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Flotation Deinking of Ultraviolet Radiation Cured Inks

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FLOTATION DEINKING OF ULTRAVIOLET
RADIATION CURED INKS

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FLOTATION DEINKING OF ULTRA VIOLET RADIATION CURED INK

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The objectives of this study were to optimize the variables of pH and surfactant concentration for the maximum flotation efficiency of ultraviolet radiation-cured inks and to increase the understanding of the behavior of the ink particles during flotation. Experiments were designed in two phases: water and ink system and water, fiber and ink system. At 7.5 pH at room temperature the flotation efficiency increased with surfactant concentration to 8.2 µM/L and then dropped with increase in surfactant concentration. Statistical analysis showed that maximum flotation efficiency of 94.8% was obtained at pH of 7.56 at 12.18 µM/L surfactant concentration. Ink particles in the size range of 26-45 µm were found most suitable for flotation. Zeta potential had a linear relationship, negatively, with pH and surfactant concentration for both the ink and water and ink, fiber and water systems.

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CHAPTER I

FLOTATION DEINKING OF ULTRAVIOLET
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Introduction

Waste paper is being recycled in order to conserve natural resources, to reduce the pollution load from solid disposal, and to reduce production costs. In many of the states in the United States recycling has become mandatory and the main recyclable material is paper. Legislation for procuring the recycled products is also being passed in many states, however the economics of recycling plants are marginal because of costs for labor-intensive collection and sorting. Further, recycling of waste papers printed with solventless inks has become more difficult when compared to papers printed with conventional inks (1).

On the other hand, one of the main problems facing the commercial printing industries is that of air pollution. The solvents used in conventional inks, whether they are aliphatic or aromatic, are generally transported out of the press room into the surrounding atmosphere resulting in pollution. As EPA regulations, in particular the Clean Air

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Act Amendment (2) of 1977, Public Law 95-95, are expected to become more strict in the years to come, printers are finding it more and more difficult to increase or even maintain their production efficiencies and product quality while trying to reduce their costs (2).

To reduce the problem of air-pollution due to the solvent effluent, ink manufacturers have introduced high-solids, catalytic inks, and 100% solids inks that reduce the amount of solvent. The viscosity dependence of flexographic and packaging gravure inks make it extremely difficult to increase the solids content. Also, catalytic inks have exhibit lower press speeds and higher drying temperatures than conventional inks and their cost is higher than that of conventional ink systems (2). As a result of these problems radiation-curable inks, which completely eliminate the usage of solvent in the inks, were found suitable for printers. Radiation curable inks are based on ultraviolet, electron beam, infrared and microwave radiation curing ("non-drying") systems.

Ultraviolet radiation curable inks (UV-curable inks) produce no solvent effluent and seal the ink film almost instantly, improving print quality at higher printing speeds. The rapid setting eliminates wet trap and the need for anti-offset powders. The cross-linked polymers

produced by the constituents of the UV-cured inks are difficult to break up during repulping of the waste papers printed with these inks. This produces particles of large size which are difficult to remove from the suspension of ink and fibers by washing processes (3).

Vanderhoff (3) showed that the conventional deinking process could not deink UV-cured inks and minor variations in deinking recipes or procedures did not solve the problem. He suggested the adaptation of the flotation method for deinking these papers. Though flotation seems to be the suitable process for removing ink particles of larger size (50-100µm), the flotation mechanism of these inks and the optimum flotation cell conditions are still not clear. Pornpaitoonsakul (4) did his research work on the deinkability of UV-cured inks and suggested the study of ink particle collection mechanisms. Dinkfeld (5) also did her research work on the feasibility of deinking papers printed with UV-cured inks suggested a search for a clearer description of the optimum flotation cell conditions.

The suggestions from Vanderhoff, Pornpaitoonskul, Dinkfeld, and others led the author to concentrate on the study of the flotation deinking of these papers. A better understanding of the flotation mechanism and behavior of UV-cured ink particles is also an objective of this study.

CHAPTER II

LITERATURE REVIEW

Overview of Washing and Flotation Processes

There are essentially two systems used for the deinking of secondary fibers: washing and flotation. The flotation process is the predominant process used around the world (greater than 65% of the installed capacity). In North America (90% of the deinking capacity) use washing systems. In Europe and Asia the flotation process is the predominant one (above 87% of the deinking capacity). The difference in the regional preference of these processes is because of the difference in the fields of application. Environmental protection regulations and waste effluent also favor one process over the other (6).

The steps involved in the deinking of secondary fiber are repulping or defibering, during which the ink is removed from the fibers; cleaning and screening; separating ink contaminants from fiber stock; and if necessary, bleaching. In the pulper, the chemicals are normally added just prior to the addition of furnish and the consistencies are usually in the range of 4% to 6%, though there is a

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trend toward higher-consistency (12% to 15%) defibering with the objective of saving chemicals, heat, and operating man hours. In order to have a high brightness, bleaching is necessary (6).

According to Pfalzer (6), deinking by washing and flotation differ with respect to:

a. The physical principle of separation,

b. The chemical process and chemical used,

c. The separating equipment, and

d. The properties of the end product.

One of the main factors for the preference of the flotation process is the high yield of the fibers obtained. Repulping of waste paper is similar in both the processes and it is done either in batch or continuous pulpers according to the mill design.

In case of washing (6), the process always requires careful repulping and dispersion, as the ink particles must be reduced to a size smaller than 25μm. In order to have the washing process with the pulper in continuous mode, the pulp should be thoroughly dispersed through the use of effective dispersing agents. The efficiency of ink removal by washing is primarily controlled by particle size. As the size of the ink particles increases, the ink removal efficiency by washing will decrease since the

tendency for ink particle entrapment in the fiber matrix will increase. The separation of ink in the washing process always corresponds to a stock thickening process. For ink particles smaller than 20μm (6), the amount of ink removed is theoretically proportional to the amount of water removed. To increase the fiber yield and save water the countercurrent washing process is usually used, in which the pulp is diluted with clarified water only ahead of the last washing stage.

In case of flotation (6), the separation of ink from the suspension is by floating the ink to the surface by introducing air bubbles and collectors. The ink is deposited by collecting agents (soaps, etc.) onto the air bubbles. In the flotation cell a required amount of air is blended with the pulp suspension, in the form of small bubbles. The air bubbles loaded with the ink particles and dirt rise to the surface of the cell where they are skimmed off as an ink-laden layer of froth.

The efficiency of ink removal by flotation is primarily controlled by the hydrophobic nature of the particle and secondarily by particle size. It is evident from the series of fundamental studies by Larsson, Stenius, and Odberg (7) that the flotation of ink particles is dependent on particle size. The efficiency of flotation

decreases below a certain size of the particles because the bubble-particle collision frequency decreases as the particle diameter decreases. In mineral flotation it is shown that above a certain size the efficiency of particle removal decreases because the particles are too large for total capture by the air bubble. Further, the reduction in the buoyancy of the bubble-particle complex reduces the velocity to surface (6).

Fuerstenau and Healy (8) explained that the operator must optimize the effect of a large number of interacting variables in the actual flotation of complex ores in practice. They (8) used a laboratory flotation device known as "Hallimond tube," in order to understand the basic chemistry of the unit operation of flotation and in particular the surface chemistry that is so critical in obtaining selective separation.

