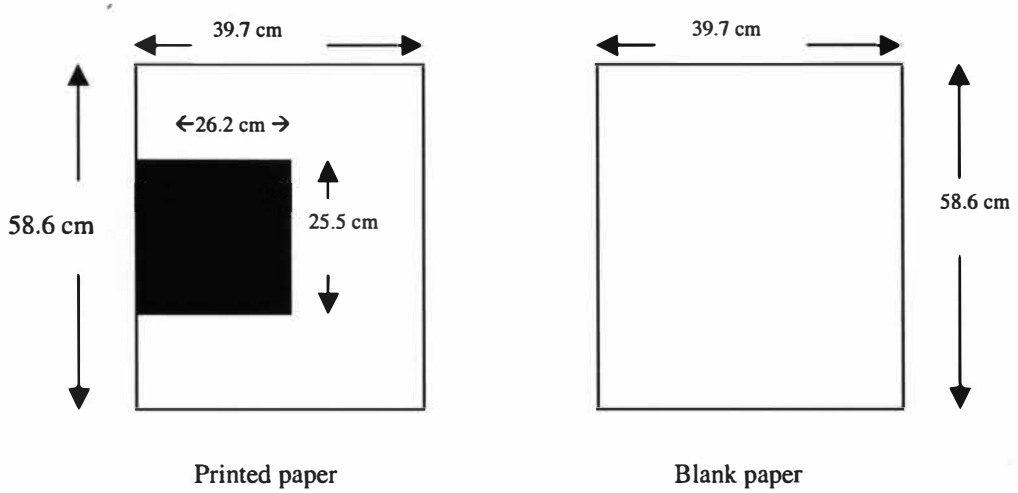


Figure 29. A Pattern Sheet for Both Printed and Blank Papers.

After printing, the printed paper was aged in a conditioned room (relative humidity = 50% ± 2% and temperature = 23°C ± 1°C) for about one month to ensure ink setting. After aging, repulping was performed by using the slush-maker and then

deinking was completed by using pilot plant flotation cell followed by washing through sidehill screen.

Repulping With the Slush-Maker

The amount of ink was controlled by using the same proportion of dark to light printed newspapers during each experimental run. The printed paper was repulped in the Morden Laboratory Slush-Maker at 6% consistency. Before repulping, the printed paper was first torn into approximately 2"x2" pieces and then soaked in soft water for about 1 hour.

During soaking, pH was adjusted to 4.8-5.0 by using sulfuric acid and sodium hydroxide, since in this pH range, the Celluclast 1.5L enzyme provides a high relative activity of 97.5%. At almost the end of the soaking time, temperature inside the slush-maker was adjusted by injecting steam to about 48-50°C for 15 min-repulping time and to about 43-45°C for 25 min-repulping time. The measured amounts of enzyme and displector were then added. Chopsticks were periodically used to mix the added chemicals with the printed paper. Repulping was then started and at the end of repulping time, temperature was increased to 90°C to denature enzyme. Pulp sample was then collected for further study.

Deinking Process

Pulp from the slush-maker was transferred to the dilution tank and diluted with soft water to about 0.7-0.8 % consistency at 38-40°C temperature. Flotation was

then performed by using the pilot plant flotation cell at Western Michigan University. This flotation cell has a 7.7 gallons capacity. The air flow rate was 8 ft^3/min. and the circulation rate of the pump was 15 gallon/min. Flotation time was 10 min. After that, the floated accept was washed through a pilot plant sidehill screen. The screen has a mesh size of 80. The screen length is 38 inches and its angle is 41° from the horizontal. The floated and washed accepts were sampled for further study.

Pulp Evaluation

Pulp collected after repulping (slushed stock) was tested for brightness, freeness, pulp viscosity and water retention value (WRV). Fiber length was also studied, in addition, for the pulp collected after repulping from second phase experiments.

Pulps collected after flotation (floated accept) and after sidehill screen (washed accept) were tested for brightness and freeness only. Overall yield, flotation yield and sidehill screen washing yield was also calculated.

Brightness

3 g O.D. weight of pulp sample was taken and diluted to 0.65% consistency (the average consistency of floated accepts). Three brightness pads were made by using distilled water and pH was adjusted to 5.0± 0.1 (TAPPI Standard, T-218 om-91). The pads were assembled by using a set of metal plates and rings and then dried in a conditioned room (50%± 2% relative humidity and 23°C±1°C temperature).

Brightness pad preparation is explained in more details in Appendix C. The measurement of brightness in this study is referred to in "unit".

Brightness was measured up to 5 readings on the side in contact with the metal plate using the S4-M Brightmeter. Brightness changes by flotation and by washing were also calculated by using equations described in Appendix C.

Freeness

3 g O.D. weight of pulp sample was taken and diluted to 0.3% consistency. Pulp freeness was measured by following TAPPI Standard, T-227 om-94 (see Appendix D for more details). Freeness changes by flotation and by washing were calculated by using equations described in Appendix D.

Water Retention Value (WRV)

Water retention value is a useful tool to evaluate the dewatering behavior of the pulp. It is a measurement of the amount of water retained by the wet pulp after centrifuging under standard conditions (30 min., 21°C ± 3°C temperature and 900g where g is acceleration due to gravity). Water retention value of the repulped stock was determined by following TAPPI useful methods 1991, UM 256. Procedure and its calculations are explained in more detail in Appendix E.

Viscosity

Viscosity is a relative indication of the pulp degradation. Viscosity of the pulp sample was determined after lignin removal (61) by following TAPPI Standard, T230 om-94. Procedures of lignin removal and pulp viscosity determination are described in more detail in Appendix F.

Fiber Length

Arithmetic average fiber length (L_N), length weighted average fiber length (L_L) and weight weighted average fiber length (L_W) of pulp samples were determined according to TAPPI Standard, T271 pm-91 by using a Kajaani FS-100 fiber analyzer (see Appendix G).

Arithmetic average fiber length is calculated based on the number of the measured fibers in different length fractions and from the average length of the fraction. Unlike arithmetic average fiber length, length weighted average fiber length and weight weighted average fiber length are more meaningful indicators of the fiber length determination, since the effect of long fibers is more emphasized in length and weight weighted average fiber length calculations.

Yield

Flotation, sidehill screen washing and overall yields were calculated by using equations described in Appendix H.

CHAPTER V

RESULTS AND DISCUSSION

First Phase: Preliminary Experiments

In this phase, three variables - repulping time, displector dosage and enzyme dosage - were used to design various enzymatic repulping conditions. Each variable was used at two different levels viz. low and high. For the low level, repulping time was 15 minutes, displector dosage was 0% and enzyme dosage was 0.1% based on oven dry (O.D.) weight of pulp. For high level, repulping time was 25 minutes and displector and enzyme dosages were 0.3% based on O.D. weight of pulp. Printed paper using flexographic water-based ink was pulped under acidic conditions (pH 4.8-5.0) which should help in preventing over-dispersion of flexographic water-based ink particles. Also, at this pH, the enzyme exhibits the highest activity.

The P-values and the signs of each effect from statistical data analysis of this phase experiments are summarized in Table 13. The full ANOVA table for each pulp evaluation is presented in the Appendix L. In Table 13, A stands for repulping time, B stands for displector dosage and C stands for enzyme dosage. A*B, A*C and B*C stands for 2-way interaction effects for corresponding variable whereas A*B*C stands for 3-way interaction effect for all these three variables.

The individual P-value from each effect was compared to significance level (α) of 0.100. If P-value was smaller than or equal to 0.100, then the null hypothesis (H_o) of that effect being equal to zero could be rejected.

Table 13

The P-Value and the Sign of Each Effect for Each Pulp Evaluation

Pulp evaluation	A	B	C	A*B	A*C	B*C	A*B*C
Brightness after repulping	0.090 -	0.000 +	0.044 +	0.008 +	0.014 +	0.000 -	0.541 +
Brightness after flotation	0.133 -	0.212 +	0.311 +	0.041 +	0.049 +	0.042 -	0.371 -
Brightness after SH screen washing	0.094 -	0.678 +	0.188 +	0.018 +	0.012 +	0.024 -	0.909 +
Brightness change by flotation	0.793 -	0.062 -	0.524 -	0.889 -	0.978 +	0.390 +	0.192 -
Brightness change by washing	0.651 +	0.216 -	0.909 -	0.613 -	0.891 -	0.541 +	0.229 +
Freeness after repulping	0.036 -	0.984 +	0.616 +	0.487 -	0.702 -	0.255 +	0.863 +
Freeness after flotation	0.102 -	0.329 +	0.429 +	0.393 -	0.454 -	0.262 +	0.749 -
Freeness after SH screen washing	0.082 -	0.278 +	0.232 +	0.662 -	0.984 -	0.256 +	0.893 +
Freeness change by flotation	0.537 +	0.157 +	0.592 +	0.716 -	0.527 -	0.877 +	0.472 -
Freeness change by washing	0.774 -	0.812 +	0.455 +	0.509 +	0.260 +	0.941 +	0.474 +

Table 13—Continued

Pulp evaluation	A	B	C	A*B	A*C	B*C	A*B*C
Water retention value (WRV)	0.317 +	0.820 -	0.632 -	0.994 +	0.864 -	0.880 +	0.385 -
Pulp viscosity	0.592 -	0.022 -	0.495 +	0.245 +	0.064 +	0.101 +	0.018 +
Flotation yield	0.072 +	0.032 +	0.056 -	0.542 +	0.568 -	0.120 +	0.096 +
SH washing yield	0.105 -	0.841 -	0.516 +	0.075 -	0.181 -	0.300 +	0.473 -
Overall yield	0.271 -	0.540 +	0.961 -	0.099 -	0.131 -	0.130 +	0.886 -

A = repulping time main effect
B = displector dosage main effect
C = enzyme dosage main effect
A*B = 2-way interaction effect of repulping time and displector dosage
A*C = 2-way interaction effect of repulping time and enzyme dosage
B*C = 2-way interaction effect of displector dosage and enzyme dosage
A*B*C = 3-way interaction effect of all these three variables
SH = sidehill
"-" sign = negative effect
"+" sign = positive effect.

Brightness After Repulping (Brightness of Slushed Pulp)

Effect of Repulping Time

Generally longer repulping time should reduce pulp brightness since more ink particles are dislodged when fibers separate during repulping. The results shown in Table 13 indicates that the repulping time main effect is statistically significant since

its P-value = 0.090. Also, its main effect is negative which means that increasing repulping time decreases pulp brightness. The experimental data shown in Table 14 also verifies the same (comparisons of control 15 minutes to control 25 minutes, run no.1 to run no.6, run no. 3 to run no. 8 and run no. 4 to run no. 5 with the exception of run no. 2 and run no. 7).

Table 14

Brightness After Repulping

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Brightness after repulping (unit)
0	control (15 min.)			45.6
00	control (25 min.)			44.5
1	-	-	-	50.4
2	+	+	+	53.8
3	+	+	-	52.8
4	+	-	+	51.2
5	-	-	+	52.9
6	+	-	-	45.8
7	-	+	+	50.7
8	-	+	-	54.1

"+" sign = high level and "- " sign = low level

Effect of Displector

The results from Table 14 show that brightness is increased with the use of displector (see comparisons of run no. 1 to run no.8, run no. 2 to run no.4, run no. 3 to run no. 6 with the exception of run no. 5 to run no. 7). The results from Table 13 also illustrate that the displector main effect is statistically significant since its P-value is

0.000. Also, the main effect is positive which indicates that the brightness at repulping increases with the use of displector. The role of the displector in helping brightness at repulping is not known and needs to be investigated in future. However, it might be possible that during the handsheet making which is similar to washing process the displector acted as a dispersant and aided in removing ink particles.

Effect of Enzyme

As shown in the Table 14, when the control experiment at 15 minutes was compared to run no. 1 and run no. 5 as well as when control experiment at 25 minutes was compared to run no. 4 and run no.6, it can be seen that by adding enzyme only (no displector), an increase in brightness can be observed. This is in agreement with the enzymatic deinking of flexographic water-based ink work of Prasad (58).

The role of enzyme in improving deinking has been studied by many researchers and many mechanisms have been proposed (54-60). Most of them suggest that hemicellulase and cellulase enzymes can modify fiber surfaces or bonds in the vicinity of the ink particles in such a way that ink particles are free for removal by washing or flotation (58,59). For the case of flexographic water-based inks in this study, cellulase enzyme which has about 20% hemicellulases in the composition seems to not only attack celluloses but dissolve hemicellose and fine contents due to easy accessibility of the latter. Moreover, it might be possible that this cellulase enzyme modifies the fiber surface and makes it less active in such a way that re-deposition of ink particles reduces. In addition to that, repulping at acidic pH should

prevent the ink particles to over disperse to fine particles which also diminishes the re-deposition of ink particles.

The effect of enzyme dosage seems to be unclear although the statistical analysis (Table 13) shows that the enzyme main effect is statistically significant. The effect is also positive that means higher enzyme dosage should result in higher brightness. The experimental results for the run no. 1 and run no. 5, run no. 2 and run no. 3, run no. 4 and run no. 6 agree with the hypothesis. The only exception is the run no. 7 and run no. 8 wherein brightness after repulping is improved by 3.4 units by using lower enzyme dosage. Therefore, it can be concluded that the enzyme helps in increasing brightness but the role of enzyme dosage needs to be studied more. Also, the time required for enzyme reaction should be examined more.

The results from Table 13 also indicate that all three of 2-way interaction effects are statistically significant. P-value of repulping time and displector interaction effect is 0.008. P-value for repulping time and enzyme interaction effect is 0.014 and P-value for displector and enzyme interaction effect is 0.000. However, the effects of these 2-way interactions are confounding. The effect of repulping time and displector interaction is positive. The effect of repulping time and enzyme interaction is also positive but the effect of displector and enzyme is negative. This leads to the conclusion that when 2-way interaction effects were examined, the dosage levels of enzyme and displector should be low whereas repulping time should be high to achieve the higher brightness at repulping.

Brightness After Flotation (Brightness of Floated Accept)

Effect of Repulping Time

The effect of repulping time on brightness after flotation should be the same as in the case of brightness after repulping since longer repulping time dislodges more ink particles to the system. However, the P-values from Table 13 exhibit that the repulping time main effect is not statistically significant (P-value = 0.133). Its effect is negative which means that longer repulping time decreases pulp brightness. Also, the majority of experimental data tabulated in Table 15 shows this trend especially in the case of run no.1 compared to run no. 6.

Table 15

Brightness After Flotation

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Brightness after flotation (unit)
0	control (15 min.)			44.6
01	control (25 min.)			44.6
1	-	-	-	51.4
2	+	+	+	51.3
3	+	+	-	51.3
4	+	-	+	51.0
5	-	-	+	51.6
6	+	-	-	45.6
7	-	+	+	49.6
8	-	+	-	51.7

"+" sign = high level and "-" sign = low level

Effect of Displector

Displector with the chemistry being a combination of dispersant (washing aid) and collector (flotation aid) should help increase the brightness after flotation. This may be because ink particles are not over-dispersed due to acidic repulping (pH= 4.8 -5.0) and displector should make ink particles hydrophobic and then agglomerate them into larger particles to be removed by flotation.

The P-value from Table 13 indicates that its main effect is not statistically significant (P-value = 0.212); the experimental data in Table 15 shows some positive trend (see comparison of run no. 1 to run no. 8, run no. 2 to run no.4, run no.3 to run no. 6 except run no. 5 to run no.7). This is also indicated by the sign in Table 13.

Effect of Enzyme

As discussed earlier in brightness after repulping that enzyme helps in increasing the brightness after repulping, it can be also seen that enzyme also improves brightness after flotation as well (see Table 15, comparison between control experiment at 15 minutes to run no.1 and run no.5 and comparison of control at 25 minutes to run no. 4 and run no. 6). The P-value results (Table 13); however, indicate that the enzymes main effect is not statistically significant (P-value = 0.311).

It is likely that the effect of enzyme dosage is inconclusive here. This may be because 15 minutes or 25 minutes was too short to differentiate the effect of different enzyme dosage. Reaction time of enzyme needed is to be studied more (see second phase).

As in the case of brightness at repulping, the rest of the results from Table 13 for brightness after flotation show that all three 2-way interaction effects are statistically significant. P-value of repulping time and displector interaction effect is 0.041. P-value of repulping time and enzyme interaction effect is 0.049 and P-value of displector and enzyme interaction effect is 0.042. This was similar to the case of brightness after repulping.

Brightness After Sidehill Screen Washing (Brightness of Washed Accept)

Effect of Repulping Time

As expected, longer repulping time is detrimental to brightness after side hill screen washing because it produces more ink particles to the pulp system during repulping. The experimental data in Table 16 also shows this trend (comparison of control 15 minutes to control 25 minutes, run no. 1 to run no. 6, run no. 3 to run no. 8, run no. 4 to run no. 5 with the exception of run no. 2 to run no. 7).

The results from Table 13 also agree with this since the repulping time main effect has P-value of 0.094 which is lower than 0.100. Also, the sign of this main effect is negative which means that longer repulping time reduces pulp brightness of washed accept.

Effect of Displector

Displector as a combination of dispersant and collector should help ink removal by washing process as well. Even though the results from statistical analysis

(Table 13) shows that its effect is not statistically significant (P-value = 0.678), the sign of its effect is positive which implies that the displector had a tendency to improve brightness. The experimental data shown in Table 16 also demonstrates this trend (run no. 1 compared to run no.8, run no. 2 compared to run no.4, run no. 3 compared to run no. 6).

Table 16

Brightness After Sidehill Screen Washing

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Brightness after sidehill screen washing (unit)
0	control (15 min.)			52.0
00	control (25 min.)			50.2
1	-	-	-	56.2
2	+	+	+	56.2
3	+	+	-	55.1
4	+	-	+	55.8
5	-	-	+	56.8
6	+	-	-	51.9
7	-	+	+	53.8
8	-	+	-	56.4

"+" sign = high level and "- " sign = low level

Effect of Enzyme

As can be seen from Table 16, the comparison of control at 15 minutes (without enzyme) to run no. 1 and run no. 5 (with enzyme dosage of 0.1% and 0.3% respectively) shows that the enzyme helps in increasing the brightness of washed accept. The comparison of control experiment at 25 minutes to the run no. 4 and run

no. 6 also indicates the same trend. However, the P-value results from Table 13 indicate that its main effect is not statistically significant since its P-value is 0.188 which is higher than 0.100. This may imply that the effect of enzyme dosage is inconclusive like in the previous cases of brightness after repulping and brightness after flotation. The data from Table 16 shows this inconclusive result since in one case where other variables were kept constant higher enzyme dosage provided higher brightness (see run no. 4 compared to run no. 6). However, in other case even though other variables were kept constant, lower enzyme dosage gave higher brightness (run no. 7 compared to run no. 8).

Thus, it seems that the enzyme reacted with the pulp slowly. That is why with 15 minute and 25 minute repulping time, the difference in enzyme dosage did not show any significant effect. The effect of enzyme dosage at longer reaction time was hence studied and discussed in detail in the second phase.

From Table 13, it can be seen that all the three 2-way interaction effects are statistically significant since their P-values are lower than 0.100 (for repulping time and displector interaction effect = 0.018, for repulping time and enzyme interaction effect = 0.012 and for displector and enzyme interaction effect = 0.024). Also the signs of all these 2-way interaction effects are just like the brightness after repulping and brightness after flotation.

Brightness Change by Flotation

This study was done to ascertain the brightness change by flotation and see how repulping and flotation stages affected the pulp brightness. Brightness change by flotation was defined as the difference between brightness after flotation and brightness after repulping (brightness change by flotation = brightness after flotation - brightness after repulping). The majority of the results from the Table 17 indicate that there is a loss of brightness due to flotation. This may imply that there might be fiber fractionation going on even though the yield loss is only about 1% - 2 % (see the discussion on flotation yield later).