They first conditioned the mineral of interest in the absence of air with the reagents to be studied and poured the mixture into the "Hallimond tube" so that the mineral settled onto the sintered glass surface at the base of the tube. A small magnetic stirrer was used to insure uniform mixing of the particles with the incoming gas bubbles. A controlled volume of a gas was passed through the sintered glass disk and into the agitated bed of minerals at a

controlled rate. The gas bubbles, with their bed of particles, rose to the water surface and since they did not use any frother, the bubbles burst at the water surface. The concentrate of the ink particles so formed by the bursting of the bubbles then dropped into the side arm and was recovered at the end of the experiment, weighed, and compared to that of the non-floated ink particles in the suspension. They found the response of the mineral to any one of the parameters by keeping the rest of the parameters constant.

The quality of the products from washing or flotation depends on the ash and fines content of the raw-material. If the stock is ash-rich, ash extraction in the washing process produces a pulp of more strength. Flotation also improves the basic stock, but not to the same extent as washing because of lower deashing. The conditions will be reversed in case of low-ash secondary fibers. Hence, with flotation and low-ash starting material, there is no significant gain in strength properties due to loss of ash between the raw material and the deinked pulp.

Printing Inks

The classification of printing inks (9) is based on the application, chemical type, drying or curing method,

and special properties. When considering the chemistry of ink removal, chemical type is considered as the most important factor. Generally printing inks consist of pigments, which provide color and contrast to the ink, incorporated into a vehicle that carries the pigment and binds it to the sheet. In order to provide the inks with some special properties, additives (modifiers) are added in small amounts.

Pigments are not soluble in the vehicle and must be mechanically dispersed. As far as deinking is concerned it is the binder in the vehicle that primarily determines the ease with which inks may be removed from the fibers. Ink vehicles usually consist of resins (either natural or synthetic), solvent, and/or oils. Many types of ink vehicles are available with different compositions depending upon the method by which the ink is to be dried or cured. While the ink industry is rapidly developing new ink vehicle systems the trend appears to be toward vehicles that are increasing-ly resistant to conventional deinking chemicals.

UV-Cured Inks

The major ingredients of the uv-curable ink (10) are:

a.   Unsaturated monomers,

b. Prepolymers and/or polymers (generally acrylic in structure) of unique construction,

c. Photoinitiators, and

d. Special additives.

The curing process of this ink follows the classical mechanism of free-radical polymerization, specifically initiation, propagation, chain transfer and termination. The ink films formed finally are strongly influenced by the cross-linking reactions that takes place. The photoinitiator or sensitizer, employed as a free radical source, absorbs energy in the 200-400 nm range from the light and produces free radicals which open the acrylic double bond and initiate the polymerization and cross-linking reactions (2).

The reasons for the preference of uv-cured inks (3) are:

a. No solvent in the effluent.

b. At higher speeds, the ink is instantly sealed by the uv-radiation improving the print quality.

c. Wet trapping between printing stages will be eliminated because of the instant drying.

d. In sheet fed operations, anti-offset powders are not needed.

These cross-linked uv-cured inks prove very difficult

to break up and disperse and are not alkali soluble. The uv-cured inks produce macro-particles of sizes ranging from 50-100 μm, after conventional dispersion during deinking, and are too large to be removed by the washing process (3).

Chemicals Used for Flotation Deinking

The chemical balance is an important criteria for the success of a deinking process. Commonly, there are six basic types of chemicals used in the flotation deinking process: alkali, bleaching agents, stabilizers, chelating agents, surface active agents and collecting agents.

Table I summarizes deinking chemicals for different types of furnishes and gives dosages on the basis of percentage of fiber.

Table 1

Deinking Chemicals for Different Furnish Types (9)

Deinking Chemical	Furnish Type	Dosage, % on fiber
NaOH	Wood-free grades	3.0-5.0
Sodium Silicate	Groundwood grades Lightly-inked ledger	2.0-6.0
Sodium Carbonate	Groundwood grades Lightly-inked grades	2.0-5.0
Sodium or Potassium phosphates	All grades	0.2-1.0
Non-ionic surfactants	All grades	0.2-2.0
Solvents	Wood-free grades	0.5-2.0
Hydrophilic polymers	All grades	0.1-0.5
Fatty acid	All grades	0.5-3.0

Alkali

The alkali (mainly caustic soda) (9) is one of the

most important deinking chemicals used. At high concentrations (equivalent to pH 11.5-12), alkali cansaponify and/or hydrolyse some ink vehicles and swell fibers to aid in breaking up inks and coatings. The aggregation of small ink particles can be prevented by using alkali. In general, increase in the alkali concentration increases the deinking efficiency.

It is thought that the absorption of OH ions increases the electrostatic repulsion between the fibers and the ink particles resulting in greater ink detachment.

The dosages of alkali are expressed as a percentage of oven-dried fiber, whereas, it is the amount of hydroxide ion that is critical for deinking performance. Hence, the dosage required to achieve a given pH will vary and sufficient alkali should be added to attain a desired pH.

Bleaching Agents

Peroxides (9) are generally used for bleaching. Besides bleaching, it also helps in saponifying the ink binders. An equivalent amount of a mixture of hydrogen peroxide and caustic soda can also be added. Another com-mon bleaching agent involves the reducing agent sodium hydrosulfite.

Stabilizers

Sodium silicate is commonly used as a stabilizer (9). Besides the stabilizing effect the silicates are also helpful in the detachment of the ink from the fibers. Compared with caustic and soda ash, silicates provide better ink removal and brighter pulps with less fiber damage.

Silicates are complex solutions containing polymeric silicate anions. It is these surface active silicate anions, which are responsible for many of the deinking functions, such as emulsification and suspension of dispersed inks. This enables the deinking to occur at lower pH's.

Chelating Agents

Heavy metal ions present in the mill water can cause catalytic decomposition of the bleaching chemical and build up deposits in the system. Hence, chelating agents (9) like polyphosphates of sodium are used which can sequester calcium and magnesium ions and produce colorless complexes with cations like iron. EDTA and DPTA are some of the common chelating agents.

Surface Active Agents

Surface active agents play a major role in the

detachment of printing ink from fibers. These agents disperse the color and form the foam needed for the flotation process. Most anionic surfactants (9) have a strong tendency to form foam, whereas non-ionic surfactants have less tendency and hence are preferred.

Surfactants consist of an organic part that has an affinity for oils (hydrophobe) and another part that has an affinity for the water phase (hydrophile). The hydrophilic group is an ionic (in the case of anionic or cationic surfactants) or a highly polar group, whereas the hydrophobic group is usually a long-chained hydrocarbon residue. Surfactants lower the surface tension of the water to enable it to wet more effectively and adsorb onto the surfaces to aid in ink removal and dispersion, by solubilization and emulsification. Ethoxylated alkyl phenols and ethoxylated alkyl linear alcohols are among the most common non-ionic surfactants.

Collecting Agents

To increase the attachment of the ink particles to the passing air bubbles, collectors (9) are used. The collectors destabilize the ink dispersion and extract the ink particles from the ink-fiber suspension. The ink particles are attached to air bubbles in the flotation cell and carried

to the surface for removal. The collectors comprise of soaps or fatty acids, which are several in numbers. Unsaturated acids such as oleic acids and the carboxylates derivatives with small amount of saturated long chain fatty acids are suitable as collecting agents (4). More effective flotation collectors (modified polyester resins) have been developed and lower dosages of these collectors are more effective.

Flotation Parameters

Some of the main flotation parameters are: water hardness, pH, consistency, particle size, bubble size, quantity of air, dwell time and temperature.

Water Hardness

Raimando (11) varied water hardness while maintaining temperature (40°C), time (10min), pH (10), rpm (2600) constant. He found that deinking was virtually negligible when distilled water was used, but he could not find an optimum region of water hardness. At very low hardness (0-36ppm), the froth formation was very poor and he believed this to be caused by the lack of frothing agent at the liquid-air interface (1DH=17.9 ppm $CaCo_3$). At the higher hardness regions (>715ppm) he found that surface tension

reduced considerably and large, black bubbles were present and difficult to control. Schweitzer (12) pointed out that for successful flotation to take place, the surface tension should increase up to a steady value throughout the flotation stage. Lack of surface tension increase showed that the flotation chemicals have not gathered at the liquid-air interface and remained dispersed in the liquid. Raimando (11) showed that the deinking results were unaffected even by increasing the hardness considerably and water hardness between 380 and 500 ppm was used without any detrimental effects.

Bechstein (13) reported maximum flotation is achieved whenever all the hardness agents flocculated and there was a slight excess of free surface- active soap. He also reported that increase in water hardness, above an optimum level,made the frother inactive. If optimum water hardness is exceeded, all the surface active soaps are precipitated, making the frother inactive.

Whereas Bechstein (13) reported that the increase in water hardness, above an optimum level, made the frother inactive, Larsson Anders, Per Stenius, and Lars Odberg (7) reported that the increase in water hardness above the optimum had no significant effect in flotation. Turvey (14) also showed, from his mill experience, that at water

hardnesses of 4 DH and 12 DH the average stock losses were around 3.5% and 6.5%, respectively (10 DH equals 179 ppm CaCO3). Though the fiber loss was more at higher hardness, the degree of deinking also should be considered and an optimum water hardness should be used. In the absence of calcium ions, which is a measurement of the hardness, the ink particles do not float. Small additions of calcium chloride can improve the flotation considerably. The zeta potential will be highly negative in the absence of calcium and decrease with increasing calcium concentrations at the same time as the ink floatability increases. Ink particles behave as rigid solid spheres in the presence of calcium ions, whereas in the absence of calcium they behave as liquid droplets. Above the optimum hardness, the surfactants will be precipitated, thereby suppressing the frother function (7). Most of the synthetic surfactants commonly used are independent of water hardness.

pH

Raimondo (11) studied the effects of pH of the pulp suspension in flotation and found that large bubbles were formed at a pH value of 5 and frothing was at times uncontrollable, with froth cascading over the sides of the flotation cell. Above a pH of 11, he observed a thick dense

froth of very small bubbles and experienced difficulty with fiber flotation. A definite yellowing of ground-wood fibers was another disadvantage of high pH. Though the sheets appeared cleaner in the high pH region, the brightness was lower because of yellowing.

Dinkfeld (5) studied the effect of pH in UV-cured ink pulp suspensions and found that in acidic conditions the suspensions exhibited much lower foam capacity, where as at high pH (10.1), foaming was excessive and carried fibers with it. She used a chemical composition of 2.5% caustic, 3.0% sodium carbonate, 2.5% silicate (40 Be) and 0.05% Triton X-114 (non-ionic). A brightness and yield of 75.1 & 90.1% respectively was obtained at a pH of 6.5. A low pH of 2.2 destroyed foaming ability and gave a lower brightness value of 74.7.

Larsson et al (7) reported that above pH 9, the floatability of ink decreased with increasing pH and below this value there were no effects.

McCool and Zabala (15) found that reducing the pH from 9 to 8.5 reduced the yellowing of pulp.

Forester (16) observed that the relationship between pH and the brightness for UV-cured ink was entirely different as compared to normal emulsified ink. From the reports of other workers, a pH range of 8-10 seemed to be

desirable for the normal emulsified inks, whereas for UV-cured ink a low pH near 6.0 was desirable for maximum brightness.

From the studies of Dinkfeld and Forester, the pH near 6.0 seems to be suitable for this study.

Consistency

Gilkey, Shinohara, and Yoshida (17) found that the high consistency pulping of non-impact printed material was an effective method of imparting fiber-to-fiber contact. When sheets made from high and low consistency pulping were compared, the ink removal was much improved by high consistency treatment and the number of ink particles produced were less when compared to low consistency pulping. They believed that the reduction in the number of ink particles is due to the rubbing action of fibers with one another under high consistency pulping conditions. This separates the ink more completely from the fibers so that it can be removed more effectively by flotation and/or washing. When they examined the ink particles under 30X magnification, they found that the high consistency sheets had small ink area, less number of ink particles and smaller ink particle diameters.

On identical raw material of 100% laser printed

material low consistency produced sheets having approximate-ly four times the total ink area compared to that of the high consistency observation sheets.

They reported another method of imparting a fiber-to-fiber rubbing/rolling action by introducing a continuous high shear dispersion device. The dispersion step involved striking the fibers numerous times with a rotor turning at high speeds. The large clearance between the rotor and stator minimized fiber degradation and freeness drop.

Mah (18) conducted tests in a pilot-scale pulper equipped with a Tri-Dyne rotor. He used a high consistency of 10% with the Tri-Dyne rotor pulper and a low consistency of 5.5% with the Shark rotor. The raw material was waste news. Tests at high consistency showed that the Tri-dyne rotor provided effective and faster defibering action than the Shark rotor at low consistency. But, he found that the pulp deinked at high consistency (10%) was of lower brightness than that of the low consistency pulp deinking.

He could not find any plausible reason for the reduced brightness for high consisteny. Brightness pads for high consistency, under microscopic examination indicated that the ink removal and dispersion at high consistency was excellent. He was not able to replace the Shark rotor with the Tri-Dyne rotor mainly for the reason of reduced

brightness due to high consistency.

From the above studies it is evident that the high consistency pulping gives good ink dispersion in less time. Studies by Gilkey et al. (17) showed that the ink area and the number of ink particles in the pads of high consistency pulping were much less when compared to that of low consistency pulping. As Mah (18) studied the brightness of the sample pad, which is not an appropriate method of finding the deinking efficiency, he was not able to identify the reason for not selecting the Tri-Dyne rotor.

However, for flotation, the size of the ink particle is not a sensitive parameter like that of washing.

Particle Size

Larsson et al.(7) showed from their experimental results (with mineral oil based ink) that flotation followed first order kinetics and it was highly dependent on ink particle size. They plotted the ink removal rate constants versus the mean ink particle diameter in a double logarith- mic graph and the resulting figure had a positive slope, showing that the rate constants were proportional to the ink particle diameter. They found that the flotation rate increased with particle diameter in the range 1-20 um and the production of ink particles in the pulper was mainly

determined by the character of the printing ink and the concentration of the calcium soap in the pulper.

During flotation the mean diameter of the ink particles in the suspension in the first five minutes was reduced showing that the larger ink particles were floated first. The small particles had very low flotation rate so that they could be removed by increasing the flotation time, which in turn resulted in excessive fiber losses without any appreciable increase in the brightness. Thus, the process variables should be adjusted to prevent the production of such small particles or minimal production of such particles by knowing the conditions under which the small particles are produced. Alternatively the small particles may be removed by washing.