Table 17

Brightness Change by Flotation

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Brightness change by flotation (unit)
0	control (15 min)			-1.0
00	control (25 min)			+0.1
1	-	-	-	+1.0
2	+	+	+	-2.5
3	+	+	-	-1.5
4	+	-	+	-0.2
5	-	-	+	-1.3
6	+	-	-	-0.2
7	-	+	+	-1.1
8	-	+	-	-2.4

"+" sign = high level and "-" sign = low level

The P-values from the Table 13 suggest that only displector main effect is statistically significant (P-value = 0.062). The sign of the effect is negative, which means that the brightness loss by flotation may increase if the displector is used. The results from the Table 17 also confirm this trend (see the comparison of run no. 1 to run no. 8, run no. 2 to run no. 4, run no. 3 to run no. 6 with the exception of run no. 5 and run no. 7).

Brightness Change by Sidehill Screen Washing

The study of brightness change by washing was done to examine the effect of each stage (washing and flotation) on the pulp brightness.

Table 18

Brightness Change by Sidehill Screen Washing

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Brightness change by washing (unit)
0	control (15 min.)			7.4
00	control (25 min.)			5.6
1	-	-	-	4.8
2	+	+	+	4.9
3	+	+	-	3.8
4	+	-	+	4.8
5	-	-	+	5.2
6	+	-	-	6.3
7	-	+	+	4.2
8	-	+	-	4.6

"+" sign = high level and "-" sign = low level

Brightness change by sidehill screen washing was defined as the difference between brightness after sidehill screen washing and brightness after flotation (brightness change by sidehill screen washing = brightness after sidehill screen washing - brightness after flotation). The results from Table 18 indicate that there is a brightness gain in all cases. This reveals that washing is still a better way to remove flexographic water-based ink particles

The P-values from Table 13 also show that all main effects, all 2-way interaction effect and 3-way interaction effect are not statistically significant.

Freeness After Repulping (Freeness of Slushed Pulp)

Effect of Repulping Time

Freeness generally decreases with increasing repulping time as more fines are generated during prolonged repulping. The results are shown in Table 19. These results are in agreement with the expectation, since freeness of control experiment at 25 minutes is lower than freeness of control experiment at 15 minutes. Similar trend was observed for run no.1 and run no.6, run no. 2 and run no. 7, run no. 3 and run no. 8 as well as run no. 4 and run no.5. In all the mentioned cases, the former run freeness is lower than the latter run. The P-values in Table 13 indicate that only the repulping time is statistically significant with the P-value of 0.036.

Table 19

Freeness After Repulping

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Freeness after repulping (mL)
0	control (15 min.)			115
00	control (25 min.)			114
1	-	-	-	126
2	+	+	+	110
3	+	+	-	98
4	+	-	+	104
5	-	-	+	124
6	+	-	-	115
7	-	+	+	140
8	-	+	-	123

"+" sign = high level and "- "sign = low level

Effect of Displector

Displector works both as a dispersant as well as a collector and should not have any significant effect on the freeness after repulping. This is confirmed by the P-values from Table 13.

Effect of Enzyme

Cellulase enzyme which contains 20% of hemicellulase in the formula generally increases freeness of the pulp as it dissolves fines and hemicelulloses. However, the results from the Table 19 are inconclusive. The comparison between the control at 15 minute repulping time (115 mL) with run no. 1 (126 mL) and run no. 5 (124 mL) indicates that using enzymes increases freeness of the pulp. However, when

the repulping time was increased to 25 minutes the freeness values were 114 mL for control, 115 mL for run no. 6 and 104 mL for run no. 4, which are not very different from each other. Also, the results from Table 13 indicate that the main effect of enzyme is not statistically significant since its P-values (0.616) is much larger than 0.100. This may be due to the slow reaction time. The effect of lower and higher enzyme dosage is not very clear as well. Therefore, the experiments were carried out in the second phase with higher enzyme reaction time to ascertain their effect.

Freeness After Flotation (Freeness of Floated Accept)

Effect of Repulping Time

The duration of the repulping time should not effect the freeness at the flotation. Even though higher repulping time generates more fines and smaller fiber fraction, it does not lead to the flotation of fines with the overflow in the flotation cell. The particle size as well as size of the air bubbles has to be optimum for efficient removal in the flotation cell. It has been suggested that the air bubble / particle size ratio of 5:1 is suitable for effective removal (28,29). It seems that in this particular case, the longer fibers are carried over with the bubbles and the accepts are richer in smaller fiber fraction as compared to the feed. This is suggested by the experimental data as shown in the following table for freeness after flotation (Table 20).

It was found that in most of the cases such as run no.1 and run no. 6, run no. 2 and run no. 7, run no. 4 and run no. 5 as well as run no. 3 and run no. 8, higher repulping time (25 minutes) results in lower freeness values in the accepts than in the

case where repulping time was 15 minutes. The only exception was observed in case of the control experiments where higher repulping time results in higher freeness value of the accepts. The results from Table 13 also indicate the same as the P-value of the repulping time main effect is 0.102 (very close to 0.100) and the sign of the effect is negative.

Table 20

Freeness After Flotation

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Freeness after flotation (mL)
0	control (15 min.)			72
00	control (25 min.)			79
1	-	-	-	86
2	+	+	+	83
3	+	+	-	75
4	+	-	+	74
5	-	-	+	87
6	+	-	-	82
7	-	+	+	120
8	-	+	-	90

"+" sign = high level and "-" sign = low level

Effect of Displector

Displector is a surfactant that is a combination of dispersant and collector. Therefore, it has properties of both. Dispersant provides particles with hydrophilic character and impedes flocculation, which enhances the removal of particles by washing. The function of the collector is to agglomerate particles into larger particles,

which then attach to the air bubbles and then float to the surface. Therefore, the longer fibers might be carried with the bubbles and fractionation might occur. The effect of the displector as shown in the Table 20 is hence not specific. The P-value (0.329) in Table 13 also confirms the same.

However, the flotation yield is not significantly less since the yields for the cases where a displector was used vary from 98.7% to 99.4% whereas the flotation yields for the cases with no displector range between 98.0% and 99.4%. This suggests that there was no significant yield loss and only a small fraction of longer fibers were floated to the top as a floated reject.

Effect of Enzyme

Enzymes should help in increasing the freeness of the recycled pulp if allowed to act for sufficient time. However, it may not have any significant effect on the freeness of the floated accept. This may be because enzyme may not be able to alter the fiber length appreciably to enhance flotation of the pulp by the air bubbles. The experimental data from this phase study is also inconclusive. The data from Table 13 shows that its main effect is not statistically significant (P-value = 0.429). This is also true for the 2-way as well as 3-way interaction effects.

Freeness After Sidehill Screen Washing (Freeness of Washed Accept)

Effect of Repulping Time

Longer repulping time should increase the fines and reduce the freeness values. Fines generally pass through the sidehill screen but some of them also get entrapped in the fiber mat after a while. This can cause some change in the freeness values. This is also seen in the Table 21. A comparison of the results between control experiments for 15 minutes and 25 minutes, run no.1 and run no. 6, run no. 2 and run no. 7, run no. 3 and run no. 8 as well as run no. 4 and run no. 5 indicates that higher repulping time has lower sidehill screen freeness values.

Table 21

Freeness After Sidehill Screen Washing

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Freeness after sidehill screen washing (mL)
0	control (15 min.)			201
00	control (25 min.)			187
1	-	-	-	208
2	+	+	+	213
3	+	+	-	188
4	+	-	+	194
5	-	-	+	210
6	+	-	-	194
7	-	+	+	236
8	-	+	-	213

"+" sign = high level and "-" sign = low level

The P-value results in Table 13 also demonstrate that the main effect of repulping time is statistically significant (P-value = 0.082). The sign of its effect is negative suggesting that shorter repulping time gives higher freeness of washed accept pulp.

Effect of Displector

The results from the Table 13 indicate that the displector main effect is not statistically significant (P-value = 0.278 which is higher than 0.100). Therefore, displector should not affect the fine or fiber contents of the washed accept that much. This is also confirmed by the sidehill washing yield, which varies from 86.0%-86.6% for no displector usage and varies between 85.1% to 88.0% for displector usage. So the difference in yield at washing stage of these two cases (with and without displector dosage) is not significant. That is why the effect of displector in freeness after sidehill screen washing could not be seen.

Effect of Enzyme

Although the P-values from Table 13 do not illustrate that the main effect of enzyme is statistically significant (P-value = 0.232), there is a clear tendency that enzyme helps in increasing the freeness of the washed pulp. The comparison of control (15 minutes and 25 minutes) with the cases where enzyme was used confirms that freeness increases with the enzyme use (Table 21). Higher dosage of enzyme also

shows a tendency of increasing freeness of washed accept as compared to lower dosage (see comparisons of run no. 2 and run 3, run no.7 and run no. 8).

Freeness Change by Flotation

Freeness change by flotation was determined by the difference between freeness after flotation and freeness after repulping. As indicated in the Table 22, there is a freeness loss in all cases. This suggests that there was fiber fractionation occurring to some extent during flotation process. Longer fibers were floated out with the air bubbles as rejects. The resulting freeness of the floated accepts was much lower than the freeness of the slushed pulp. The results are in agreement with the work of Walaipachara (62).

Table 22

Freeness Change by Flotation

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Freeness change by flotation (mL)
0	control (15 min)			-43
00	control (25 min)			-35
1	-	-	-	-40
2	+	+	+	-27
3	+	+	-	-23
4	+	-	+	-30
5	-	-	+	-37
6	+	-	-	-33
7	-	+	+	-20
8	-	+	-	-33

"+" sign = high level and "- "sign = low level

The summerized P-values as shown in Table 13 reveal that all the main effects, 2-way as well as 3-way interaction effects are not statistically significant, since all P-values are higher than 0.100. However, the role of repulping time in freeness loss can be due to the fact that longer repulping time minimizes the freeness loss by flotation since longer repulping time reduces the freeness after repulping (freeness change by flotation = freeness after flotation - freeness after repulping). This can be seen from the experimental data in Table 22 and subsequent comparisons between control experiments at 15 minutes and 25 minutes, run no. 1 and run no. 6, run no. 3 and run no. 8, run no. 4 and run no. 5 except run no.2 and run no.7.

Even though the displector main effect is not statistically significant, it might increase the freeness loss by flotation as it might aid in fractionation of longer fibers by flotation and cause lower freeness after flotation (freeness of floated accept). However, the experimental results indicate that 0.3% displector dosage did not affect the freeness at flotation at all.

Enzyme should increase the freeness loss by flotation because it increases the freeness of the pulp after repulping. The role of the enzymes on freeness of the pulp after flotation is inconclusive. As indicated earlier, the role of enzymes on freeness after repulping here is not as expected since enzyme seems to require longer reaction time with the pulp. So, this makes the role of the enzyme on the freeness change by flotation in this experiment inconclusive as well.

Freeness Change by Sidehill Screen Washing

Freeness change by washing was determined by the difference between freeness after sidehill screen washing and freeness after flotation (freeness change by washing = freeness after sidehill washing - freeness after flotation). As can be seen from Table 23, freeness change by washing is positive for all the cases. This is because the freeness after screen washing is higher than after the flotation since most of the fines have been removed through screening and this increases the pulp freeness. The results from Table 13 indicate that there are no effects (all main effects viz. 2-way and 3-way interaction effects) that are statistically significant since all P-values are higher than 0.100.

Table 23

Freeness Change by Sidhill Screen Washing

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Freeness change by washing (mL)
0	control (15 min.)			129
00	control (25 min.)			108
1	-	-	-	122
2	+	+	+	130
3	+	+	-	113
4	+	-	+	120
5	-	-	+	123
6	+	-	-	112
7	-	+	+	116
8	-	+	-	123

"+" sign = high level and "- "sign = low level

The role of repulping time here should be to decrease the freeness gain by washing since longer repulping time lowers the freeness at the sidehill screen washing due to the fact that more fine contents are produced. The majority of the experimental results in Table 23 displays this trend (comparison of control 15 minutes and control 25 minutes, run no. 1 and run no. 6, run no. 3 and run no. 8, run no. 4 and run no. 5 except run no. 2 and run no. 7). Since, displector did not affect both freeness after flotation and after sidehill screen washing, it should not effect the freeness gain by washing as well.

It was found earlier that the enzyme has a tendency to help increase freeness after sidehill screen washing since it attacks hemicelluloses and fines. It was also reported earlier that effect of enzymes on freeness after flotation is inconclusive. So, enzyme should help increase the freeness gain by washing. This tendency is more pronounced in the case of longer repulping time since enzymes has longer time to react with the pulp as shown in Table 23 (see control experiment at 25 minutes compared to run no. 4 and run no. 6, run no. 2 to run no. 3, run no. 4 to run no. 6, run no. 1 to run no. 5 as well as run no.7 to run no. 8).

Water Retention Value

Fines fraction and hemicellulose content influence water retention value. Generally, this value increases with the increase in the fines fraction (due to higher surface area) and decreases with decrease in hemicellulose content (reduction in the swelling ability of fibers).

The P-values from Table 13 indicate that all main effects, 2-way interaction and 3-way interaction effects are not statistically significant since all P-values are much higher than 0.100. Anyway, the effect of each variable could be explained as follows:

Effect of Repulping Time

Water retention value is expected to increase with an increase in repulping time due to generation of more fines. This is also confirmed by the experimental results as shown in Table 24.

Table 24

Water Retention Value (WRV)

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Water retention value (WRV) (g/g)
0	control (15 min.)			1.252
00	control (25 min.)			1.266
1	-	-	-	1.516
2	+	+	+	1.411
3	+	+	-	1.668
4	+	-	+	1.588
5	-	-	+	1.266
6	+	-	-	1.572
7	-	+	+	1.414
8	-	+	-	1.280

"+" sign = high level and "-" sign = low level

A comparison between the two controls, between run no. 1 and run no. 6, between run no.3 and run no. 8 and between run no. 4 and run no. 5 suggests and

confirms the hypothesis that increasing the repulping time does increase the water retention value. The exception is a comparison between the run no.2 and run no. 7 where the difference in the water retention values is marginal and can be attributed to the experimental error.

Effect of Displector

The experiments were undertaken to analyze the effect of displector on the water retention values. Generally, the role of displector in water retention values should be to disperse fines and hence more fines should pass through the screen during WRV determination. This may cause lower retention values since lesser fines are left behind in the pulp system.

However, the results here are inconclusive. Some experiments in Table 24 such as run no. 1 and run no. 8 as well as run no. 2 and run no. 4 indicate that using displector results in lower water retention values. However, this trend is negated in case of run no. 3 compared to run no. 6 as well as run no. 5 compared to run no. 7. Therefore, the role of the displector in the water retention values cannot be ascertained decisively. This may be due to sampling or experimental errors.

Effect of the Enzyme

An effort was made to analyze the effect of the enzyme treatment of the pulp on its water retention values. A comparison between the control and the enzyme treatment indicates that the water retention values are higher for the enzyme treated

pulp for both 15 minute and 25 minute treatment time (see Table 24). This is contradictory to the expected results, as this cellulase enzyme should attack fines (higher surface area) which should subsequently lower the water retention values. The plausible explanation for this observation could be attributed to the inadequate reaction time for enzyme (15 minutes and 25 minutes). Therefore, it is suggested to increase the time to analyze the effect of the enzyme treatment on the water retention values (discussed later in the second phase).

Pulp Viscosity

Pulp viscosity gives an indication of the degree of polymerization of the pulp cellulose. Higher the viscosity, higher is the degree of polymerization. Lower pulp viscosity indicates cellulose degradation happened during the process.

Effect of Repulping Time

It is expected that repulping time should not affect pulp viscosity but if it does, it might be because of the acidic hydrolysis in this case since repulping was done at pH 4.8-5.0. Pulp viscosity then might be reduced with the increase in the repulping time due to enhanced cellulose degradation (acidic hydrolysis). The hydrolysis breaks the glucosidic linkages in the cellulose chains that leads to lower cellulose molecular weight. This is confirmed by the experimental results showed in Table 25 when run no. 1 is compared to run no. 6, run no. 3 to run no. 8, run no. 4 and run no. 5 as well as the control cases. The only exception was observed for run no. 2

and run no. 7, which could be due to other factors affecting the viscosity. Therefore, It can be seen that longer repulping time reduces pulp viscosity

Table 25

Pulp Viscosity

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Viscosity (cP)
0	control (15 min.)			15.3
00	control (25 min.)			14.6
1	-	-	-	15.1
2	+	+	+	15.2
3	+	+	-	12.0
4	+	-	+	13.8
5	-	-	+	14.9
6	+	-	-	14.6
7	-	+	+	12.8
8	-	+	-	13.8

"+" sign = high level and "- "sign = low level

Effect of Displector

It was thought that the displector should not have any significant effect on the pulp viscosity. Experiments were carried out where no displector was used and also with 0.3% displector dosage. It can be observed from Table 25 that for the run no. 1 and run no. 8, run no. 3 and run no.6 as well as run no. 5 and run no. 7, viscosity decreases to some extent at 0.3% displector dosage as compared to no displector use. However, for the run no. 2 and run no. 4 the opposite trend was observed, wherein viscosity increases at higher displector dosage.

Effect of Enzyme

The enzymatic treatment results in the decrease in the pulp viscosity. As indicated in Table 25, in case of experiments where repulping time was 15 minutes, the viscosity was observed to be 15.3 cP for the control case. The resultant viscosity for all the cases viz. 0.1% and 0.3% enzymatic dosage is lower than the viscosity from the control. This trend can also be observed in the all the cases where the repulping time was increased to 25 minutes with the exception of run no 2. This could be attributed to sampling or experimental error.

A comparison between different enzyme dosages (0.1% and 0.3%) on the pulp viscosity suggests that the higher enzyme dosage reduces pulp viscosity as shown between run no. 1 and run no. 5, run no. 4 and run no.6 as well as run no. 7 and run no.8. The exception to this trend was observed in case of run no. 2 and run no. 3. It seems that the enzyme not only attacks fines and hemicellulose (due to easy access) but also cellulose.

Flotation Yield

The experimental results for the flotation yield did not show any significant difference. The lowest value is 98.0% for control experiment (15 min- repulping time) and the highest value is 99.4% for run no. 2 and run no.6 as shown in Table 26.

The P-values from Table 13 on the other hand indicates that all three variables viz. repulping time, displector dosage and enzyme dosage are significant statistically, since their P-values are lower than 0.100 (0.072 for repulping time, 0.032 for

displector dosage and 0.056 for enzyme dosage). Also, 3-way interaction effect is statistically significant since its P-value is 0.096. The effect of repulping time and displector dosage is positive as shown in Table 13. It seems that the higher repulping time and the use of displector aids in flotation yield. This is in contrast to the expectation, as it should indirectly decrease flotation yield.

It is not because longer repulping time creates more fines or short fibers since not all fines and short fibers need to be floated out as rejects. The particle size of floated particles needs to be in the optimal range to be effectively removed by flotation process as discussed earlier in freeness after flotation.

Table 26

Flotation Yield

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Flotation yield (%)
0	control (15 min.)			98.0
00	control (25 min.)			98.2
1	-	-	-	98.7
2	+	+	+	99.4
3	+	+	-	99.3
4	+	-	+	98.2
5	-	-	+	98.4
6	+	-	-	99.4
7	-	+	+	98.7
8	-	+	-	99.1

"+" sign = high level and "- "sign = low level

The reason that longer repulping time might reduce flotation yield might be that pulp is subjected to acid hydrolysis for longer time and this may lead to lower

yield of the slushed pulp as well as the floated accept. However, longer repulping time gives more time for the enzymatic reaction to occur. This might negatively affect the flotation yield as well. The higher enzyme dosage as well as longer repulping time is detrimental to flotation yield.

The use of displector should reduce the flotation yield as it helps in fiber flotation and elutriation to the top of the column. However, the experimental data indicate that the effect of all these variables is very small because the yield is relatively constant with subtle variations. Even 25 minutes of repulping time seems safe with little flotation yield loss.