The diameter of the ink particles increased in the presence of the precipitated calcium soap in the pulper due to the agglomeration of the ink particles by the calcium soap. pH of the dispersion slightly affected the particle size, whereas alkyl polyglycol ether, water glass concentration, and temperature were without importance at the levels studied (7).

Bechstein (13) explained that the frequency of the non-floated ink particles was highest in the area below five microns and decreased with increasing particle

diameter. Gilkey et al. (17) found that for 100% laser printed papers the average ink particle diameter decreased from 41 µm for low consistency pulping to 23 µm for high consistency pulping. In flotation, the efficiency of ink removal is primarily controlled by the hydrophobic nature of the ink particles and secondly by the ink particle size. Below a certain size, the efficiency of flotation decreases due to the reduction of the bubble-particle collision frequency as the particle size decreases. Similarly, above a certain size, the particles are too large to be captured by the bubbles. The buoyancy of the bubble-particle complex is also reduced by increased particle size thus reducing the ink particle velocity to the surface (8).

Gilkey et al. (17) indicated that the flotation process was suitable for removing ink from 100% laser printed material as flotation by nature is more effective in removing large ink particles compared to washing.

Vanderhoff (3) found that hand-sheets made from the UV-cured ink-pulp suspensions contained ink particles of 50-100 µm diameter. The 50-100 µm ink particles produced by the deinking of UV-cured ink were probably fragments of the ink film and essentially the particles were two dimensional in configuration. His approach to reduce the average size of the ink colloid was to: (a) Increase the digestion time,

(b) Increase the intensity of mechanical agitation, and (c) Add surfactants to the recipe.

Air Quantity and Bubble Size

The efficiency of deinking operation is largely controlled by air addition. This control should be exerted in two distinct areas: air volume and air bubble size.

Flynn (19) showed the differences between the ink removal efficiencies for two different furnishes. He explained that two points should be noted as far as the air addition to the flotation process was concerned. First, a time limit existed beyond which further air addition did not improve performance of ink removal. Secondly, the upper limit varied from furnish to furnish.

Raimando (11) carried out experiments to find the effect of air quantity in the pulp during flotation. He adjusted the valve apertures to regulate the air flow. To overcome the reduction in the speed of the motor due to the reduction of the air quantity, he adjusted the rheostat to keep the motor at same speed. He maintained the other flotation variables constant: temperature (40°C), time (5 min), water hardness (179 ppm), pH (10), and rpm (2300). The air flow through the aperture was assumed to be proportional to the aperture opening.

Raimondo (11) found that the quantity of air required for successful flotation was in fact very small, approximately 10% of the total valve capacity of 15 L/min. He explained that the lower air consumption for efficient flotation might probably correspond to the presence of sufficient air bubbles to lift the ink particles and fillers in the system. Too much air, more than 30%, resulted in unfavorable results. He explained that this could be due to the disturbance of conditions on the liquid surface, thus preventing the formation of a stable froth which was capable of supporting the separated particles. It appeared that once a minimum quantity of air requirement was satisfied, no further advantages would be obtained by the addition of additional air. Maximum brightness was obtained in the first 10% opening of the air valve aperture beyond which there was no significant increase in brightness.

Bubble size is the second air control variable. Flynn (19) found for a given quantity of air, smaller bubbles yielded a larger surface area and hence the bubble size was important. If the surface area generated when a cubic meter of air is uniformly divided into spherical bubbles of the diameters 0.2 and 1.2 mm, the associated surface areas are 30,000 sq.m. and 5000 sq.m., respectively. The additional 25,000 sq.m. of surface area will be able to increase

greatly the opportunity for air/ink contact and attachment. Hence, the practical application of this surface area control allows the cell operation to be tailored to the needs of different types of furnishes.

In a deinking plant the number of air introductions exerts a strong influence on system performance.The efficiency of ink removal was increased by the number of air introductions. The number of introductions can be increased by increasing the number of cells (19).

The size of the air bubble is determined by the air quantity, material and geometry of the opening where the bubbles are formed. Very small bubbles do not break on the surface and too large or too many bubbles causes coalescing thereby reducing the available bubble surface for reaction. Above a certain quantity of the air supply, a point of jet aeration is reached and should be avoided as the bubbles formed may be of any size and number.

Larsson Anders, Per Stenius, and Lars Odberg (7) used an amount of 60 ml/min of air during their experiments in a Hallimond tube holding approximately 100ml of pulp volume they passed the air through a capillary tube, which gave rise to air bubbles of uniform size. In this research work, the amount of air was set at 30 ml/min after a series of trial and error method of determination of optimum air flow.

Dwell Time

To find the influence of the dwell time in ink removal efficiency in flotation deinking, Raimando (11) collected pulp samples at certain intervals throughout the flotation stage and compared the brightness of the pulp samples with the flotation time. Experimental results on laboratory basis was found to be similar to that of mill results, when the brightness of the sample pads were plotted against the flotation time. Brightness values of the two sides of the sheets approached a common value with increasing time of treatment. This was attributed to the more efficient removal of ink, fillers, and fines present in the stock during a longer period of time, until eventually only the fibers were left behind (11).

Raimondo (11) showed a rapid brightness increase during the first 10 min of flotation beyond which the rise was slow. Forester (16) maintained a dwell time of 20 min before flotation.

Temperature

Raimando (11) reported that temperature appeared to have a marked effect on the surface tension of the suspension and at low temperatures bubble formation on the

surface of the suspension was poor with a relatively thin layer. Bubble formation was improved by increase in temperature and good frothing was observed in the upper regions. He found that the brightness increase with temperature was of the order of 0.03% per degree centigrade. Hence, an increase in a temperature of 33°C was required for an increase of 1% brightness. But, in the regions of 42-45°C, there was a sudden increase in brightness which was difficult to explain. Though surface tension decreases notably with temperature, the volume of gas liberated increases with temperature at constant pressure. The combined effect produced by these two variables in this region could have a maximum value.

Further, the waxes, fillers, and ink present in the paper melt at this temperature region and the increased brightness could be due to sticky particles having a greater adherence to the air bubbles in this region. This in turn was helpful in the flotation of the undesirable constituents such as pitch, rubber and plastics, resulting in a clean sheet. The temperature in the region of 40°C appeared the most satisfactory in practice.

Wood (20) recommended a temperature range of 40-45°C for papers containing ground wood. He pointed out that the decomposition of peroxide, if used, takes place above 50°C

and hence when utilizing peroxides, 45$^{\circ C}$ should not be exceeded.

Forester (16), found a large and sudden increase in brightness (for papers printed with UV-cured ink) in the temperature region of 40-45°C. The visual ranking of the cleanliness showed that in the 40-45°C region the sheet looked cleanest but the yield was the lowest (around 65%).

Larsson et al.(7) showed that the floatability decreased somewhat with temperature. The graph plotted with the fraction of non-floated ink versus temperature showed a positive slope of the resulting line indicating that the fraction of non-floated ink increased as the temperature increased.