Sidehill Screen Washing Yield

The experimental results indicate a pretty similar trend as was observed in case of flotation yield. There is marginal difference in the sidehill screen washing yields as shown in Table 27.

The highest value of sidehill screen washing yield is 88.0% for run no. 7 and the lowest is 85.1% for run no.2 (2.9% difference). The P-values in Table 13 also indicate that 2-way interaction effect of repulping time and displector dosage is statistically significant as the P-value was 0.075 which was lower than 0.100. Its effect was negative, so that in order to achieve higher washing yield, repulping time should be lower and displector should not be used. Shorter repulping time creates less fines and short fibers which has a greater tendency to pass through the screen

Overall Conclusions From First Phase

Overall conclusion that could be made from the first phase of the study is that repulping time should still be kept at low (15 minutes) since it provides better brightness, higher pulp freeness and better pulp viscosity. Enzyme did help in increasing the pulp brightness; however, the role of enzyme dosage was still unclear since enzyme reaction took place very slowly so the reaction time needs to be increased. Displector did not affect pulp freeness but helped in increasing pulp brightness. Yields were not significantly lowered by these three variables. Therefore, for the next phase experiment (second phase), repulping time was kept at 15 minutes but after repulping time, enzyme was not denatured right away but was left to react with the pulp for different time intervals. Displector was used throughout the experiment in the second phase and the amount was fixed at 0.3% based on oven dry (O.D.) of the pulp whereas enzyme was fixed at the same two levels (0.1% and 0.3%).

Second Phase: Optimal Conditions for Enzymatic Deinking

The experiments in this phase were performed by repulping floxographic water-based printed paper for 15 min. Displector dosage was fixed at 0.3% based on O.D. (oven dry) weight of the pulp. Enzyme dosage was still at the same two level (0.1% and 0.3% based on O.D. weight of the pulp). After repulping was done, pulp was left in the insulated slush-maker for 0, 30, 60 and 90 min. before enzyme was

killed. So, the total reaction times for the enzyme were 15, 45, 75 and 105 min. The experiments were done with only single replicate in each case.

The statistical analysis of completely randomized two-factor factorial design was applied to analyze the data from this phase. When any experiment is performed with single replicate, there will be no degree of freedom available for estimating error. So, the error variance is not estimable; that is, the interaction effect and the experimental error cannot be separated in any obvious manner (63). Consequently, there are no tests on main effects unless the interaction effect is zero. If the interaction effect is not significant, then $MS_{interaction}$ (mean square of interaction) can be viewed as an unbiased estimator of the error variance. Thus, the main effects can be tested by comparing MS_{main} (mean square of main effect) to $MS_{interaction}$ or F-values of main effects can be calculated by F_{main} = the ratio of MS_{main} to $MS_{interaction}$. If F-value of the main effect is higher than F-value from F-distribution table, then that particular main effect is statistically significant since the null hypothesis (H_o) of main effects equal to zero can be rejected.

So, the interaction effect needs to be examined first. The significance of the interaction effect can be considered by these following two ways. First is by looking at the Adj SS (adjusted sum of squares) of the interaction effect. If, the value is smaller than Adj SS of main effects, then the interaction effect is not statistically significant. The other way is by looking at the lplot from Minitab. If the lines are parallel or near parallel, then the interaction effect is not significant.

It should be noted that normally in the lplot from Minitab, each data point is not connected to each other. So, all lplot figures presented in this second phase results were modified by adding the lines to connect each data point for easy understanding.

Also, in case that interaction effect was not significant, F-value of main effect was calculated and then compared to the F-value from F-distribution table. For enzyme main effect, F-value was compared to $F_{0.10,1,3}$ which has a value of 5.54. For reaction time main effect, F-value was compared to $F_{0.10,3,3}$ which has a value of 5.39. Since in the first phase data analysis the significance level (α) of 0.10 was used as a criteria, so in this second phase the same significance level of 0.10 was used as well.

For the following discussion, only the lplot figures and ANOVA tables are presented for each pulp evaluation whereas the actual data is separately tabulated in Appendix J.

Brightness After Repulping (Brightness of Slushed Pulp)

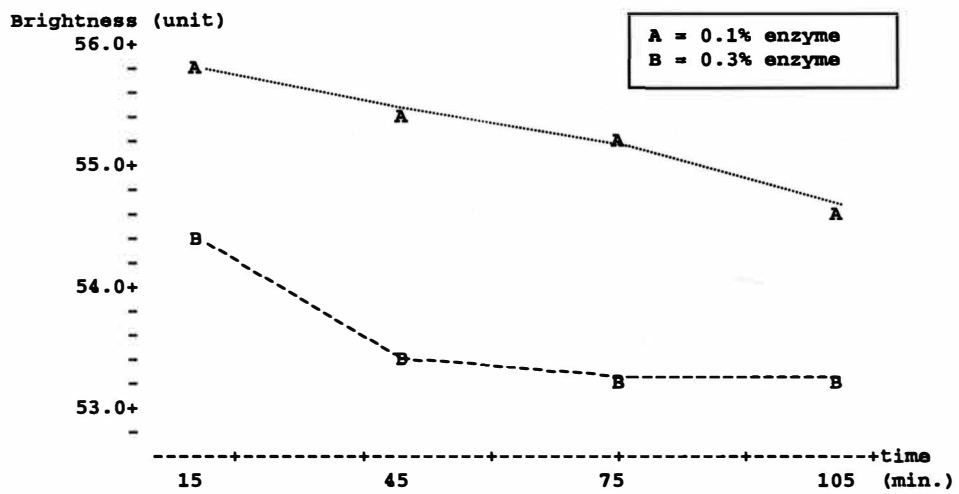


Figure 30. The Lplot From Data Analysis of Brightness After Repulping.

From Figure 30, it could be seen that these two lines are almost parallel. Also, the Adj SS of interaction effect is also much smaller than for both main effects (Table 29). Therefore, the interaction effect is not significant and the main effect can be reviewed separately. The calculated F_{enzyme} (the ratio of Adj MS of enzyme to Adj MS of interaction) is 202.25 which is higher than 5.54 whereas the calculated F_{time} (the ratio of Adj MS of time to Adj MS of interaction) is 18.81 which is higher than 5.39. So, both main effects are statistically significant.

Table 29

ANOVA Table for Brightness After Repulping

Source	DF	Seq SS	Adj SS	Adj MS	F	P
enzyme	1	5.8653	5.8653	5.8653	**	
time	3	1.6361	1.6361	0.5454	**	
enzyme*time	3	0.0870	0.0870	0.0290	**	
Error	0	0.0000	0.0000	0.0000		
Total	7	7.5885				

As shown in Figure 30, longer reaction time has a tendency to decrease the brightness after repulping. This may be because enzyme had more time to react with the pulp to release ink particles to the system. Also, lower enzyme dosage provides higher brightness after repulping than higher enzyme dosage. This may be because more ink particles can be dislodged by using higher enzyme amount. By the same time, hemicellulose and fine contents, which are more accessible to enzyme can be attacked more by higher enzyme dosage. This leads to the higher average fiber length

in the case of high enzyme dosage as compared to low enzyme dosage (the effect of enzyme on fiber length of slushed pulp is discussed later in this second phase). This may lower the light scattering coefficient in the case of higher enzyme dosage and cause the lower brightness value. Moreover, enzyme itself has a dark brown color, which may affect the pulp brightness as well.

The regression equation for brightness after repulping is:

Brightness = 56.9 - 8.60 enzyme - 0.0131 time + 0.003 interact (R^2 = 97.1%)

For all regression equations in this second phase, enzyme is in percentage (%) and time is in minutes (min.)

Brightness After Flotation (Brightness of Floated Accept)

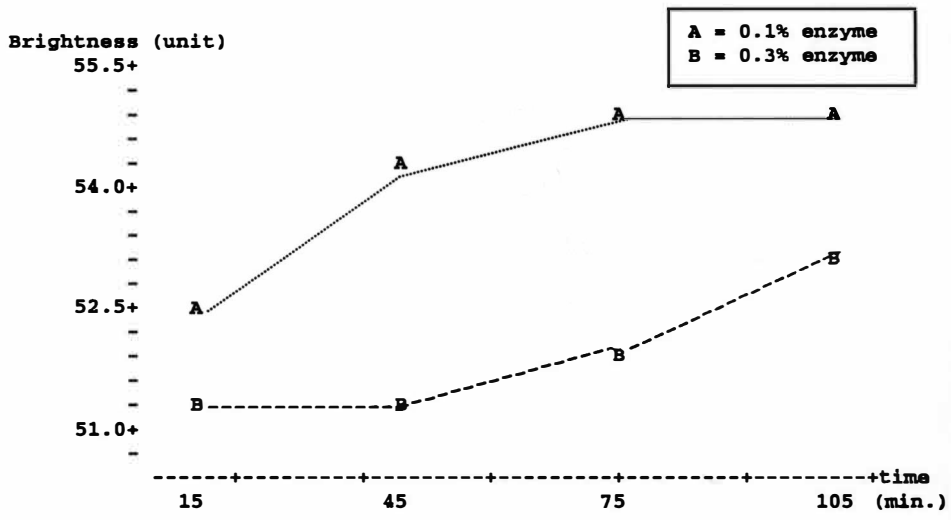


Figure 31. The Lplot From Data Analysis of Brightness After Flotation.

From Figure 31, it can be seen that the parallel between the lines of low and high enzyme dosages is not grossly violated. Also, Adj SS of the interaction effect is

much lower than Adj SS of the two main effects (Table 30). So, the interaction effect is not statistically significant.

The calculated F_{enzyme} (the ratio of Adj MS of enzyme to Adj MS of interaction) is 27.24 which is higher than 5.54. However, the calculated F_{time} (the ratio of Adj MS of time to Adj MS of interaction) is 4.32 which is lower than 5.39. So, only Enzyme main effect is statistically significant.

Table 30

ANOVA Table for Brightness After Flotation

Source	DF	Seq SS	Adj SS	Adj MS	F	P
enzyme	1	10.5341	10.5341	10.5341	**	
time	3	5.0162	5.0162	1.6721	**	
enzyme*time	3	1.1601	1.1601	0.3867	**	
Error	0	0.0000	0.0000	0.0000		
Total	7	16.7104				

As shown in Figure 31, lower enzyme provides higher brightness after flotation. The explanation can be the same as in the case of brightness after repulping. In addition to that, high enzyme dosage started with more ink particles released into the system during repulping stage as compared to low enzyme dosage. Thus, it might be possible that by the end of flotation time the amount of ink particles left in the floated accept might be higher in the case of high enzyme dosage.

The regression equation for brightness after flotation is:

Brightness = 53.6 - 9.10 enzyme + 0.0329 time - 0.190 interact (R^2 = 93.2%)

Brightness After Sidehill Screen Washing (Brightness of Washed Accept)

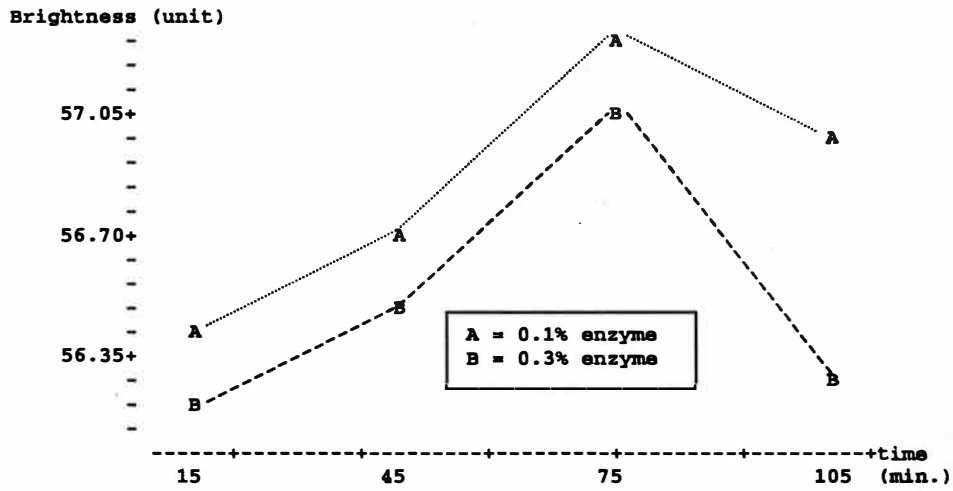


Figure 32. The Lplot From Data Analysis of Brightness After Screen Washing.

Figure 32 indicates that the lines are almost parallel except the last segment at 105 min-reaction time. The Adj SS of interaction effect is also smaller than the Adj SS of the two main effects (Table 31). The calculated F_{enzyme} is 7.74 whereas the calculated F_{time} is 8.73. Both are higher than the F-table values (5.54 and 5.39 respectively) and hence both main effects are statistically significant.

Table 31

ANOVA Table for Brightness After Sidehill Screen Washing

Source	DF	Seq SS	Adj SS	Adj MS	F	P
enzyme	1	0.22111	0.22111	0.22111	**	
time	3	0.74744	0.74744	0.24915	**	
enzyme*time	3	0.08564	0.08564	0.02855	**	
Error	0	0.00000	0.00000	0.00000		
Total	7	1.05419				

The role of enzyme in brightness after sidehill screen washing is similar to the case of brightness after flotation since brightness after sidehill screen washing for high enzyme dosage is lower than low enzyme dosage (Figure 32). Also, longer reaction time of enzyme seems to improve the brightness after sidehill screen washing except the one at 105 min-reaction time. This again may be because enzyme had longer time to react with the pulp so more ink particles, which were dislodged during repulping had more chances to be removed during flotation and washing stages. Also, it is likely that the enzyme with the combination of acidic repulping helps in decreasing the re-deposition problem of flexographic water-based inks because at longer time more ink particles were removed by flotation and washing stages.

The brightness after sidehill screen washing for the case of 105 min-reaction time is lower for both low and high enzyme dosages when compared to the other reaction times. This may be because at longest reaction time the amount of ink particles was so high or the size of ink particles was so large that washing may not be effective as compared to flotation.

The regression equation for brightness after sidehill screen washing is:

Brightness = 56.4 - 0.10 enzyme + 0.0120 time - 0.141 interact $(R^2 = 46.9\%)$

Brightness Change by Flotation

Brightness change by flotation was defined as the difference between brightness after flotation and brightness after repulping (brightness change by flotation = brightness after flotation - brightness after repulping).

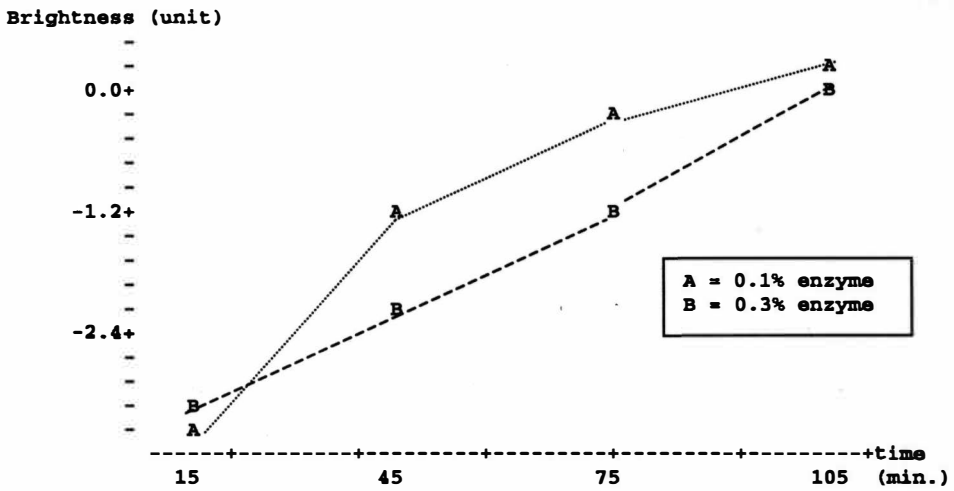


Figure 33. The Lplot From Data Analysis of Brightness Change by Flotation.

As already seen in the first phase results, Figure 33 reveals that there is a loss of brightness due to the flotation. It is suspected that there might be the fiber fractionation occurring during the flotation stage although the yield loss at flotation is low.

Table 32

ANOVA Table for Brightness Change by Flotation

Source	DF	Seq SS	Adj SS	Adj MS	F	P
enzyme	1	0.6786	0.6786	0.6786	**	
time	3	12.2342	12.2342	4.0781	**	
enzyme*time	3	0.6402	0.6402	0.2134	**	
Error	0	0.0000	0.0000	0.0000		
Total	7	13.5531				

The results from lplot also indicate that interaction might not be significant here since the parallel of the lines is not severely violated and Adj SS of interaction is lower than Adj SS of both main effects (Table 32). However, the calculated F_{enzyme} which is equal to 3.18 is lower than F-table value (5.54). So, the enzyme main effect is not statistically significant. The value of F_{time} is also calculated and its value of 19.11 is much larger than F-table value (5.39). This means that only reaction time is statistically significant. Longer reaction time decreases the brightness loss due to flotation stage. This is so obvious since brightness after repulping is lower with increasing reaction time whereas brightness after flotation is increased with increasing reaction time. So, the brightness loss should be lower when the reaction time was longer (see the definition of brightness change by flotation as mentioned earlier). Though the main effect of enzyme is not significant, it should be noticed that brightness loss is decreased by using low enzyme dosage.

The regression equation for brightness change by flotation was:

Brightness change = -3.32 - 0.50 enzyme + 0.0460 time - 0.193 interact (R^2= 93.4%)

Brightness Change by Sidehill Screen Washing

Brightness change by sidehill screen washing was defined as the difference between brightness after sidehill screen washing and brightness after flotation (brightness change by sidhill screen washing = brightness after sidehill screen washing - brightness after flotation). As shown in Figure 34, there is a brightness gain due to the washing stage since all brightness change values are positive.

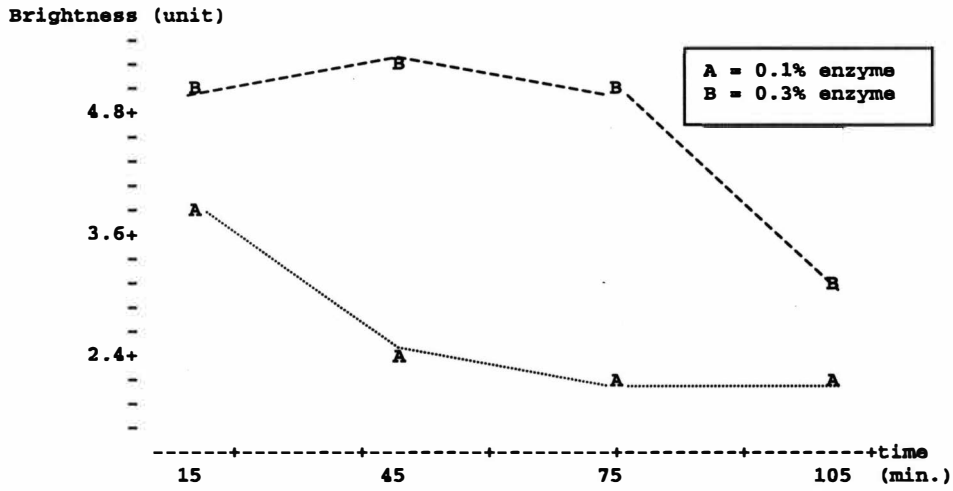


Figure 34. The Lplot From Data Analysis of Brightness Change by Screen Washing.

As shown in Figure 34, the parallel of these two lines was not grossly violated. Also, Table 33 illustrates that Adj SS of interaction is smaller than Adj SS of reaction time and enzyme main effects. Consequently, interaction effect is not statistically significant and the main effect could be reviewed separately. The calculated F_{enzyme} is 15.65 which is much higher than the F-table value of 5.54. However, the calculated F_{time} which is equal to 2.24 is smaller than the F-table value of 5.39. So, only enzyme main effect is statistically significant.

It could be clearly seen that brightness gain by sidehill screen washing is increased by using higher enzyme dosage. This is because the values of brightness after flotation for high enzyme dosage are much lower than low enzyme dosage whereas the values of brightness after sidehill screen washing for high enzyme dosage are only slightly lower than low enzyme dosage (see Appendix J). This makes the

difference between brightness after sidehill screen washing and after flotation much higher for the case of high enzyme dosage. So, the brightness gain by washing is much higher for the high enzyme dosage case.

Table 33

ANOVA Table for Brightness Change by Sidehill Screen Washing

Source	DF	Seq SS	Adj SS	Adj MS	F	P
enzyme	1	7.7028	7.7028	7.7028	**	
time	3	3.3132	3.3132	1.1044	**	
enzyme*time	3	1.4766	1.4766	0.4922	**	
Error	0	0.0000	0.0000	0.0000		
Total	7	12.4927				

Also, it should be noted that in all repulping conditions, brightness after sidehill screen washing is higher than brightness after flotation and brightness after repulping. So, washing still seems to be a very effective way to remove flexographic water based ink particles.

The regression equation for brightness change by sidehill screen washing is:

Brightness change = 2.88+9.20 enzyme-0.0209 time+0.049 interact (R^2 = 86.2%)

Freeness After Repulping (Freeness of Slushed Pulp)

The interaction effect appears to be statistically significant since the parallel of the lines in Figure 35 is violated. In addition, Adj SS of interaction is larger than Adj SS of time (Table 34). So, there is no need to discuss the main effects because the

effects of enzyme and reaction time are already incorporated into the interaction effect.

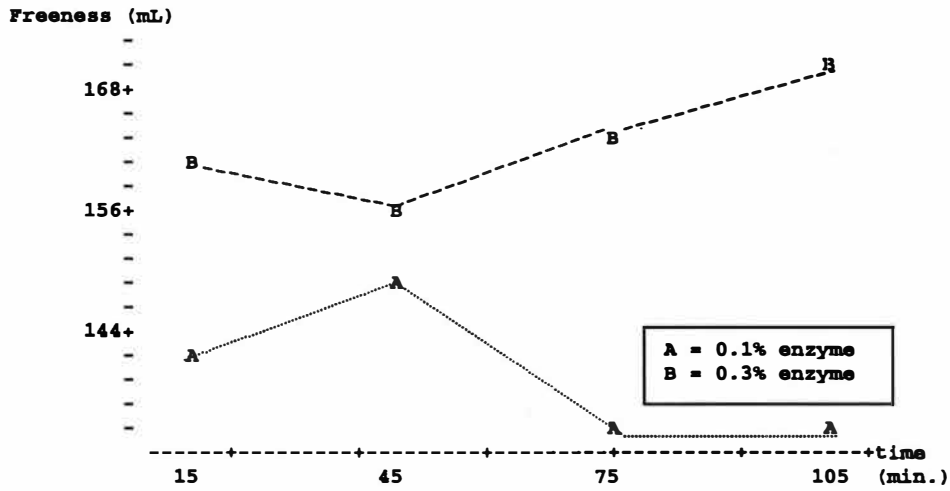


Figure 35. The Lplot From Data Analysis of Freeness After Repulping.

Table 34

ANOVA Table for Freeness After Repulping

Source	DF	Seq SS	Adj SS	Adj MS	F	P
enzyme	1	1035.13	1035.13	1035.13	**	
time	3	22.75	22.75	7.58	**	
enzyme*time	3	227.62	227.62	75.88	**	
Error	0	0.00	0.00	0.00		
Total	7	1285.50				

Nonetheless, by examining the lplot in Figure 35 it could be noticed that high enzyme dosage provides higher freeness at repulping as compared to low enzyme dosage and this is in agreement with several researches (5-10, 47). This may be

because hemicellulose and fine contents are attacked more and removed by higher amount of enzyme. This causes higher freeness for the case of high enzyme dosage.

Reaction time seems to give the positive effect for the case of high enzyme dosage since freeness after repulping shows a tendency to increase with longer reaction time. However, for low enzyme dosage, there was a 15 mL drop in freeness after repulping from reaction time of 45 minutes (freeness = 150 mL) to 75 and 105 minutes (freeness = 135 mL). This is contradictory to what is expected from fiber length studies (discussed later).

The regression equation for freeness after repulping is:

Freeness = 141 + 23.7 enzyme - 0.348 time + 7.20 interact $(R^2 = 90.7\%)$

Freeness After Flotation (Freeness of Floated Accept)

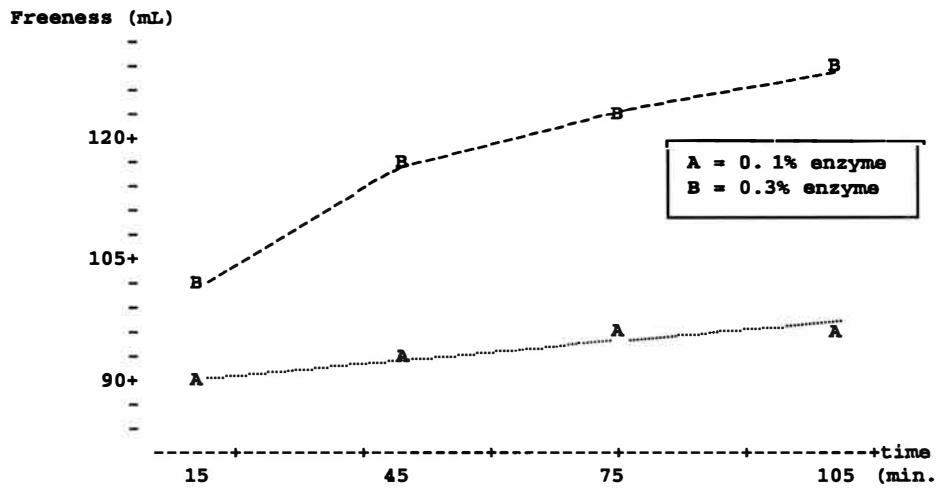


Figure 36. The Lplot From Data Analysis of Freeness After Flotation.

Table 35

ANOVA Table for Freeness After Flotation

Source	DF	Seq SS	Adj SS	Adj MS	F	P
enzyme	1	1081.13	1081.13	1081.13	**	
time	3	262.50	262.50	87.50	**	
enzyme*time	3	94.38	94.38	31.46	**	
Error	0	0.00	0.00	0.00		
Total	7	1438.00				

As illustrated by Figure 36, the two lines are almost parallel. Adj SS of interaction is also much smaller than Adj SS of both main effects (Table 35). Accordingly, interaction effect is not statistically significant.

The enzyme main effect is statistically significant because the calculated F_{enzyme} is 34.36 and much higher than 5.54, the value from the F-table. However, the reaction time main effect is not statistically significant since the calculated F_{time} of 2.78 is smaller than 5.39, the value from the F-table.

It is clear from Figure 36 that high enzyme dosage increases the freeness after flotation as compared to low enzyme dosage. As already mentioned in freeness after repulping, this is because hemicellulose and fine contents were more dissolved in the case of high enzyme.

The regression equation for freeness after flotation is:

Freeness = 82.6 + 42.5 enzyme - 0.128 time + 5.90 interact $(R^2 = 98.6\%)$

Freeness After Sidehill Screen Washing (Freeness of Washed Accept)

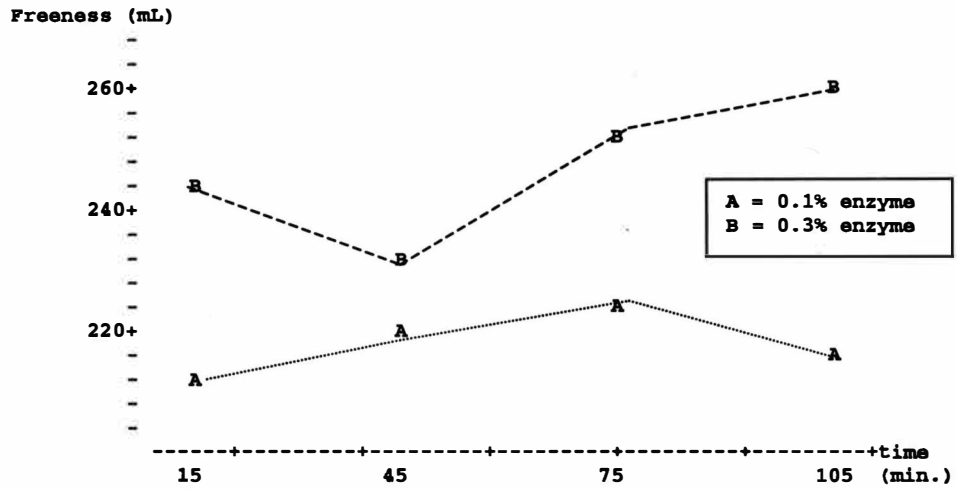


Figure 37. The Lplot From Data Analysis of Freeness After Screen Washing.

Figure 37 shows that the line of low enzyme (A) is not parallel to the one of high enzyme dosage (B). However, the Adj SS of interaction is smaller than Adj SS of both main effects (Table 36). So, interaction effect is likely to be not significant here.

Table 36

ANOVA Table for Freeness After Sidehill Screen Washing

Source	DF	Seq SS	Adj SS	Adj MS	F	P
enzyme	1	1800.00	1800.00	1800.00	**	
time	3	292.25	292.25	97.42	**	
enzyme*time	3	283.25	283.25	94.42	**	
Error	0	0.00	0.00	0.00		
Total	7	2375.50				

The calculated F_{enzyme} has a value of 19.06 which is higher than 5.54, the value from F-table; however, the calculated F_{time} which is 1.03 is much smaller than the value of 5.39 from F-table. So, only enzyme main effect is statistically significant.

As in the cases of freeness after repulping and flotation, freeness after sidehill screen washing is also higher in the case of high enzyme dosage as compared to low enzyme dosage. The effect of enzyme dosage on freeness after sidehill screen washing then could be explained by the same reason as discussed earlier in freeness after repulping and flotation.

The regression equation for freeness after sidehill screen washing is:

Freeness = 201 + 93.7 enzyme - 0.073 time + 4.50 interact $(R^2 = 86.6\%)$

Freeness Change by Flotation

Freeness change by flotation was defined as the difference between freeness after flotation and freeness after repulping (freeness change by flotation = freeness after flotation - freeness after repulping).

It can be seen from Figure 38 that the lines are not parallel and Table 37 also verifies that Adj SS of interaction is much higher than Adj SS of the enzyme main effect. Therefore, interaction effect is likely to be statistically significant and it is not necessary to individually examine the main effects because the main effects of enzyme and reaction time are already combined into the interaction effect. Figure 38 also points out that the roles of enzyme dosage and reaction time as main effects are not so clear here.

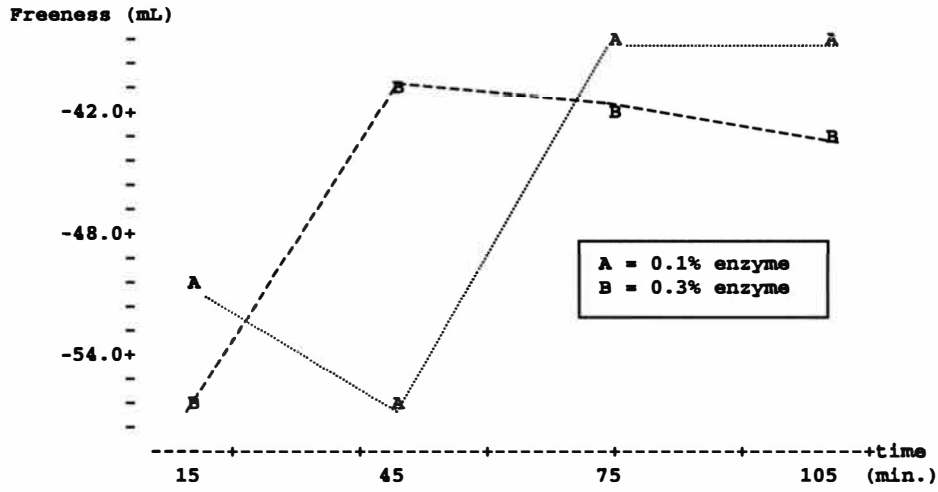


Figure 38. The Lplot From Data Analysis of Freeness Change by Flotation.

Table 37

ANOVA Table for Freeness Change by Flotation

Source	DF	Seq SS	Adj SS	Adj MS	F	P
enzyme	1	0.500	0.500	0.500	**	
time	3	255.250	255.250	85.083	**	
enzyme*time	3	163.250	163.250	54.417	**	
Error	0	0.000	0.000	0.000		
Total	7	419.000				

It should be noted that freeness change by flotation is a loss in freeness due to flotation process. This might be due to the fractionation of longer fibers in the rejects during flotation process. However, the flotation yield indicates that the yield loss is very small (less than 1.5%). Therefore, the loss of freeness by flotation stage needs to be further investigated.

The regression equation for freeness change by flotation is:

Freeness change = -58.1 +18.7 enzyme + 0.220 time -1.30 interact (R^2 = 52.7%)

Freeness Change by Sidehill Screen Washing

Freeness change by sidehill screen washing was defined as the difference between freeness after sidehill screen washing and freeness after flotation (freeness change by sidehill screen washing = freeness after sidehill screen washing - freeness after flotation).

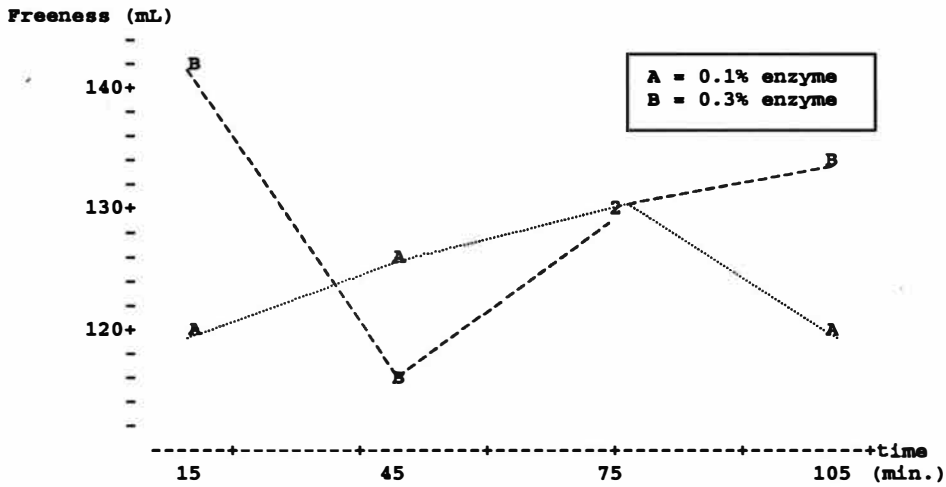


Figure 39. The Lplot From Data Analysis of Freeness Change by Screen Washing.

Figure 39 and Table 38 illustrates that interaction effect is statistically significant since the lines of low and high enzyme dosages are not parallel. In addition to that, Adj SS of interaction is much larger than Adj SS of both enzyme dosage and reaction time main effects. Because the interaction effect is significant,

there are no tests for the individual main effect. It is also interesting to find that the roles of enzyme dosage and reaction time as main effects are inconclusive (Figure 39).

Table 38

ANOVA Table for Freeness Change by Sidehill Screen Washing

Source	DF	Seq SS	Adj SS	Adj MS	F	P
enzyme	1	91.125	91.125	91.125	**	
time	3	107.250	107.250	35.750	**	
enzyme*time	3	315.625	315.625	105.208	**	
Error	0	0.000	0.000	0.000		
Total	7	514.000				

It should be noted that freeness change by sidehill screen washing is a gain in freeness. This might be due to the fact that a lot of fines had been removed through the screen. This is also verified by the lower washing yield (87.1% - 88.4%) as compared to flotation yield (98.7% - 99.4%).

The regression equation for freeness change by sidehill screen washing is:

Freeness change = 119 + 51.2 enzyme + 0.055 time - 1.40 interact $(R^2 = 19.1\%)$

Water Retention Value

As could be seen in Figure 40 that the parallel between the low enzyme dosage line and the high enzyme dosage line is not severely violated. Furthermore, the Adj SS of interaction is smaller than Adj SS of both main effects (Table 39), so the interaction effect is not significant. The investigation of main effects reveals that

both calculated F_{enzyme} and F_{time} values are larger than F-table values (13.84 compared to 5.54 for the case of enzyme dosage main effect and 17.95 compared to 5.39 for the case of reaction time main effect). So, both main effects are statistically significant.

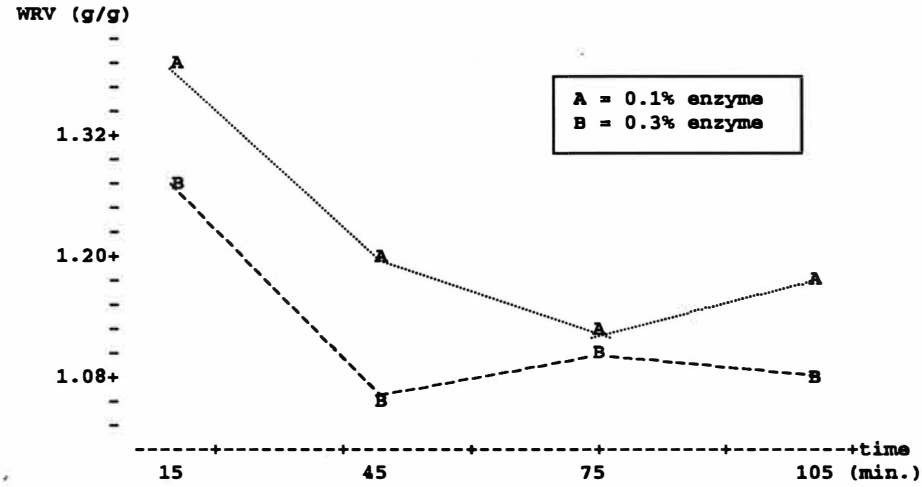


Figure 40. The Lplot From Data Analysis of Water Retention Value (WRV).

Table 39

ANOVA Table for Water Retention Value (WRV)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
enzyme	1	0.017485	0.017485	0.017485	**	
time	3	0.068003	0.068003	0.022668	**	
enzyme*time	3	0.003788	0.003788	0.001263	**	
Error	0	0.000000	0.000000	0.000000		
Total	7	0.089276				

Water retention value is decreased by increasing enzyme dosage. This observation is in agreement with earlier researchers (64,65). This is because more

hemicellulose and fine contents are attacked and dissolved by higher enzyme dosage and this limits the swelling ability of the pulp in water and lowers water retention value of the pulp as compared to lower enzyme dosage. Figure 40 also shows a tendency that longer reaction time of enzyme lowers the water retention value of the pulp.

The regression equation for water retention value (WRV) is:

WRV = 1.43 - 0.675 enzyme - 0.00296 time + 0.0166 interact $(R^2 = 66.2\%)$

Pulp Viscosity

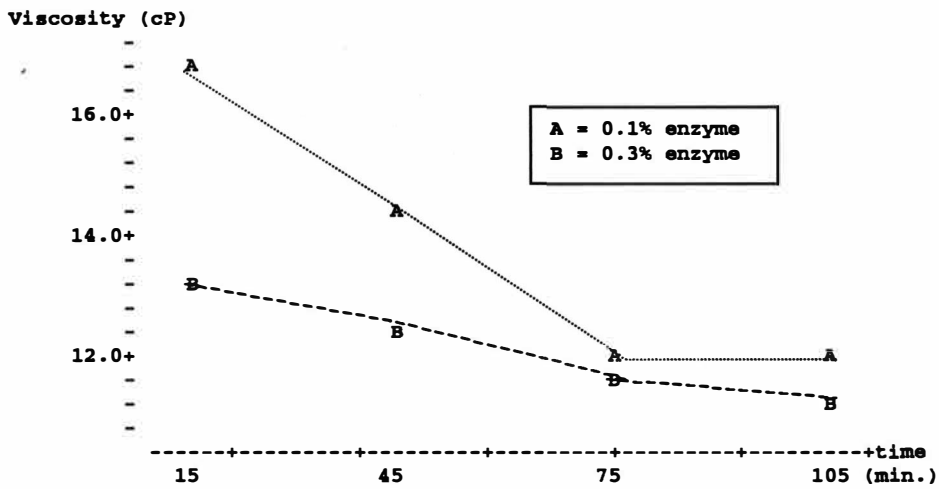


Figure 41. The Lplot From Data Analysis of Pulp Viscosity.