Zeta Potential

Experimental studies and discussion by Larsson et al. (7) explained that under alkaline conditions normally prevailing in a flotation deinking plant, the ink particles obtain a high and negative zeta potential even without the addition of soap. They believed that it was because of the organic acids present in the ink. The acids are enriched in the surface layer and become ionized at increasing pH values. Whenever soap is added to the suspension, it will adsorb on the ink particles to a great extent and make their

zeta potential even more negative. Ink particles having high negative charges will have electrostatic stabilization, which is favorable for the release of ink from fibers. But this high negative charge efficiently prevents the particle from attaching to air bubbles. Hence zeta potential on the ink particles should be reduced before the flotation step. The reduction in is often done by adding calcium ions, which precipitate the soap and lower the zeta potential to around -30 to -40 mV. In the experiments of Larsson et al. (7), though calcium ions were added in excess, the concentration of calcium ions used was insufficient to remove all the electrostatic stabilization of the particles. The calcium ions when precipitate a part of the soap as a small layer of particles on the surface of the much larger ink particles. This, in principle, is a micro-encapsulation giving the ink particles the hydrophobic surface properties of calcium soap. When air bubbles come in contact with these hydrophobic ink particles, the ink particles will have a strong tendency to attach to the air bubbles.

Calcium ions, by lowering the charge on the ink particles, minimize the repulsion between ink particles thereby forming ink aggregates. These small aggregates will have a much better floatability than the individual particles and the overall flotation rate can be improved

efficiently.

From the data of the experimental work by Porpaitoonskul (4) it was found that with increasing pH from 6 to 10, the zeta potential increased from -27.7 to -52.2 and hence it was evident that under alkaline conditions the zeta potential of the UV-cured ink became more negative. This enabled the detachment of ink particles from the fibers during pulping more effectively.

Contact Angle

Measurement of contact angles has been a standard research technique in flotation studies over many years and a great deal of information has been obtained using the technique (7).

Because of the polar nature of most mineral surfaces, clean mineral solids are wet by water and therefore the contact angle is low to zero. A finite contact angle can be obtained by controlling the wettability of the solids through the adsorbtion of the collectors, which can change the interfacial tension. Depressants are used to selectively reduce the action of the collectors (21).

It is helpful to consider qualitatively the numerical magnitude of the surface tensional stabilization of a particle at a liquid--liquid interface. The free energy to

displace the particle from its stable position will be r
^{AB} (21), where r is the radius of the particle and ^{AB} is
the total surface free energy.

In usual situations, the particle is supported at a
liquid--air interface against gravitational attraction
(Figure 1).
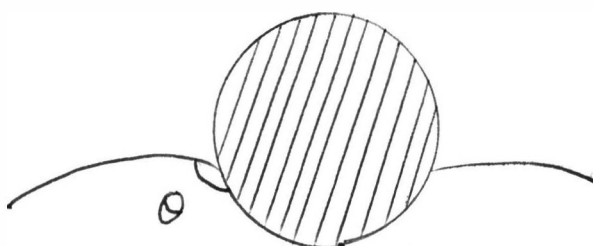
Figure 1.   Particle Supported at a Liquid-Air Interface
            Against Gravitational Attraction.

Source: (20)

The restoring force stabilizing the particle at the
interface varies approximately with the particle radius.
For the particle to remain flotating, this restoring force
must be equal to or exceed that of gravity. As the gravity
varies with the cube of the radius, It is clear that there
will be a maximum size of particle that can remain at the
interface.

In practice, it may be possible with care to float
somewhat larger particles than those corresponding to the
theoretical maximum.
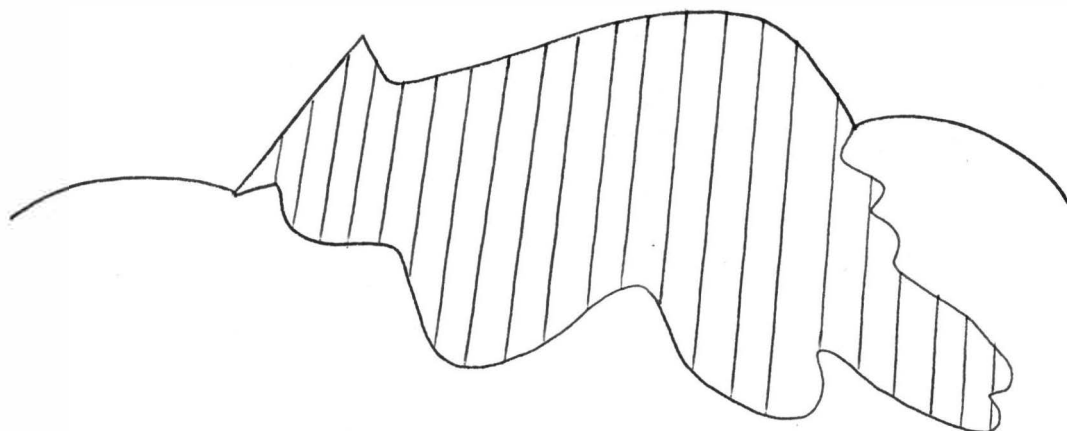
Figure 2. Particle with an Irregular Shape.

Source: (20).

If the particle has an irregular shape (Figure 2), it will tend to float such that the three phase contact occurs at an asperity since the particle would have to be depressed considerably for the line of contact to advance further. The preceding upper limit to particle size can be exceeded if more than one bubble is attached to the particle.

According to Vanderhoff (3), the 50-100 μm particles produced by the deinking of UV-cured ink films are probably fragments of the ink film, essentially two dimensional in configuration. Figure 3 gives the schematic representation of the flotation of such a particle .
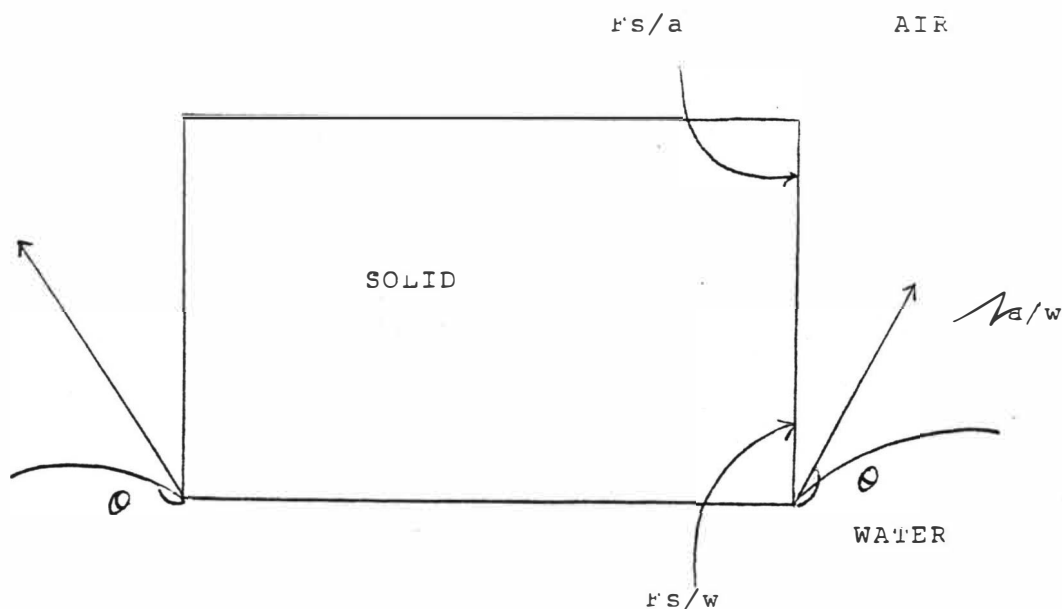
Figure 3.   Schematic Representation of Flotation of
            Cylindrical Particle.