Figure 41 exhibits that the parallel between these two lines is not entirely violated. Table 40 also demonstrates that Adj SS of interaction is smaller than Adj SS of both main effects. The value of calculated F_{enzyme} (6.34) is higher than the value from the F-table (5.54) and for that reason the main effect of enzyme dosage is

statistically significant. However, the calculated F_{time} which is 5.32 is lower than 5.39, the value from F-table. Therefore, the main effect of reaction time is not significant.

Table 40

ANOVA Table for Pulp Viscosity

Source	DF	Seq SS	Adj SS	Adj MS	F	P
enzyme	1	6.1952	6.1952	6.1952	**	
time	3	15.5897	15.5897	5.1966	**	
enzyme*time	3	2.9305	2.9305	0.9768	**	
Error	0	0.0000	0.0000	0.0000		
Total	7	24.7154				

Higher enzyme dosage is detrimental to pulp viscosity. This cellulase enzyme which has about 20% of hemicellulases in it not only dissolved hemicellulose and fine contents but also attacked cellulose and decreased the average molecular weight of cellulose as a whole. Therefore, pulp viscosity is decreased with increasing enzyme dosage. Though the reaction time main effect is not significant, Figure 41 reveals that longer reaction time has a tendency to reduce pulp viscosity.

The regression equation for pulp viscosity is:

Viscosity = 18.9 - 21.2 enzyme - 0.0901 time + 0.994 interact $(R^2 = 94.4\%)$

Flotation Yield

It can be seen from Figure 42 that the lines of low and high enzyme dosage are not parallel. Adj SS of interaction as shown in Table 41 though lower than Adj SS

of both main effects, is not so significant. The value of calculated F_{enzyme} (3.28) is lower than the value from the F-table (5.54) whereas the value of calculated F_{time} (6.58) is higher than the value from the F-table (5.39). Consequently, only the reaction time main effect is statistically significant.

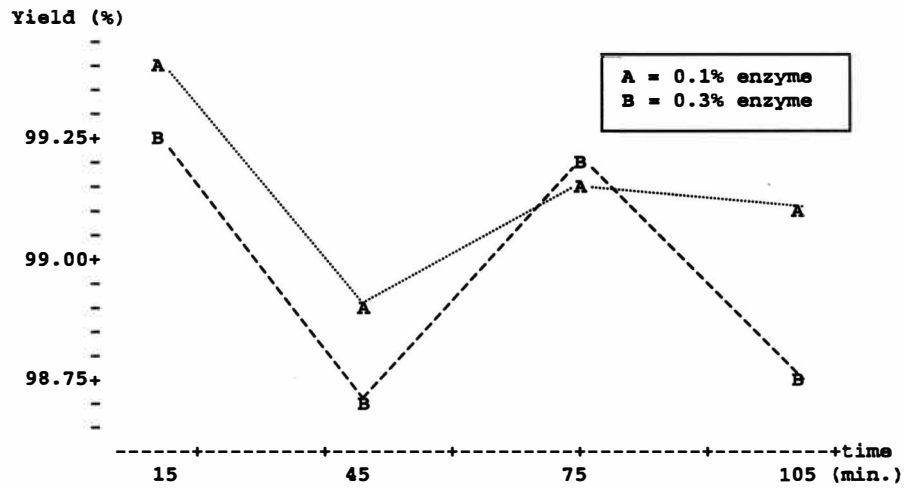


Figure 42. The Lplot From Data Analysis of Flotation Yield.

Table 41

ANOVA Table for Flotation Yield

Source	DF	Seq SS	Adj SS	Adj MS	F	P
enzyme	1	0.049611	0.049611	0.049611	**	
time	3	0.298936	0.298936	0.099645	**	
enzyme*time	3	0.045439	0.045439	0.015146	**	
Error	0	0.000000	0.000000	0.000000		
Total	7	0.393986				

Nevertheless, the lplot in Figure 42 evidently indicates that the roles of enzyme dosage and reaction time are inconclusive and the experimental data also clarifies this trend. The flotation yields of low enzyme dosage at 15, 45, 75 and 105 min- reaction times are 99.4%, 98.9%, 99.1% and 99.1% respectively while the flotation yields of high enzyme dosage at 15, 45, 75 and 105 min-reaction time are 99.2%, 98.7%, 99.2% and 98.8% respectively. By looking at this data, the effects of both enzyme dosage and reaction time on flotation yield are likely to be very small.

The regression equation for flotation yield is:

Flotation yield = 99.3- 0.25enzyme - 0.00057time - 0.043interact (R^2 = 30.6%)

R^2 of the regression equation for flotation yield is rather small attesting to the statistical insignificance of some variables and their ranges used in the experiment.

Sidehill Screen Washing Yield

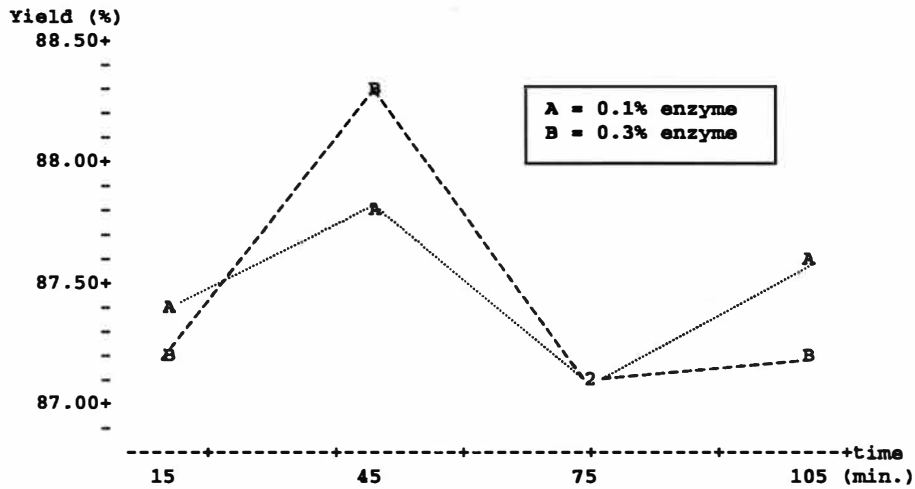


Figure 43. The Lplot From Data Analysis of Sidehill Screen Washing Yield.

The lplot from Minitab as presented in Figure 43 demonstrates that these two lines are not parallel. Apart from that, Adj SS of interaction as shown in Table 42 is larger than Adj SS of the main effect of enzyme. This led to the conclusion that interaction effect was statistically significant.

Table 42

ANOVA Table for Sidehill Screen Washing Yield

Source	DF	Seq SS	Adj SS	Adj MS	F	P
enzyme	1	0.00045	0.00045	0.00045	**	
time	3	1.11405	1.11405	0.37135	**	
enzyme*time	3	0.22825	0.22825	0.07608	**	
Error	0	0.00000	0.00000	0.00000		
Total	7	1.34274				

Like in the case of flotation yield, the roles of enzyme dosage and reaction time are inconclusive. By looking at the experimental data, the effects of both enzyme dosage and reaction time on flotation yield are extremely small or none. The sidehill screen washing yields of low enzyme dosage at 15, 45, 75 and 105 min- reaction times are 87.4%, 87.8%, 87.1% and 87.6% respectively while the sidehill screen washing yields of high enzyme dosage at 15, 45, 75 and 105 min-reaction time are 87.2%, 88.4%, 87.1% and 87.2% respectively. So, it is not significantly different between these yield values.

It should be noted that washing yield is lower than flotation yield since most fines had been removed through the screen during washing stage.

The regression equation for sidehill (SH) screen washing yield is:

SH washing yield = 87.5 + 0.67 enzyme - 0.0000 time- 0.0123 interact (R^2 = 5.2%)

R^2 of the regression equation for sidehill screen washing yield is considerably small attesting to the lack of significance of the variables in their ranges.

Overall Yield

The results in Table 43 point out that Adj SS of interaction effect is larger than Adj SS of the enzyme main effect. It is also clear from Figure 44 that the parallel between the low and high enzyme dosage lines is extremely violated. So, the effect of interaction is statistically significant and it is not necessary to statistically analyze the effect of both main effects.

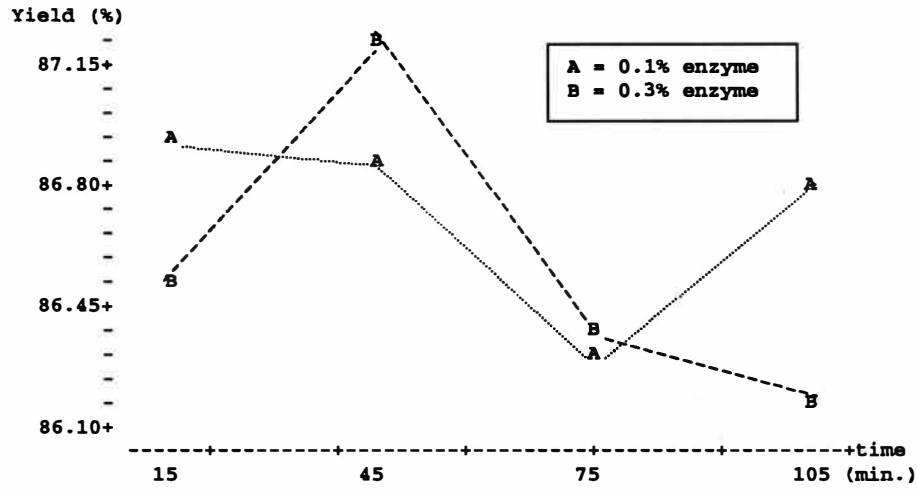


Figure 44. The Lplot From Data Analysis of Overall Yield.

Table 43

ANOVA Table for Overall Yield

Source	DF	Seq SS	Adj SS	Adj MS	F	P
enzyme	1	0.04651	0.04651	0.04651	**	
time	3	0.57404	0.57404	0.19135	**	
enzyme*time	3	0.28924	0.28924	0.09641	**	
Error	0	0.00000	0.00000	0.00000		
Total	7	0.90979				

By looking at the experimental data, both enzyme dosage and reaction time seems to have very little effects on overall yield. The overall yields of low enzyme dosage at 15, 45, 75 and 105 min- reaction times are 86.9%, 86.9%, 86.3% and 86.8% respectively while the overall yields of high enzyme dosage at 15, 45, 75 and 105 min-reaction time are 86.5%, 87.2%, 86.4% and 86.2% respectively. Clearly, the differences between all these values are too small.

The regression equation for grand yield is:

Overall yield = 86.9 - 0.00118 time + 0.35 enzyme - 0.0185 interact (R^2 = 32.1%)

Like flotation yield, R^2 of the regression equation for grand yield is rather small attesting to the statistical insignificance of the variables and their ranges.

Fiber Length

The average fiber length of cellulase enzyme treated pulp was determined and results are tabulated in Table 44.

Table 44

Average Fiber Length of Slushed Pulp

Enzyme Dosage (%)	L_N (mm)			L_L (mm)			L_W (mm)		
	15min	75min	105min	15min	75min	105min	15min	75min	105min
0	0.49	0.49	0.48	1.22	1.25	1.19	2.04	2.07	2.03
0.1	0.47	0.45	0.49	1.19	1.15	1.25	2.00	1.94	2.07
0.3	0.51	0.48	0.49	1.33	1.23	1.24	2.15	2.03	2.06

L_N = Arithmethic average fiber length
L_L = Length weighted average fiber length
L_W = Weight weighted average fiber length

It can be seen from Table 44 that reaction time and enzyme both have effects on pulp fiber length. For the case of control experiment (no enzyme added), fiber length should remain the same since there was no mechanical action after 15 minute repulping time. The results show this trend except in the case of 75 minute reaction time. The effect of reaction time in the case of enzyme treated pulp seems to be very interesting since fiber length first decreases with the reaction time up to the certain point (75 min-reaction time) and after that fiber length increases with longer reaction time.

The effect of enzyme in fiber length, especially in L_L and L_W is clear that enzyme increases the overall average fiber length of the pulp. This effect is more pronounced in the case of high enzyme dosage whereas for the case of low enzyme dosage, the effect is apparently seen at 105min-reaction time. So, for high enzyme dosage, enzyme has more chances to attack hemicellulose, cellulose and fine contents This is the reason for the earlier observations that high enzyme dosage decreases

water retention value but increase pulp freeness (lower hemicellulose and fine contents). Also, pulp brightness is lower due to less light scattering coefficient of the pulp as compared to low enzyme dosage.

Overall Conclusions From Second Phase

The role of enzyme is more clearly seen in this phase. Low enzyme dosage provides not only economics advantage but also better pulp brightness, higher pulp viscosity and higher water retention value as compared to high enzyme dosage. However, higher enzyme dosage should be considered to provide higher freeness values. 15 minute reaction time of enzyme seems to be enough since it provides highest pulp viscosity and water retention values. Nonetheless, if high pulp brightness and freeness is required, enzyme reaction time should be increased. In this study, however, it was found that up to 45 min-reaction time pulp brightness and freeness can be higher by up to 3.5-4.5% and 4.0-5.0% respectively while visocity and water retention value can be reduced by up to 13.0% approximately.

Third Phase: The Effect of Heat on Pulp Brightness

In enzymatic repulping, hot steam was introduced at the end of reaction time to increase the temperature of the repulping system to 90°C to denature enzyme. The effect of this heat on pulp brightness was examined in this phase. The initial temperature before repulping was adjusted to be about 50°C. The temperature at the end of 15 minute repulping time was about 58°C. After repulping, pulp was left in the

insulated slush-maker for 0, 30, 60, 90, and 120 minute time intervals. Pulp was sampled at the end of each time interval. Hot steam was injected to the pulp to increase the temperature to 90°C and handsheets were made after that. The brightness values of all these pulps at different time intervals are tabulated in Table 45 as brightness of pulp A. In order to compare this heat effect, pulp from 90 and 120 minute time intervals were also sampled without heat treatment at the end of the reaction time and handsheets were made to measure pulp brightness. The brightness values of these pulps are shown in Table 45 as brightness of pulp B.

Table 45

The Effect of Heat Treatment on Pulp Brightness

Elapsed time after repulping (min.)	Brightness of pulp A (unit)	Brightness of pulp B (unit)
0	48.9	-
30	48.4	-
60	48.6	-
90	48.6	50.4
120	48.9	50.2

A = pulp with heat treatment B = pulp without heat treatment

Table 45 indicates that there is no significant difference in pulp brightness values from each time interval after pulp is heated to 90°C. The brightness values of pulp A vary from 48.4 units to 48.9 units. This may be because the pulp temperature before being heated was slightly changed at the end of each time interval since pulp was left in the insulated slush-maker. Temperature was about 58°C at the end of

repulping time (0 minute time interval that pulp was left in the slush-maker) whereas temperature dropped to 56°C at the end of 120 minute elapsed time interval. Furthermore, each pulp was treated by the same way to increase the temperature to the same point at 90°C.

The effect of heat on pulp brightness is more noticeable when pulps are treated at different temperatures. For pulp A, pulp was heated to 90°C whereas temperature of pulp B was about 56-58°C. It was clear that pulp brightness was decreased by about 1.8 units for 90 minute time interval and about 1.3 units for 120 minute time interval when pulp temperature was increased to 90°C.

The heat-induced brightness reduction is mainly caused by the lignin contents in the pulp. Newspaper is generally made from mechanical pulps which have high lignin contents. Yellowing is a complex process and pulp containing lignin normally yellows with thermal or light reactions. It is the free phenolic hydroxyl groups in lignin that are responsible for the facile formation of colored structures (chromophore structures) in high-yield pulp (66,67). The initial step involves the heat-induced formation of phenoxy radicals from phenolic lignin units. These radicals will then react with oxygen to form quinone structures. These reactions are influenced by the presence of oxygen (O_2) and water in the pulp.

Gellersteat and co-workers (66) studied the effects of moisture content on the heat-induced color formation of mechanical pulps by heating the pulp in polyethylene bags at 80°C for 24 hours in the presence of different amounts of water. They found that the heat-induced relative color formation of mechanical pulps is strongly

dependent on the moisture content in the pulp. The color formation is increased with higher moisture content.

Based on this enzymatic deinking study, enzyme itself might also decrease the pulp brightness due to its brown color in nature. However, from the results of this phase, it can be seen that the increased temperature to 90°C for the purpose of denaturing enzyme at the end of repulping time has a strong negative effect on pulp brightness since the pulp brightness was decreased by 1-2 units due to the heat-induced yellowing of the lignin containing pulp.

Fourth Phase: Double Repulping and Washing

In this phase, flxographic water-based printed paper was repulped in the slush-maker with 0.3% displector dosage and 0, 0.1% and 0.3% enzyme dosages respectively for 15 min. At the end of repulping time, temperature was raised to 90°C even for the case of "0%" enzyme dosage. After that, pulp was washed through "250 count" pillowcase until the filtrate was clear and then was centrifuged to remove water out. The washed pulp was repulped in the slush-maker again with 0.3% displector dosage and 0, 0.1 and 0.3% enzyme dosages respectively for 15 min. Again, temperature was increased to 90°C at the end of repulping time for enzyme denature propose. Then, 3 g oven dry (O.D.) weight of this washed stock was randomly sampled to wash through the Britt jar with the combination of distilled water and 0.3% displector until the filtrate was clear.

The pictures of filtrates from 1st washing and 2nd washing of all three cases (0, 0.1 and 0.3 % enzyme dosages) are presented in Figure 45 to Figure 49. Figure 45 shows the picture of filtrates from "0%" enzyme dosage case where beaker number 1 was 1st washing filtrate and beaker number 2 was 2nd washing filtrate. Figure 46 indicates the picture of filtrates from "0.1%" enzyme dosage case where beaker number 3 contained 1st washing filtrate and number 4 contained 2nd washing filtrate. Figure 47 illustrates the picture of filtrates from "0.3%" enzyme dosage case where filtrate from 1st washing was in beaker number 5 and filtrate from 2nd washing was in beaker number 6. The comparison of 1st washing filtrates from 0, 0.1 and 0.3% enzyme dosage is shown in Figure 48 whereas Figure 49 indicates the comparison of 2nd washing filtrates from 0, 0.1 and 0.3% enzyme dosage. The brightness results from this phase are also tabulated in Table 46 and Table 47.

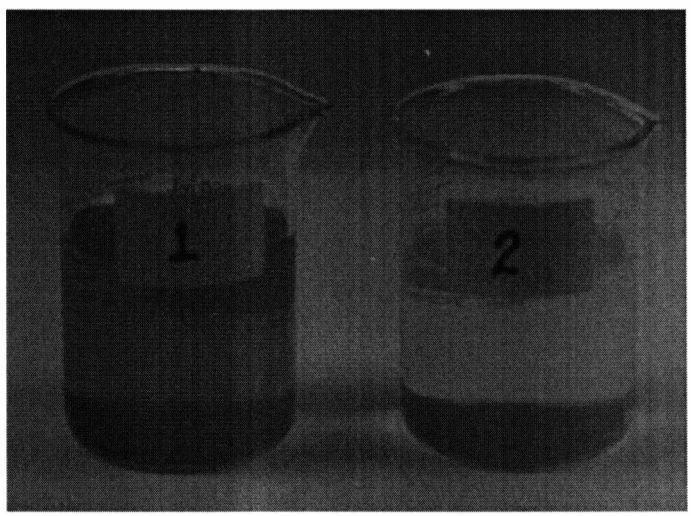


Figure 45. Filtrates From 1st Washing (Beaker No.1) and 2nd Washing (Beaker No.2) for "0%" or "Without" Enzyme Dosage.