Source: (<u>3</u>)

For the particle to float, the total upward pull of the
meniscus on the vertical sides of the particle must balance
its apparent weight. By theory, the vertical force applied
to such a particle is given by a/w cos 0. Perimeter of the
particle= V(dp-dw)g.

Where

a/w is the surface tension of the water phase.

0-- The contact angle of the water phase on the vertical
sides of the particle

V-- The volume of the particle

dp and dw-- Densities of the particle and the water

phase, respectively and

g-- The gravitational Constant

The maximum size of an ink-film particle that can be floated can be calculated by using the relationship

DH= a/w cos 0 / (dp-dw)g

Where

D and H are the diameter and height of the cylindrical ink particle.

If we substitute the value for (dp-dw) of 1.0 g/cc (corresponding to an ink film density 2.0 gm/cc), gives a value for DH of 0.24 sq.cm or D=H=0.2 cm. These values are greater than the size of the ink particles observed in the deinked paper specimens. The ink particles may be fragments of the original film and thus rather thin, so that the value of D and H may not be equal. If the values of D and H are equal, for a value of H=5 μ, the corres- ponding values of D are 1100 and 480 cm for ink-film densities of 1.2 and 2.0 gm/cc respectively.

Hence, the theory shows that the flotation process can remove particles far larger than the largest particles observed in these laboratory experiments.

CHAPTER III

PROBLEM STATEMENT

From the literature review it is evident that the conventional deinking process effectively does not deink UV-cured ink (3). Vanderhoff (3) concluded that minor variations in deinking recipes or procedures did not solve the problem. His experimental studies suggested the adaptation of the flotation process for deinking these papers. Because of the large size of the UV-cured ink particles, flotation seemed to be a suitable process. Because the flotation mechanism of UV-ink particles and optimum cell conditions are still not clear, the study of the particle collection mechanism for the UV-inks was suggested by Pornpaitoonsakul (4). Dinkfeld (5) suggested the study of optimum flotation cell conditions.

The objectives of this study are to determine:

1.   The effect of temperature, pH and surfactant concentration on flotation efficiency

2. The relationship between zeta potential, pH and surfactant concentration

3.   The optimum values of the pH and surfactant concentration for best flotation. A better understanding

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of the flotation mechanism is also a part of the objective of this research.

CHAPTER IV

EXPERIMENTAL

Summary of Experimental Design

To reach the goal of this study extensive preliminary experiments were carried out to have hands-on experience with the procedure, equipment and analysis of the data. After overcoming the problems encountered in the preliminary study the actual experiments were undertaken.

It was decided to study flotation deinking at different conditions of pH and surfactant concentration. From the simultaneous effect of the two variables on flotation efficiency the optimum conditions for maximum response were determined. In order to reach the close range of the values of variables, pH and surfactant concentration, preliminary experiments were designed with a broad range of the variables. From the results of the preliminary experiments approaches were made so that the optimum range of the variables for the experimental condition could be determined.

Experiments were done using the optimum range of variables obtained from the preliminary studies. The samples from the experiments were analyzed with the Image

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Analyzer to determine the flotation efficiency. The experimental results were then analyzed using polynomial response surface methodology. The polynomial response surface model gave the optimum values of the variables pH and surfactant concentration for the maximum flotation efficiency.

Procedure

Printed papers are ganerally used for deinking research. But in this research, instead of pulping UV-ink printed paper and studying the flotation parameters, a different approach was followed. First ink particles dispersed in water alone were used for fundamental studies of the flotation parameters and after getting an idea of the range of the variables to be used and the type of approach, actual experiments were conducted. The behavior of the ink particles was then evaluated in the presence of pulp fibers.

Preparation of Model UV-Ink Dispersions

Instead of pulping paper printed with UV-cured ink to study the flotation parameters, a dispersion of ink in water similar to that obtained from a deinking mill pulp slurry was used. A series of experiments were carried out

to prepare the dispersions and are explained in detail in Appendix A. The ink dispersions were analyzed with the Image analyzer. The initial particle size distribution was in the range of 50-100 microns diameter. Samples for image analyses were prepared with Millipore membranes to obtain a uniform monolayer of ink particles. The monolayer Millipore sample preparation procedure is explained in Appendix B. The Image analysis technique and procedure are described in Appendix C.

The dispersions prepared in the Waring blender and the ball mill were not useful for this study. The limitations in the preparation of the ink dispersion with these units are discussed in Appendix A. The Morehouse mill, shown in Figure 4, which could disperse the ink particles to the required size distribution (50-100 μm) was chosen for preparing the final ink-water dispersions.

The Hallimond Tube

Instead of a conventional laboratory flotation cell a newly designed flotation device called "Hallimond tube" was used (Figure 5). The operation and the modification of the Hallimond tube are explained in Appendix D. The Hallimond tube like a conventional flotation cell has provisions for the the removal of the floated part of the dispersion. A
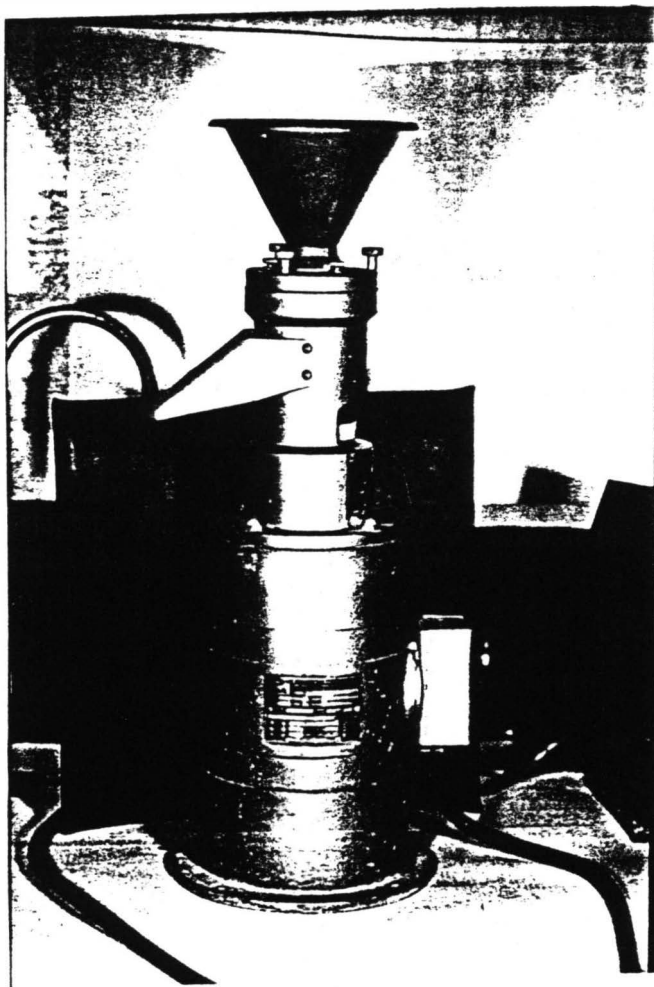
Figure 4. The Morehouse Mill (The Morehouse Industries, Inc. Model: A200)

sintered bottom glass tube acts as the main chamber for flotation which is executed by the controlled flow of air or nitrogen from the bottom. As the flotation is completed the floated portion can be separated and a comparitive study of the floated and non-floated ink dispersion may be made and flotation efficiency can be calculated.

Study of Zeta potential, pH and SurfactantConcentraton

Zeta potential measuring instrument, LASER ZEE[tm] Model 500 (Pen Kem Inc.), was used for this study. The Laser Zee[tm] Model 500 measures the zeta potential of colloidal particles by determining the rate at which these particles move in a known electric field. It is common to refer to this method as micro-electrophoresis as the particles are observed with a microscope.

The dispersion to be measured is placed in an electrophoresis chamber consisting of two electrode compartments and a connecting chamber. A voltage is applied between two electrodes, one located in each compartment. The charged particles respond to the applied voltage by moving toward one or the other electrode.
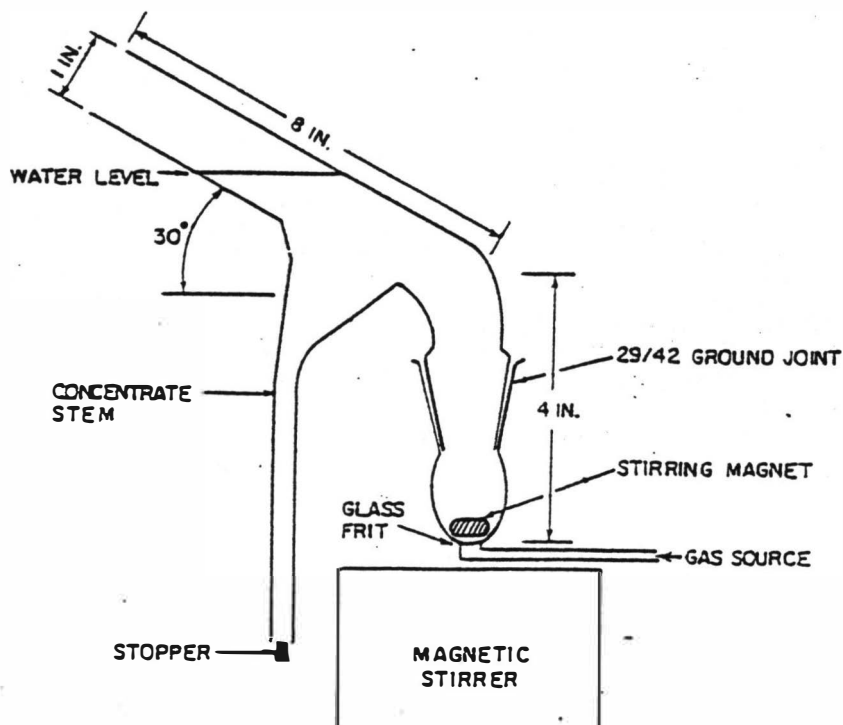
Figure 5. The Hallimond Tube Which Can Be Used for
Controlled Testing of Flotation Behavior (8).

The direction of movement is determined by the sign of the
charge and the speed of the particles is directly
proportional to the magnitude of the particle charge or
zeta potential. The instrument works on the principle of
determining the magnitude and sign of the colloid charge
by observing the speed and direction of the particle
movement under the influence of the applied field.
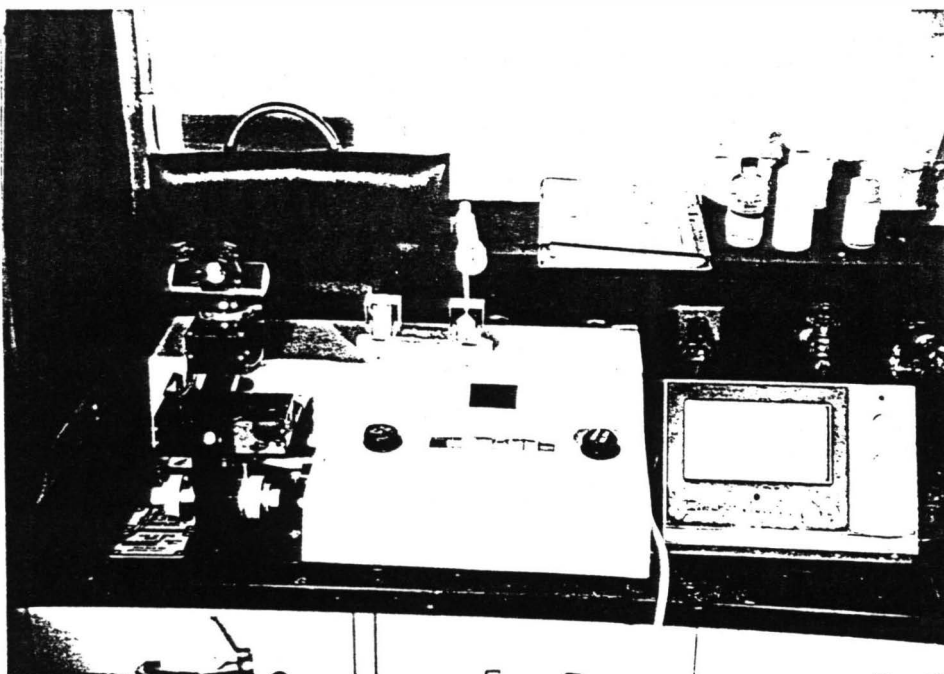
Figure 6. Zeta Potential and pH Measurement System

Initially, trial measurements of zeta potential were done with a standard colloidal silica solution. The instrument system for measuring zeta potential and pH is shown in Figure 6. An ink dispersion with particle size less than 50 μm was filled in the zeta potential cell. The readings were noted for as many as five samples. The reproducibility was good. The readings varied from -5 mV to +4 mV.

Pluronic L-10 surfactant, a non-ionic polyoxypropylene -polyoxyethylene block copolymer, manufactured by BASF, was used. The specifications are explained in detail in Appendix E. For the experimental series the samples were

taken from a solution conditioned for the experimental requirement. The pH and the surfactant concentration were varied at three levels of each variable.

A total of nine samples were used for pH and zeta potential measurement. A duplicate was also done for each sample to check the reproducibility.

Flotation Experiments

Several series of flotation experiments were done to find the optimum values of pH and surfactant concentration. First the pH values were maintained at 4.5, 6.0 and 7.5 for three levels of surfactant concentrations (49.3, 82.2 and 115.1 micromoles/liter) at room temperature with a regulated supply of nitrogen at 40 ml/min. The ranges of pH and surfactant concentrations were selected from the literature (Table 1). The experimental set-up is shown in Figure 7. The amount of nitrogen was set constant by trial and error for good froth formation with no jet aeration (see Appendix D). The same conditions were maintained for another series of experiments at 45°C (Figure 8).

After finding the effects of pH and surfactant concentration at different temperatures it was decided to study the effect of surfactant concentration at room temperature at a pH of 7.5. From this study it was possible

to find the range of surfactant concentration at which the flotation efficiency was maximum.