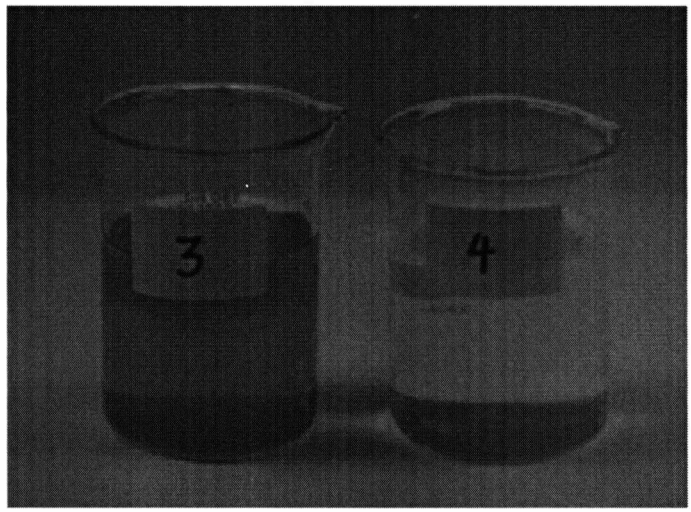


Figure 46. Filtrates From 1st Washing (Beaker No.3) and 2nd Washing (Beaker No.4) for "0.1%" Enzyme Dosage.

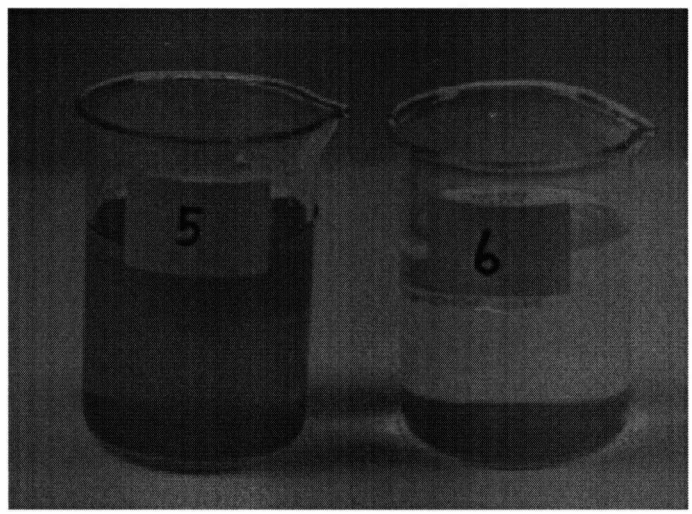


Figure 47. Filtrates From 1st Washing (Beaker No.5) and 2nd Washing (Beaker No.6) for "0.3%" Enzyme Dosage.

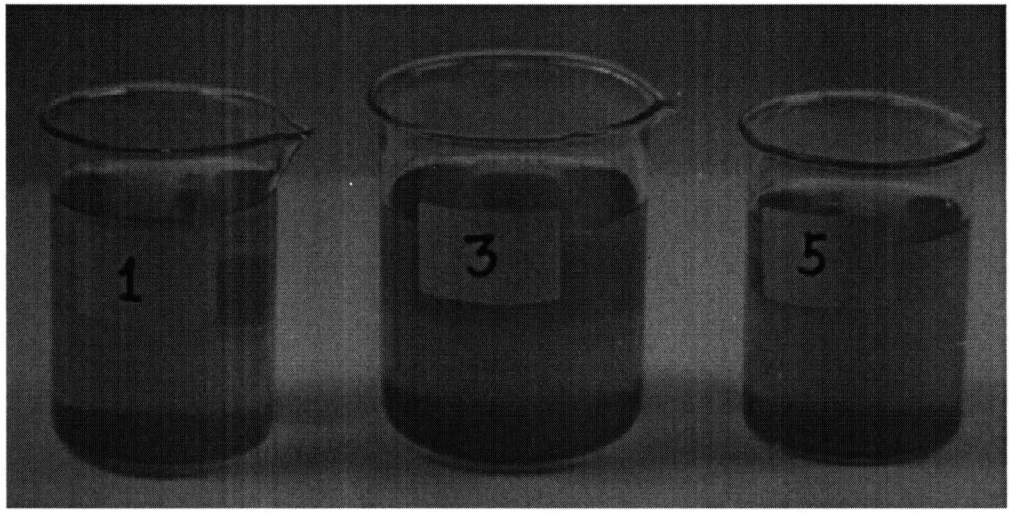


Figure 48. Filtrates From 1st Washing of "Without" Enzyme Dosage (1), "0.1%" Enzyme Dosage (3), and "0.3%" Enzyme Dosage (5).

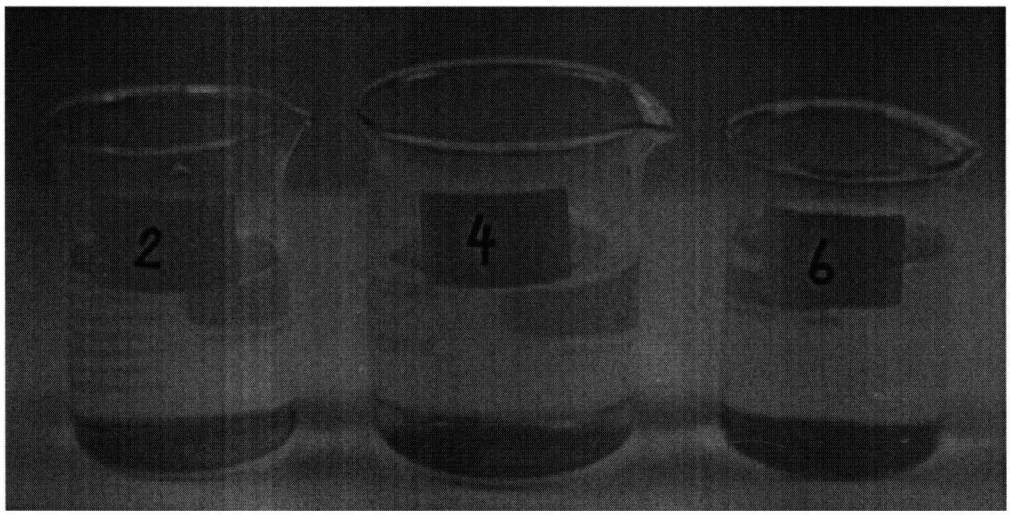


Figure 49. Filtrates From 2nd Washing of "Without" Enzyme Dosage (2), "0.1%" Enzyme Dosage (4), and "0.3%" Enzyme Dosage (6).

Table 46

Brightness of First and Second Stage Repulped and Washed Pulps.

Enzyme Dosage	Pulp brightness after each stage (units)			
	1st repulping	1st washing	2nd repulping	2nd washing
0%	54.3	55.1	54.2	55.7
0.1%	55.5	55.2	53.8	55.3
0.3%	55.4	53.6	52.0	53.0

Table 47

Brightness Change in Each Stage

Enzyme dosage	Brightness change in each stage (unit)		
	1st washing	2nd repulping	2nd washing
0%	+0.8	-0.9	+1.5
0.1%	-0.3	-1.4	+1.5
0.3%	-1.8	-1.6	+1.0

1st Repulping Results

As shown in Table 46, by using enzyme pulp brightness after 1st repulping is increased by 1.2 units for 0.1% enzyme dosage and 1.1 units for 0.3% enzyme dosage as compared to the control pulp (0% enzyme dosage). Like in first and second phase, this indicates that enzyme increases pulp brightness after 1st repulping stage. However, there is no significant difference in pulp brightness after 1st repulping between low and high enzyme dosage levels. Also, the brightness of the control pulp

is quite high (very close to the enzyme treated pulp). It was hypothesized that all brightness values after 1st repulping are very high due to the aging effect. The control pulp in this phase has a brightness value of 54.3 units whereas the control pulp in the first phase has a value of 45.6 units. The experiments in this phase were performed about five months after the experiments in the first phase. So, it is likely that the longer the printed paper has been aged before deinking, the easier the ink particles are removed. This observation is in agreement with Ciampa's work (68) and because of this aging effect, the roles of enzyme and enzyme dosage seem to be unclear as compared to what was found earlier in second phase.

1st Washing Results

The results from Table 46 and 47 also show that 1st washing increased brightness when enzyme was not used in 1st repulping stage since pulp brightness is increased by 0.8 units. On the contrary, by using enzyme in the 1st repulping stage, brightness after 1st washing is reduced by 0.3 units for 0.1% enzyme dosage and 1.8 units for 0.3 % enzyme dosage. There are several explanations for this. First, all pulps seemed to start at a high brightness after 1st repulping due to the aging effect. Second, washing by using pillowcase might not be an effective way as compared to the sidehill screen washing. The pillowcase washing began with a larger amount of pulp where ink particles have a tendency to get entrapped in the fiber mats easier than in sidehill screen washing. Third, it is possible that by repulping the flexographic water-based printed paper under acidic condition (pH = 4.8-5.0) with the aid of enzyme,

most flexographic ink particles are not dispersed into very small size and they could not be washed out effectively by washing process.

The use of enzyme during 1st repulping stage increases pulp brightness after 1st repulping but decreases pulp brightness after 1st washing as compared to the control case (no enzyme use). The decrease in brightness after 1st washing may be first caused by the brown color of enzyme itself. Also, it might be explained by the amount of ink particles released during 1st repulping process. Enzyme added during 1st repulping may help release ink particles by attacking the fiber surfaces or fine particles. The released ink particles are not dispersed into very small size due to the acidic repulping condition. So, the amount of ink particles that were left behind in the pulp after 1st washing was more than the case of the control. Also, since fines which have very high specific surface areas are attacked more by enzyme, pulp brightness of higher enzyme dosage in all cases is always lower than the case of lower enzyme dosage due to the lower light scattering coefficient of long fibers, as compared with fines.

2nd Repulping Results

The results from Table 46 and 47 illustrate that pulp brightness after 2nd repulping is lower in all cases. The drop is between 0.9 units for control and 1.4 and 1.6 units for enzyme usage (0.1 and 0.3%). The brightness drop in all cases may be explained by the role of heat that is used to denature enzyme at the end of reaction time. This heat could reduce the pulp brightness by about 1.3-1.8 units as already

shown in the third phase experiments. The reason why using enzyme decreased pulp brightness after 2nd repulping as compared to without enzyme may be partly due to the color of enzyme itself. The reduction of pulp brightness in the case of using enzyme may also be caused by the higher amount of ink particles released and reabsorbed.

2nd Washing Results

As indicated in Table 46 and 47, pulp brightness after 2nd washing is increased by 1.0 to 1.5 units for all cases (with and without enzyme use). However, the results still show that washing still seems not to be an effective way to remove ink particles in this particular experiment since brightness increase is very small even by using infinite washing. Like brightness after 1st washing, this might be because all pulps seemed to start with a high brightness. Also, this may be due to the size of released ink particles, which were not so small to be efficiently removed by washing. Color of enzyme itself may provide some negative effect in this case as well. In addition to all of those explanations, a displector might not be as functional compared to a dispersant in washing stage.

Higher enzyme dosage may help in releasing ink particles more than lower enzyme dosage. So the amount of ink particles that remain in the system of higher enzyme dosage were more than lower enzyme dosage. This may cause lower pulp brightness in the case of higher enzyme dosage. Also, higher enzyme dosage could

attack more fines than lower enzyme dosage. This leads to lower light scatting coefficient and lower brightness in the case of using higher dosage of enzyme.

It can be concluded from this phase that enzyme seems to help deinking of flexographic water-based ink since it provided higher brightness after 1st repulping. Anyway, it might need longer time to react as in the case of second phase experiments. Also, lower enzyme dosage provided higher pulp brightness than higher enzyme dosage.

Double (or multiple) repulping and washing stages are not likely to be beneficial to deinking process because the average size of ink particles might not be suitable for washing. However, it should be noticed that there is an aging effect taking place during experiments, which might upset the roles of these double stages. It might also be a good idea to try flotation instead of washing in these double stages. Furthermore, the right chemical should be used for this particular work. If washing is chosen, a dispersant type of surfactant should be utilized since it is a washing aid whereas if flotation is chosen, a collector type of surfactant should be used instead since it is a flotation aid.

CHAPTER VI

CONCLUSIONS

1. It was found that this cellulase enzyme aids in the deinking of flexographic water-based ink. Pulp brightness is increased with the use of enzyme as compared to the control pulp.

2. Lower enzyme dosage (0.1% based on O.D. weight of the pulp in this study) provides higher pulp brightness, water retention value and pulp viscosity than higher enzyme dosage (0.3% based on O.D. weight of the pulp in this study). However, pulp freeness is higher by using high enzyme dosage.

3. Enzyme seems to attack hemicellulose and fines first due to easy accessibility. This brings about the lower water retention value but higher pulp freeness. The cellulose is also attacked by enzyme and this leads to lower pulp viscosity.

4. High enzyme dosage might reduce the light scattering coefficient of the pulp and this can lower the pulp brightness. The dark brown color of enzyme itself might have a negative impact on pulp brightness as well.

5. Enzyme in this particular study is not detrimental to the pulp yield.

6. Heat has a negative effect in pulp brightness since pulp brightness is decreased by almost up to 2 units by the hot steam injected to denature enzyme.

7. Aging of printed paper seems to help deinking of flexographic water-based ink as seen by the increased pulp brightness.

8. In this study it was found that double repulping and washing stages do not provide any benefit on deinking of flxographic water based-ink. This may be because the aging effect might disturb the roles of these double stages. Also, the use of displector might not be suitable for the washing stage.

CHAPTER VII

RECOMMENDATIONS FOR FURTHER STUDY

1) Ink particle sizes should be determined for both control (without enzyme) and enzyme treated pulp. Experiments should be set in such a way that the repulping time is fixed and the paper is repulped at acidic pH (pH = 5). The ink particle size distribution should be determined on the handsheets from these slushed pulps to check how enzyme affects the ink particle and ink redeposition to the fibers and also to check whether flotation and/or washing process should be used after repulping.

2) To check how the enzyme changes the fiber structures and how this will affect the ink redeposition onto the fibers, the unprinted paper should be repulped at fixed repulping time and acidic pH with and without enzyme addition. Ink deposition characteristics can then be studied by adding a limited amount of ink to these pulps.

3) Cationic polymers might be used to flocculate the ink particles. This might improve the flotation process.

4) Hemicellulase enzyme should be tried in the enzymatic deinking. However, this enzyme would work effectively only in acidic pH.

5) Carboxymethylcellulose (CMC) might be used to prevent the redeposition of ink particles. CMC, which acts like an anionic surfactant will absorb onto fiber surfaces and add more negative charges to fibers. This should repel the ink particles. However, since CMC provides dispersion effect, it might hurt the flotation process.

Appendix A

Standard Conditions for Determining the Activity of Celluclast 1.5L

Standard Conditions for Activity Determination of Celluclast 1.5L

Substrate	10 g/L CMC (Hercules 7 LFD)
Temperature	40°C
pH	4.8
Reaction time	20 min.
Buffer	0.1 M acetate
Enzyme concentration	approx. 0.041 NCU/ml

Appendix B

Displector Properties

DI-600 Displector Properties

Product name:	DI-600
Product class:	Alkoxylated Nonionic Fatty Acid Deinking Surfactant
Application:	For the deinking of newsprint, magazine and/or woodfree wastepaper grades in flotation/washing deinking systems
Appearance:	Yellow and brown liquid with a grassy odor
Water content:	Less than 2%
Freeze point:	Less than 32 °F
Could point:	48 °C or 118.4 °F
Specific gravity:	1.02g/cc @ 110 °F
BOD-5:	0.19g oxygen/ g
COD:	1.1g oxygen/g
Ionic character:	Nonionic
Soulubility:	Soluble in water

Appendix C

Procedure to Make a Brightness Pad, Equations to Calculate Brightness Change by Flotation, and Brightness Change by Sidehill Screen Washing

Procedure to Make a Brightness Pad

1) Weigh the pulp sample equivalent to 3 g oven dry (O.D.) weight.
2) Dilute it with distilled water to 0.65% consistency, the consistency of the floated accepts.
3) Adjust the pH to 5.0 ± 0.1.
4) Place a 150 mm filter paper in a Buchner Funnel and filter the diluted pulp.
5) Put the pad on the metal plate by allowing the pad surface in contact with the metal plate.
6) Lay the brightness pad and the metal plate on the press.
7) Put two blotters before assembling the next set of the brightness pad and the metal plate.
8) Cover the top with two blotters after up to four brightness pads are assembled. Then, put on the cover of the press.
9) Hand-tighten all four wing nuts and then raise the pressure to 50 psig within 30 seconds and maintain it for 90 seconds.
10) Peel the filter papers from the brightness pads.
11) Assemble the metal plates and brightness pads into a set of drying rings. Place it in a conditioned room (50% ± 2% relative humidity and 23°C± 1°C temperature).

Equation 1: Brightness Change by Flotation

B change by flotation (unit) = B after flotation - B after repulping

Equation 2: Brightness Change by Sidehill Screen Washing

B change by washing (unit) = B after SH screen washing - B after flotation

Where B = Brightness and SH = Sidehill

Appendix D

Procedure to Measure Pulp Freeness, Equations to Calculate Freeness Change by Flotation, and Freeness Change by Sidehill Screen Washing

Procedure to Measure Pulp Freeness

1) Weigh the pulp sample equivalent to 3 g oven dry (O.D.) weight.
2) Dilute it to 0.30% ± 0.02% consistency and mix the pulp stock well.
3) Transfer 1000 mL into a clean 1-L cylinder and measure the temperature of the sample.
4) Mix the sample in cylinder by closing the top of the cylinder with the hand and gently invert the cylinder 180° three times.
5) Pour the stock into the drainage chamber.
6) Close the lid and the air-cock. Open the bottom lid.
7) Fully open the air-cock after 5 seconds from the time that the addition of the stock is completed.
8) Record the volume discharged from the side orifice in milliliters (mL) when the side drainage has stopped. This is the freeness reading before the correction.
9) Combine the discharges from the side and bottom orifices with the pulp left in the drainage chamber. Then, determine the consistency.
10) Correct the freeness reading according to the consistency and the temperature by using the table of freeness corrections to 0.3% consistency and 20°C temperature.

Equation 3: Freeness Change by Flotation

F change by flotation (mL) = F after flotation - F after repulping

Equation 4: Freeness Change by Sidehill Screen Washing

F change by washing (mL) = F after sidehill screen washing - F after flotation

Where F = Freeness

Appendix E

Procedure to Determine Water Retention Value (WRV)

Procedure to Determine Water Retention Value (WRV)

1) Determine the amount of dry fiber required, which yields a specimen of 1400 g/m^2 oven dry (O.D.) weight on the filter screen of the specimen holder by using the following equation:

$$\text{Dry fiber required} = \pi r^2(1400 g/m^2)$$

Where r = radius of the filter screen of the specimen holder (m)

In this experiment, r = 0.0125 m, so the amount of dry fiber required = 0.6875 g.

2) Dilute pulp sample to 0.5 % consistency. Then, determine the volume of the diluted pulp required for the weight of 0.6875 g of dry fibers.

$$\text{Volume (ml)} = \frac{0.6875 \text{ g}}{0.005 \text{ g/mL}}$$

$$= 137.5 \text{ mL}$$

3) Place the specimen holder on the top of the rubber adapter which is put on top of the Erlenmeyer flask. Then, apply a gentle vacuum to the flask.

4) Pour the pulp specimen onto the specimen holder and allow the pulp to drain water out.

5) Centrifuge the drained pulp for 30 min. at a temperature of 21°C ± 3 °C and 900g, where g is the acceleration due to gravity.

6) The speed of the centrifuge at 900g can be calculated as follows:

$$W = (3600 * 900 * g / 4\pi^2 r)^{1/2}$$

Where W = revolution per min. (rpm)

$g = 9.8 m/s^2$ or $32.174 ft/s^2$

r = radius from a specimen holder to the center of the centrifuge (m or ft)

7) Weigh as quickly as possible, each specimen and holder to the nearest 0.001g. Then, dry the specimens and holders in an oven for at least two hours at 105°C ± 3 °C.

8) After drying, cool the specimens and holders in a desiccator jar for 30 min.

9) Weigh each specimen and holder to the nearest 0.001g. Cover the desiccator jar all the time except when removing a holder.

10) Calculate water retention value (WRV) as follows:

$$WRV = (W_1 - W_2)/ W_2$$

Where W_1 = the wet specimen weight calculated by subtracting the weight of the specimen holder alone from the weight of the specimen and specimen holder after centrifuging.

W_2 = The dry specimen weight calculated by subtracting the weight of the specimen holder alone from the weight of the specimen and specimen holder after drying.

Appendix F

Procedure of Lignin Removal and Pulp Viscosity Determination

Procedure of Lignin Removal

1) Put 5 g over dry (O.D.) weight of pulp sample into a 500 ml Erlenmeyer flask.
2) Add 160 mL of distilled water and ten drops (about 0.5 mL) of 10% glacial acetic acid into the flask.
3) Add about 1.5 g Sodium chlorite ($NaClO_2$) into the flask and then cover the flask by an inverted 50 mL Erlenmeyer flask.
4) Heat the flask for 1 hour at 70- 80 °C. Rotate gently at intervals during the reaction.
5) After 1 hour, add again ten drops of glacial acetic acid and 1.5 g of Sodium chlorite into the flask. Then, repeat step 4.
6) Overall four additions are required. After the fourth heating is completed, cool the flask in an ice bath overnight to stop the reaction.
7) Rinse the treated pulp until the filtrate is clear. The pulp is ready to be tested for its viscosity.

Pulp Viscosity Determination

1) Weigh 0.2500 g ± 0.005 g of oven dry (O.D.) pulp and put it into a dissolving bottle containing several 6-mm glass beads.
2) Add exactly 25 mL of distilled water. Then, cover the bottle and put it in a mechanical shaker.
3) Shake the bottle to completely disperse pulp in the water.
4) Allow the bottle to stand for 2 min.
5) Add exactly 25 mL of Cupriethylenediamine solution. Then, purge with nitrogen for 1 min.
6) Cover the bottle and shake it for at least 15 min. to completely dissolve the pulp fibers.
7) Allow the bottle to stand on its side for 2 min. to degas the pulp solution.
8) Fill the viscometer with the pulp solution to the second etch mark. Then, place it in the constant temperature bath at 25.0°C ± 0.1°C and allow it to reach the same temperature by standing it for at least 5 min.
9) With a suction bulb, draw the pulp solution into the measuring leg of the viscometer. Then, drain it down to wet the surface of the viscometer.
10) Draw the pulp solution above the upper mark.
11) Allow it to drain and record the efflux time in seconds needed for the meniscus to pass from the upper mark to the lower mark.
12) Repeat the experiment. The efflux times should agree within ± 0.2 seconds.

13) Calculate the viscosity of the pulp solution by using the following equation.

$$V = Ctd$$

Where V = viscosity of the pulp solution (cP)

C = viscometer constant found by calibration

= 0.03391 for "X952" viscometer with the size number 150

= 0.03394 for "829A" viscometer with the size number 150

t = average efflux time (sec.)

d = density of the pulp solution

= 1.052 g/cm^3

Appendix G

Fiber Length Determination and Determination of Standard Deviation of FS-100 Fiber Analyzer

Fiber Length Determination by Using Kajaani FS-100 Fiber Analyzer

1) Weigh 4.5 g of oven dry (O.D.) pulp and then add 1350 ml of distilled water into it.
2) Disintegrate it for 5 min. to disperse pulp fibers.
3) Dilute this pulp sample with distilled water to 0.01% consistency with a minimum quantity of 5 L.
4) Add about 50 ml ± 0.05 ml into a glass capillary tube of the fiber analyzer and then start the experiment.
5) Stop the experiment when it reaches 8,000 counts.
6) Arithmetic average fiber length (L_N), length weighted average fiber length (L_L) and weight weighted average fiber length (L_W) are automatically determined and provided by the fiber analyzer.
7) Arithmetic average fiber length (L_N) is calculated by the following equation.

$$L_N = \frac{\sum_{i=1}^{144} n_i l_i}{\sum_{i=1}^{144} n_i}$$

Where n_i = the number of measured fibers in different length fractions

i = the total number of fractions = 144 when resolution is 50 μm

l_i = the average length of the fraction

8) Length weighted average fiber length (L_L) and weight weighted average fiber length (L_W) are calculated by the following equations.

$$L_L = \frac{\sum_{i=1}^{144} n_i l_i^2}{\sum_{i=1}^{144} n_i l_i}$$

$$L_W = \frac{\sum_{i=1}^{144} n_i l_i^3}{\sum_{i=1}^{144} n_i l_i^2}$$

Standard Deviation of FS-100 Fiber Analyzer

The sample of enzyme treated pulp (0.1% enzyme dosage based on oven dry pulp weight) was used to run FS-Fiber Analyzer. It was done in three replicates and about 5,000 of fibers was counted within each replicate. The data are presented in Table 48.

Table 48

Average and Standard Deviation of FS-100 Fiber Analyzer

Replicate number	L_N (mm)	L_L (mm)	L_W (mm)
1	0.48	1.24	2.04
2	0.48	1.25	2.08
3	0.49	1.25	2.06
Average	0.48	1.25	2.06
Standard Deviation	0.0058	0.0058	0.02

L_N = Arithmethic average fiber length
L_L = Length weighted average fiber length
L_W = Weight weighted average fiber length

Appendix H

Flotation Yield, Sidehill Screen Washing Yield, and Overall Yield

Flotation Yield, Sidehill Screen Washing Yield, and Overall Yield

1) Flotation yield (Y_F)

$$Y_F = \frac{((29121 * \text{feed consistency}) - (\text{reject weight} * \text{reject consistency})) * 100}{(29121 * \text{feed consistency})}$$

Where 29121 is a capacity of the flotation cell in grams

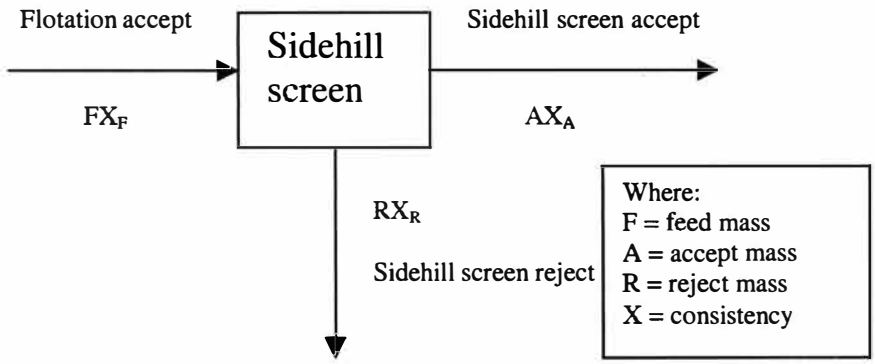
2) Sidehill screen washing yield (Y_W)

$$Y_W = \frac{(\text{SH accept consistency} * (\text{F accept consistency} - \text{SH reject consistency})) * 100}{(\text{F accept consistency} * (\text{SH accept consistency} - \text{SH reject consistency}))}$$

Where SH = Sidehill screen

F = Flotation

Washing yield is derived as follows:



$$F = A + R$$

$$F*X_F = (A*X_A) + (R*X_R)$$

$$F*X_F = (A*X_A) + ((F-A)*X_R)$$

$$F*X_F = (A*X_A) + (F*X_R) - (A*X_R)$$

$$F*(X_F - X_R) = A*(X_A - X_R)$$

$$A/F = (X_F - X_R)/(X_A - X_R)$$

$$\text{Yield} = (A*X_A)/(F*X_F)$$

$$\text{Yield} = (X_A*(X_F - X_R))/(X_F*(X_A - X_R))$$

3) Overall yield (Y_O)

Y_O = (flotation yield * sidehill screen washing yield)/100

Appendix I

Raw Data From First Phase

Table 49

Brightness After Repulping

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Brightness after repulping (unit)
0	control (15 min.)			44.27 46.84
00	control (25 min.)			43.91 45.08
1	-	-	-	49.93 50.82
2	+	+	+	53.63 53.94
3	+	+	-	51.77 53.93
4	+	-	+	51.21 51.24
5	-	-	+	53.48 52.38
6	+	-	-	45.19 46.37
7	-	+	+	49.43 51.92
8	-	+	-	55.41 52.79

"+" sign = high level and "- "sign = low level

Table 50

Brightness After Flotation

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Brightness after flotation (unit)
0	control (15 min.)			43.29 45.88
00	control (25 min.)			43.69 45.57
1	-	-	-	51.62 51.25
2	+	+	+	52.99 49.59
3	+	+	-	50.91 51.61
4	+	-	+	51.99 49.91
5	-	-	+	52.66 50.57
6	+	-	-	46.30 44.90
7	-	+	+	49.72 49.48
8	-	+	-	53.81 49.67

"+" sign = high level and "- "sign = low level

Table 51

Brightness After Sidehill Screen Washing

Run	Repulping ime (min.)	Displector dosage (%)	Enzyme dosage (%)	Brightness after washing (unit)
0	control (15 min.)			50.51 53.46
00	control (25 min.)			48.33 51.99
1	-	-	-	56.22 56.12
2	+	+	+	56.98 55.46
3	+	+	-	54.15 56.13
4	+	-	+	56.27 55.40
5	-	-	+	56.97 56.59
6	+	-	-	51.19 52.53
7	-	+	+	52.85 54.83
8	-	+	-	57.60 55.16

"+" sign = high level and "- " sign = low level

Table 52

Brightness Change by Flotation

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Brightness change by flotation (unit)
0	control (15 min.)			-0.98 -0.96
00	control (25 min.)			-0.22 +0.49
1	-	-	-	+1.69 +0.43
2	+	+	+	-0.64 -4.35
3	+	+	-	-0.86 -2.32
4	+	-	+	+0.78 -1.33
5	-	-	+	-0.82 -1.81
6	+	-	-	+1.11 -1.47
7	-	+	+	+0.29 -2.44
8	-	+	-	-1.60 -3.12

"+" sign = high level and "- "sign = low level

Table 53

Brightness Change by Sidehill Screen Washing

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Brightness change by washing (unit)
0	control (15 min.)			+7.22 +7.58
00	control (25 min.)			+4.64 +6.42
1	-	-	-	+4.60 +4.87
2	+	+	+	+3.99 +5.87
3	+	+	-	+3.24 +4.52
4	+	-	+	+4.28 +5.49
5	-	-	+	+4.31 +6.02
6	+	-	-	+4.89 +7.63
7	-	+	+	+3.13 +5.35
8	-	+	-	+3.79 +5.49

"+" sign = high level and "- "sign = low level

Table 54

Freeness After Repulping

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Freeness after repulping (mL)
0	control (15 min.)			100.0 130.0
00	control (25 min.)			111.0 116.5
1	-	-	-	112.5 138.5
2	+	+	+	100.5 120.2
3	+	+	-	88.0 107.0
4	+	-	+	85.5 123.5
5	-	-	+	120.0 128.8
6	+	-	-	103.5 127.0
7	-	+	+	128.0 151.0
8	-	+	-	112.0 134.0

"+" sign = high level and "-" sign = low level

Table 55

Freeness After Flotation

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Freeness after flotation (mL)
0	control (15 min.)			70.5 73.5
00	control (25 min.)			79.0 79.5
1	-	-	-	87.5 85.0
2	+	+	+	75.5 91.2
3	+	+	-	68.0 81.5
4	+	-	+	60.0 87.8
5	-	-	+	87.0 87.0
6	+	-	-	75.5 87.8
7	-	+	+	91.5 147.5
8	-	+	-	73.5 106.5

"+" sign = high level and "- "sign = low level

Table 56

Freeness After Sidehill Screen Washing

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Freeness after washing (mL)
0	control (15 min.)			201.0 201.5
00	control (25 min.)			188.0 185.5
1	-	-	-	206.5 208.8
2	+	+	+	192.5 234.5
3	+	+	-	176.5 200.0
4	+	-	+	182.5 204.8
5	-	-	+	209.5 210.2
6	+	-	-	176.0 213.0
7	-	+	+	221.5 250.0
8	-	+	-	198.0 227.5

"+" sign = high level and "- "sign = low level

Table 57

Freeness Change by Flotation

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Freeness change by flotation (mL)
0	control (15 min.)			-29.5 -56.5
00	control (25 min.)			-32.0 -37.0
1	-	-	-	-25.0 -53.5
2	+	+	+	-25.0 -29.0
3	+	+	-	-20.0 -25.5
4	+	-	+	-25.5 -35.7
5	-	-	+	-33.0 -41.8
6	+	-	-	-28.0 -39.2
7	-	+	+	-36.5 -3.5
8	-	+	-	-38.5 -27.5

"+" sign = high level and "-" sign = low level

Table 58

Freeness Change by Sidehill Screen Washing

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Freeness change by washing (mL)
0	control (15 min.)			+130.5 +128.0
00	control (25 min.)			+109.0 +106.0
1	-	-	-	+119.0 +123.8
2	+	+	+	+117.0 +143.3
3	+	+	-	+108.5 +118.5
4	+	-	+	+122.5 +117.0
5	-	-	+	+122.5 +123.2
6	+	-	-	+100.5 +125.2
7	-	+	+	+130.0 +102.5
8	-	+	-	+124.5 +121.0

"+" sign = high level and "-" sign = low level

Table 59

Water Retention Value

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Water Retention Value (WRV) (g/g)
0	control (15 min.)			1.366 1.138
00	control (25 min.)			1.403 1.130
1	-	-	-	1.737 1.294
2	+	+	+	1.520 1.302
3	+	+	-	2.015 1.322
4	+	-	+	1.848 1.329
5	-	-	+	1.192 1.339
6	+	-	-	1.868 1.277
7	-	+	+	1.730 1.099
8	-	+	-	1.550 1.009

"+" sign = high level and "- " sign = low level

Table 60

Pulp Viscosity

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Pulp viscosity (cP)
0	control (15 min.)			13.81 16.75
00	control (25 min.)			14.19 15.04
1	-	-	-	15.35 14.78
2	+	+	+	14.94 15.43
3	+	+	-	11.42 12.56
4	+	-	+	14.72 12.95
5	-	-	+	15.28 14.58
6	+	-	-	15.82 13.48
7	-	+	+	12.92 12.62
8	-	+	-	13.92 13.73

"+" sign = high level and "- "sign = low level

Table 61

Flotation Yield

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Flotation yield (%)
0	control (15 min.)			97.89 98.00
00	control (25 min.)			98.01 98.45
1	-	-	-	98.79 98.58
2	+	+	+	99.74 99.15
3	+	+	-	99.68 98.93
4	+	-	+	98.42 98.05
5	-	-	+	98.42 98.42
6	+	-	-	99.64 99.11
7	-	+	+	98.37 99.11
8	-	+	-	98.92 99.19

"+" sign = high level and "- " sign = low level

Table 62

Sidehill Screen Washing Yield

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Washing yield (%)
0	control (15 min.)			87.11 85.56
00	control (25 min.)			86.45 86.04
1	-	-	-	85.67 86.58
2	+	+	+	84.46 85.78
3	+	+	-	85.23 85.42
4	+	-	+	85.17 86.88
5	-	-	+	86.76 85.76
6	+	-	-	85.65 87.49
7	-	+	+	87.15 88.90
8	-	+	-	85.49 86.74

"+" sign = high level and "- " sign = low level

Table 63

Overall Yield

Run	Repulping time (min.)	Displector dosage (%)	Enzyme dosage (%)	Overall yield (%)
0	control (15 min.)			85.27 83.85
00	control (25 min.)			84.73 84.71
1	-	-	-	84.63 85.35
2	+	+	+	84.24 85.05
3	+	+	-	84.96 84.51
4	+	-	+	83.82 85.18
5	-	-	+	85.39 84.40
6	+	-	-	85.34 86.71
7	-	+	+	85.73 88.11
8	-	+	-	84.57 86.04

"+" sign = high level and "-" sign = low level

Appendix J

Raw Data From Second Phase

Table 64

Brightness After Repulping, Brightness After Flotation and Brightness After Sidehill Screen Washing

Run	Reaction time (min.)	Enzyme dosage (%)	B after R (unit)	B after F (unit)	B after W (unit)
1	15	0.1	55.86	52.49	56.41
2	45	0.1	55.35	54.22	56.67
3	75	0.1	55.15	55.00	57.25
4	105	0.1	54.63	54.89	56.95
5	15	0.3	54.36	51.23	56.18
6	45	0.3	53.40	51.23	56.48
7	75	0.3	53.26	51.94	57.03
8	105	0.3	53.12	53.02	56.26

B = Brightness, R = Repulping, F = Flotation, W = Sidehill screen washing

Table 65

Brightness Change by Flotation and by Sidehill Screen Washing

Run	Reaction time (min.)	Enzyme dosage (%)	B change by F (unit)	B change by W (unit)
1	15	0.1	-3.37	+3.92
2	45	0.1	-1.13	+2.45
3	75	0.1	-0.15	+2.25
4	105	0.1	+0.26	+2.06
5	15	0.3	-3.13	+4.95
6	45	0.3	-2.17	+5.25
7	75	0.3	-1.32	+5.09
8	105	0.3	-0.10	+3.24

B = Brightness, F = Flotation, W = Sidehill screen washing

Table 66

Freeness After Repulping, Freeness After Flotation and Freeness After Sidehill Screen Washing

Run	Reaction time (min.)	Enzyme dosage (%)	F′ after R (mL)	F′ after F (mL)	F′ after W (mL)
1	15	0.1	141.0	90.5	210.0
2	45	0.1	149.5	92.5	219.0
3	75	0.1	135.0	96.5	225.5
4	105	0.1	135.0	96.0	215.5
5	15	0.3	160.0	103.0	245.0
6	45	0.3	157.0	116.0	232.0
7	75	0.3	163.5	122.0	252.0
8	105	0.3	171.0	127.5	261.0

F′ = Freeness, R = Repulping, F = Flotation, W = Sidehill screen washing

Table 67

Freeness Change by Flotation and by Sidehill Screen Washing

Run	Reaction time (min.)	Enzyme dosage (%)	F′ change by F (mL)	F′ change by W (mL)
1	15	0.1	-50.5	+119.5
2	45	0.1	-57.0	+126.5
3	75	0.1	-38.5	+129.0
4	105	0.1	-39.0	+119.5
5	15	0.3	-57.0	+142.0
6	45	0.3	-41.0	+116.0
7	75	0.3	-41.5	+130.0
8	105	0.3	-43.5	+133.5

F′ = Freeness, F = Flotation, W = Sidehill screen washing

Table 68

Water Retention Value (WRV) and Pulp Viscosity

Run	Reaction time (min.)	Enzyme dosage (%)	WRV (g/g)	Pulp viscosity (cP)
1	15	0.1	1.391	16.86
2	45	0.1	1.203	14.52
3	75	0.1	1.131	12.20
4	105	0.1	1.174	11.94
5	15	0.3	1.284	13.36
6	45	0.3	1.055	12.25
7	75	0.3	1.104	11.71
8	105	0.3	1.082	11.16

Table 69

Flotation Yield, Sidehill Screen Washing Yield, and Overall Yield

Run	Reaction time time (min.)	Enzyme dosage (%)	F yield (%)	W yield (%)	O yield (%)
1	15	0.1	99.38	87.45	86.91
2	45	0.1	98.92	87.82	86.87
3	75	0.1	99.13	87.08	86.32
4	105	0.1	99.11	87.57	86.79
5	15	0.3	99.25	87.19	86.54
6	45	0.3	98.72	88.35	87.22
7	75	0.3	99.19	87.08	86.37
8	105	0.3	98.75	87.24	86.15

F = Flotation, W = Sidehill screen washing , O = Overall

Appendix K

An Example of Minitab Program for First Phase

An Example of Minitab Program for First Phase

```
MTB > set c4
DATA> 94 70.3 92 114.3 88.5 93 97 81.3 89.5 81.4 72.5 86 93.5 99 86 93.5
DATA> end
MTB > name c4 'shsm'
MTB > print c1-c4
MTB > ffactorial shsm = time|disp|enzyme
MTB > ffactorial shsm = time|disp|enzyme;
SUBC> cube time*disp time*enzyme disp*enzyme;
SUBC> fits c10;
SUBC> residuals c11. MTB > let c16 = 'time'*'disp'
MTB > let c17 = 'time'*'enzyme'
MTB > let c18 = 'disp'*'enzyme'
MTB > let c19 = 'time'*'disp'*'enzyme'
MTB > regress 'shsm' 7 'time' 'disp' 'enzyme' c16 c17 c18 c19
MTB > name c16 'td' c17 'te' c18 'de' c19 'threeway'
MTB > regress 'shsm' 7 'time' 'disp' 'enzyme' c16 c17 c18 c19
MTB > nooutfile
```

Appendix L

ANOVA Tables of First Phase Results

Table 70

ANOVA Table for Brightness After Repulping

Estimated Effects and Coefficients for smbright

Term	Effect	Coef	Std Coef	t-value	P
Constant		51.465	0.2879	178.75	0.000
time	-1.110	-0.555	0.2879	-1.93	0.090
disp	2.775	1.388	0.2879	4.82	0.000
enzyme	1.378	0.689	0.2879	2.39	0.044
time*disp	2.040	1.020	0.2879	3.54	0.008
time*enzyme	1.813	0.906	0.2879	3.15	0.014
disp*enzyme	-2.623	-1.311	0.2879	-4.55	0.000
time*disp*enzyme	0.367	0.184	0.2879	0.64	0.541

Analysis of Variance for smbright

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	3	43.321	43.3209	14.4403	10.89	0.003
2-Way Interactions	3	57.297	57.2971	19.0990	14.40	0.001
3-Way Interactions	1	0.540	0.5402	0.5402	0.41	0.541
Residual Error	8	10.611	10.6108	1.3263		
Pure Error	8	10.611	10.6108	1.3263		
Total	15	111.769				

Table 71

ANOVA Table for Brightness After Flotation

Estimated Effects and Coefficients for fbright

Term	Effect	Coef	Std Coef	t-value	P
Constant		50.4362	0.3955	127.53	0.000
time	-1.3225	-0.6612	0.3955	-1.67	0.133
disp	1.0725	0.5363	0.3955	1.36	0.212
enzyme	0.8550	0.4275	0.3955	1.08	0.311
time*disp	1.9275	0.9637	0.3955	2.44	0.041
time*enzyme	1.8350	0.9175	0.3955	2.32	0.049
disp*enzyme	-1.9100	-0.9550	0.3955	-2.41	0.042
time*disp*enzyme	-0.7500	-0.3750	0.3955	-0.95	0.371

Analysis of Variance for fbright

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	3	14.5211	14.5211	4.840	1.93	0.203
2-Way Interactions	3	42.9223	42.9223	14.307	5.72	0.022
3-Way Interactions	1	2.2500	2.2500	2.250	0.90	0.371
Residual Error	8	20.0193	20.0193	2.502		
Pure Error	8	20.0193	20.0193	2.502		
Total	15	79.7128				

Table 72

ANOVA Table for Brightness After Sidehill Screen Washing

Estimated Effects and Coefficients for shbright

Term	Effect	Coef	Std Coef	t-value	P
Constant		55.2781	0.2711	203.92	0.000
time	-1.0287	-0.5144	0.2711	-1.90	0.094
disp	0.2337	0.1169	0.2711	0.43	0.678
enzyme	0.7813	0.3906	0.2711	1.44	0.188
time*disp	1.5988	0.7994	0.2711	2.95	0.018
time*enzyme	1.7462	0.8731	0.2711	3.22	0.012
disp*enzyme	-1.5113	-0.7556	0.2711	-2.79	0.024
time*disp*enzyme	0.0637	0.0319	0.2711	0.12	0.909

Analysis of Variance for shbright

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	3	6.8933	6.8933	2.2978	1.95	0.200
2-Way Interactions	3	31.5571	31.5571	10.5190	8.95	0.006
3-Way Interactions	1	0.0163	0.0163	0.0163	0.01	0.909
Residual Error	8	9.4059	9.4059	1.1757		
Pure Error	8	9.4059	9.4059	1.1757		
Total	15	47.8725				

Table 73

ANOVA Table for Brightness Change by Flotation

Estimated Effects and Coefficients for fsm

Term	Effect	Coef	Std Coef	t-value	P
Constant		-1.029	0.3920	-2.62	0.030
time	-0.213	-0.106	0.3920	-0.27	0.793
disp	-1.702	-0.851	0.3920	-2.17	0.062
enzyme	-0.523	-0.261	0.3920	-0.67	0.524
time*disp	-0.112	-0.056	0.3920	-0.14	0.889
time*enzyme	0.023	0.011	0.3920	0.03	0.978
disp*enzyme	0.712	0.356	0.3920	0.91	0.390
time*disp*enzyme	-1.117	-0.559	0.3920	-1.43	0.192

Analysis of Variance for fsm

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	3	12.867	12.867	4.2889	1.74	0.235
2-Way Interactions	3	2.083	2.083	0.6944	0.28	0.837
3-Way Interactions	1	4.995	4.995	4.9952	2.03	0.192
Residual Error	8	19.668	19.668	2.4584		
Pure Error	8	19.668	19.668	2.4585		
Total	15	39.613				

Table 74

ANOVA Table for Brightness Change by Washing

Estimated Effects and Coefficients for shf

Term	Effect	Coef	Std Coef	t-value	P
Constant		4.8419	0.3122	15.51	0.000
time	0.2938	0.1469	0.3122	0.47	0.651
disp	-0.8388	-0.4194	0.3122	-1.34	0.216
enzyme	-0.0737	-0.0369	0.3122	-0.12	0.909
time*disp	-0.3288	-0.1644	0.3122	-0.53	0.613
time*enzyme	-0.0888	-0.0444	0.3122	-0.14	0.891
disp*enzyme	0.3988	0.1994	0.3122	0.64	0.541
time*disp*enzyme	0.8137	0.4069	0.3122	1.30	0.229

Analysis of Variance for shf

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	3	3.181	3.181	1.0603	0.68	0.589
2-Way Interactions	3	1.100	1.100	0.3666	0.24	0.870
3-Way Interactions	1	2.649	2.649	2.6488	1.70	0.229
Residual Error	8	12.480	12.480	1.5600		
Pure Error	8	12.480	12.480	1.5600		
Total	15	19.409				

Table 75

ANOVA Table for Freeness After Repulping

Estimated Effects and Coefficients for free

Term	Effect	Coef	Std Coef	t-value	P
Constant		117.50	4.198	27.99	0.000
time	-21.20	-10.60	4.198	-2.52	0.036
disp	0.17	0.09	4.198	0.02	0.984
enzyme	4.37	2.19	4.198	0.52	0.616
time*disp	-6.13	-3.06	4.198	-0.73	0.487
time*enzyme	-3.33	-1.66	4.198	-0.40	0.702
disp*enzyme	10.30	5.15	4.198	1.23	0.255
time*disp*enzyme	1.50	0.75	4.198	0.18	0.863

Analysis of Variance for free

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	3	1874.45	1874.45	624.815	2.22	0.164
2-Way Interactions	3	618.64	618.64	206.215	0.73	0.562
3-Way Interactions	1	9.00	9.00	9.000	0.03	0.863
Residual Error	8	2255.89	2255.89	281.986		
Pure Error	8	2255.89	2255.89	281.986		
Total	15	4757.98				

Table 76

ANOVA Table for Freeness After Flotation

Estimated Effects and Coefficients for free

Term	Effect	Coef	Std Coef	t-value	P
Constant		87.050	4.670	18.64	0.000
time	-17.275	-8.637	4.670	-1.85	0.102
disp	9.700	4.850	4.670	1.04	0.329
enzyme	7.775	3.887	4.670	0.83	0.429
time*disp	-8.425	-4.213	4.670	-0.90	0.393
time*enzyme	-7.350	-3.675	4.670	-0.79	0.454
disp*enzyme	11.275	5.637	4.670	1.21	0.262
time*disp*enzyme	-3.100	-1.550	4.670	-0.33	0.749

Analysis of Variance for free

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	3	1811.86	1811.86	603.95	1.73	0.238
2-Way Interactions	3	1008.52	1008.52	336.17	0.96	0.456
3-Way Interactions	1	38.44	38.44	38.44	0.11	0.749
Residual Error	8	2792.06	2792.06	349.01		
Pure Error	8	2792.06	2792.06	349.01		
Total	15	5650.88				

Table 77

ANOVA Table for Freeness After Sidehill Screen Washing

Estimated Effects and Coefficients for free

Term	Effect	Coef	Std Coef	t-value	P
Constant		206.988	4.789	43.22	0.000
time	-19.025	-9.512	4.789	-1.99	0.082
disp	11.150	5.575	4.789	1.16	0.278
enzyme	12.400	6.200	4.789	1.29	0.232
time*disp	-4.350	-2.175	4.789	-0.45	0.662
time*enzyme	-0.200	-0.100	4.789	-0.02	0.984
disp*enzyme	11.725	5.863	4.789	1.22	0.256
time*disp*enzyme	1.325	0.662	4.789	0.14	0.893

Analysis of Variance for free

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	3	2560.13	2560.13	853.377	2.33	0.151
2-Way Interactions	3	625.75	625.75	208.584	0.57	0.651
3-Way Interactions	1	7.02	7.02	7.022	0.02	0.893
Residual Error	8	2935.41	2935.41	366.926		
Pure Error	8	2935.41	2935.41	366.926		
Total	15	6128.32				

Table 78

ANOVA Table for Freeness Change by Flotation

Estimated Effects and Coefficients for smflot

Term	Effect	Coef	Std Coef	t-value	P
Constant		-30.45	3.046	-10.00	0.000
time	3.92	1.96	3.046	0.64	0.537
disp	9.53	4.76	3.046	1.56	0.157
enzyme	3.40	1.70	3.046	0.56	0.592
time*disp	-2.30	-1.15	3.046	-0.38	0.716
time*enzyme	-4.03	-2.01	3.046	-0.66	0.527
disp*enzyme	0.97	0.49	3.046	0.16	0.877
time*disp*enzyme	-4.60	-2.30	3.046	-0.76	0.472

Analysis of Variance for smflot

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	3	470.77	470.77	156.92	1.06	0.419
2-Way Interactions	3	89.76	89.76	29.92	0.20	0.892
3-Way Interactions	1	84.64	84.64	84.64	0.57	0.472
Residual Error	8	1187.71	1187.71	148.46		
Pure Error	8	1187.71	1187.71	148.46		
Total	15	1832.88				

Table 79

ANOVA Table for Freeness Change by Sidehill Screen Washing

Estimated Effects and Coefficients for flotsh

Term	Effect	Coef	Std Coef	t-value	P
Constant		119.938	2.947	40.69	0.000
time	-1.750	-0.875	2.947	-0.30	0.774
disp	1.450	0.725	2.947	0.25	0.812
enzyme	4.625	2.313	2.947	0.78	0.455
time*disp	4.075	2.038	2.947	0.69	0.509
time*enzyme	7.150	3.575	2.947	1.21	0.260
disp*enzyme	0.450	0.225	2.947	0.08	0.941
time*disp*enzyme	4.425	2.212	2.947	0.75	0.474

Analysis of Variance for flotsh

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	3	106.22	106.22	35.41	0.25	0.856
2-Way Interactions	3	271.72	271.72	90.57	0.65	0.604
3-Way Interactions	1	78.32	78.32	78.32	0.56	0.474
Residual Error	8	1112.03	1112.03	139.00		
Pure Error	8	1112.03	1112.03	139.00		
Total	15	1568.30				

Table 80

ANOVA Table for Water Retention Value (WRV)

Estimated Effects and Coefficients for wrv

Term	Effect	Coef	Std Coef	t-value	P
Constant		1.46444	0.08960	16.34	0.000
time	0.19138	0.09569	0.08960	1.07	0.317
disp	-0.04213	-0.02106	0.08960	-0.24	0.820
enzyme	-0.08912	-0.04456	0.08960	-0.50	0.632
time*disp	0.00138	0.00069	0.08960	0.01	0.994
time*enzyme	-0.03163	-0.01581	0.08960	-0.18	0.864
disp*enzyme	0.02787	0.01394	0.08960	0.16	0.880
time*disp*enzyme	-0.16463	-0.08231	0.08960	-0.92	0.385

Analysis of Variance for wrv

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	3	0.18537	0.18537	0.061790	0.48	0.704
2-Way Interactions	3	0.00712	0.00712	0.002372	0.02	0.996
3-Way Interactions	1	0.10841	0.10841	0.108406	0.84	0.385
Residual Error	8	1.02756	1.02756	0.128445		
Pure Error	8	1.02756	1.02756	0.128445		
Total	15	1.32845				

Table 81

ANOVA Table for Pulp Viscosity

Estimated Effects and Coefficients for avevis

Term	Effect	Coef	Std Coef	t-value	P
Constant		14.0312	0.2081	67.42	0.000
time	-0.2325	-0.1162	0.2081	-0.56	0.592
disp	-1.1775	-0.5887	0.2081	-2.83	0.022
enzyme	0.2975	0.1488	0.2081	0.71	0.495
time*disp	0.5225	0.2613	0.2081	1.26	0.245
time*enzyme	0.8925	0.4463	0.2081	2.14	0.064
disp*enzyme	0.7725	0.3862	0.2081	1.86	0.101
time*disp*enzyme	1.2325	0.6162	0.2081	2.96	0.018

Analysis of Variance for avevis

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	3	6.116	6.116	2.0388	2.94	0.099
2-Way Interactions	3	6.665	6.665	2.2218	3.21	0.083
3-Way Interactions	1	6.076	6.076	6.0762	8.77	0.018
Residual Error	8	5.545	5.545	0.6931		
Pure Error	8	5.545	5.545	0.6931		
Total	15	24.402				

Table 82

ANOVA Table for Flotation Yield

Estimated Effects and Coefficients for fyield

Term	Effect	Coef	Std Coef	t-value	P
Constant		98.9075	0.08823	1120.98	0.000
time	0.3650	0.1825	0.08823	2.07	0.072
disp	0.4575	0.2288	0.08823	2.59	0.032
enzyme	-0.3950	-0.1975	0.08823	-2.24	0.056
time*disp	0.1125	0.0562	0.08823	0.64	0.542
time*enzyme	-0.1050	-0.0525	0.08823	-0.60	0.568
disp*enzyme	0.3075	0.1538	0.08823	1.74	0.120
time*disp*enzyme	0.3325	0.1662	0.08823	1.88	0.096

Analysis of Variance for fyield

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	3	1.9942	1.9942	0.6647	5.34	0.026
2-Way Interactions	3	0.4730	0.4730	0.1577	1.27	0.350
3-Way Interactions	1	0.4422	0.4422	0.4422	3.55	0.096
Residual Error	8	0.9965	0.9965	0.1246		
Pure Error	8	0.9965	0.9965	0.1246		
Total	15	3.9059				

Table 83

ANOVA Table for Sidehill Screen Washing Yield

Estimated Effects and Coefficients for shyield

Term	Effect	Coef	Std Coef	t-value	P
Constant		86.1956	0.2383	361.66	0.000
time	-0.8713	-0.4356	0.2383	-1.83	0.105
disp	-0.0988	-0.0494	0.2383	-0.21	0.841
enzyme	0.3238	0.1619	0.2383	0.68	0.516
time*disp	-0.9762	-0.4881	0.2383	-2.05	0.075
time*enzyme	-0.6988	-0.3494	0.2383	-1.47	0.181
disp*enzyme	0.5288	0.2644	0.2383	1.11	0.300
time*disp*enzyme	-0.3588	-0.1794	0.2383	-0.75	0.473

Analysis of Variance for shyield

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	3	3.4946	3.4946	1.1649	1.28	0.345
2-Way Interactions	3	6.8836	6.8836	2.2945	2.52	0.131
3-Way Interactions	1	0.5148	0.5148	0.5148	0.57	0.473
Residual Error	8	7.2706	7.2706	0.9088		
Pure Error	8	7.2706	7.2706	0.9088		
Total	15	18.1636				

Table 84

ANOVA Table for Overall Yield

Estimated Effects and Coefficients for tyield

Term	Effect	Coef	Std Coef	t-value	P
Constant		85.2519	0.2331	365.74	0.000
time	-0.5513	-0.2756	0.2331	-1.18	0.271
disp	0.2988	0.1494	0.2331	0.64	0.540
enzyme	-0.0237	-0.0119	0.2331	-0.05	0.961
time*disp	-0.8713	-0.4356	0.2331	-1.87	0.099
time*enzyme	-0.7838	-0.3919	0.2331	-1.68	0.131
disp*enzyme	0.7862	0.3931	0.2331	1.69	0.130
time*disp*enzyme	-0.0688	-0.0344	0.2331	-0.15	0.886

Analysis of Variance for tyield

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	3	1.5748	1.57477	0.52492	0.60	0.631
2-Way Interactions	3	7.9661	7.96612	2.65537	3.05	0.092
3-Way Interactions	1	0.0189	0.01891	0.01891	0.02	0.886
Residual Error	8	6.9545	6.95445	0.86931		
Pure Error	8	6.9545	6.95445	0.86931		
Total	15	16.5142				

Appendix M

An Example of Minitab Program for Second Phase

An Example of Minitab Program for Second Phase

For Lplot and ANOVA Table

```
MTB > set c1
DATA> 1.391 1.203 1.131 1.174 1.284 1.055 1.104 1.082
DATA> end
MTB > set c2
DATA> 2(1,2,3,4)
DATA> end
MTB > set c3
DATA> (1,2)4
DATA> end
MTB > set c4
DATA> (1,2,3,4,5,6,7,8)1
DATA> end
MTB > name c1 'wrv' c2 'time' c3 'enzyme'
MTB > print c1-c4
MTB > set c20
DATA> 1.391 1.203 1.131 1.174 1.284 1.055 1.104 1.082
DATA> end
MTB > set c21
DATA> 1 1 1 1 2 2 2 2
DATA> end
```

```
MTB > set c22
DATA> 1 2 3 4 1 2 3 4
DATA> end
MTB > name c20 'cellmean' c21 'enzymel' c22 'timel'
MTB > lplot 'cellmean' 'timel' 'enzymel'
MTB > glm wrv = enzyme|time;
SUBC> fits c10;
SUBC> residuals c11.
```

For Regression Equation

```
MTB > set c1
DATA> 1.391 1.203 1.131 1.174 1.284 1.055 1.104 1.082
DATA> end
MTB > set c2
DATA> 2(1,2,3,4)
DATA> end
MTB > set c3
DATA> (1,2)4
DATA> end
MTB > set c4
DATA> (1,2,3,4,5,6,7,8)1
DATA> end
```

```
MTB > name c1 'wrv' c2 'time' c3 'enzyme'

MTB > print c1-c4

MTB > set c2
 DATA> 2(15,45,75,105)

DATA> end

MTB > set c3

DATA> (.1,.3)4

DATA> end

MTB > print c1-c4

MTB > regress 'wrv' 3 'enzyme' 'time' c13

MTB > nooutfile
```

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