Another series of flotation experiments was designed with the range of values of pH and surfactant concentration obtained from the exploratory study. In this series the pH values were maintained at 7.0, 7.5 and 8.0 at three levels of surfactant concentrations 4.1, 8.2, 16.4 µM/L.
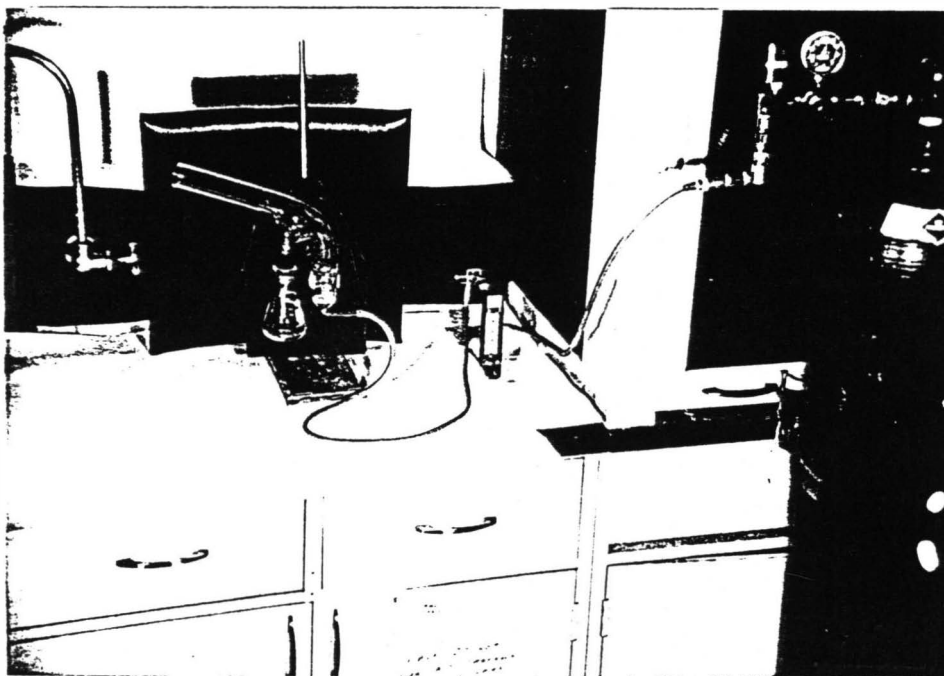
Figure 7. Experimental Set-up for Flotation at Room
        Temperature
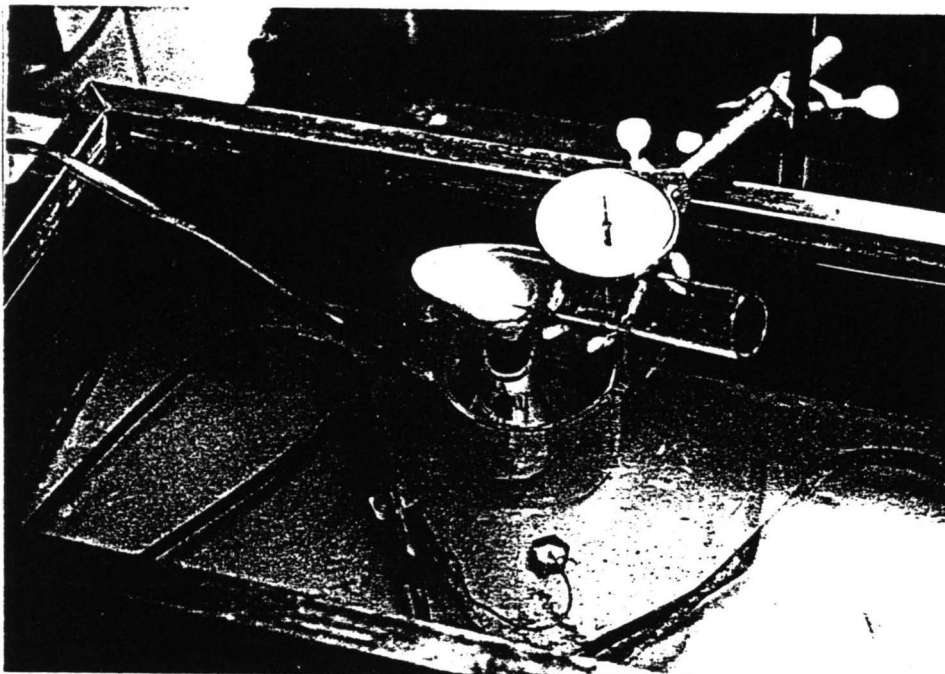
Figure 8. Experimental Set-up for Flotation at 45°C.

Data Analysis and Findings

The flotation efficiency was determined using the area of the floated and non-floated ink particles and is explained in detail in Appendix F. The data of the flotation efficiencies obtained for different flotation conditions were analyzed statistically using SAS response surface regression methodology (21). Graphs were plotted for the determination of the effects of pH and surfactant concentration on zeta potential and flotation efficiencies. Particle size distributions for ink dispersions before flotation and the flotated portion of the dispersion were analyzed for the best flotable size range.

CHAPTER V

PRESENTATION OF RESULTS AND DISCUSSION

Though the experimental data were analyzed statistically the results include the experimental limitations and the subjectivity of the equipment involved. A new method for determination of flotation efficiency using the image analysis technique was developed (Appendix F).

Determination of the Flotation Model

Three levels of pH (7.0, 7.5 and 8.0) at three levels of surfactant concentrations (4.1, 8.2 and 16.4 µM/L) were studied to get the optimum values of pH and surfactant concentration for maximum flotation efficiency. The data for the experiments are tabulated in Table 2. From Table 2 it is evident that at constant surfactant concentration the flotation efficiency increased as the pH increased from 7.0 to 7.5. But as the pH increased form 7.5 to 8.0 the flotation efficiency dropped.

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Table 2

The Effect of pH and Surfactant Concentration on
Flotation Efficiency

Surfactant Concentration µM/L	pH	% Efficiency
4.1	7.0	58.1
4.1	7.5	64.3
4.1	8.0	60.1
8.2	7.0	78.6
8.2	7.5	88.6
8.2	8.0	80.9
16.4	7.0	76.5
16.4	7.5	87.5
16.4	8.0	81.1

Similarly the flotation efficiency increased as the surfactant concentration increased from 4.1 to 8.2 µM/L. But as the surfactant concentration increased further the flotation efficiency dropped slightly. The increase and decrease in flotation efficiency for different conditions of pH and surfactant concentration showed that the values at maximum flotation efficiencies were close to the optimum values of the parameters. The dependent variable, flotation

efficiency, and the independent variables, pH and surfactant concentrations, were analyzed using polynomial response surface regression. The results of the analyses are given in Table 3, 4 and 5. A three dimensional representation of the model is shown in Figure 9. A two dimensional plot of Flotation efficiency plotted against pH at the three levels of surfactant concentration is shown in Figure 10.

Table 3 shows the data input to the computer for analysis. The column "Y" represents the flotation efficiency, the dependent variable. "X1" represents the pH values and "X2" represents the surfactant concentra- tion, the independent variables. Table 4 shows the results obtained from the computer analysis. The factor critical values of pH and surfactant concentration was determined as 7.6 and 12.2 μM/L respectively. The solution was maximum and the predicted value of the solution, the flotation efficiency, was 94.8%. The T-ratio and P values of the variables and the models are shown in Table 5 which is helpful in checking the strength of the statistical analyses.
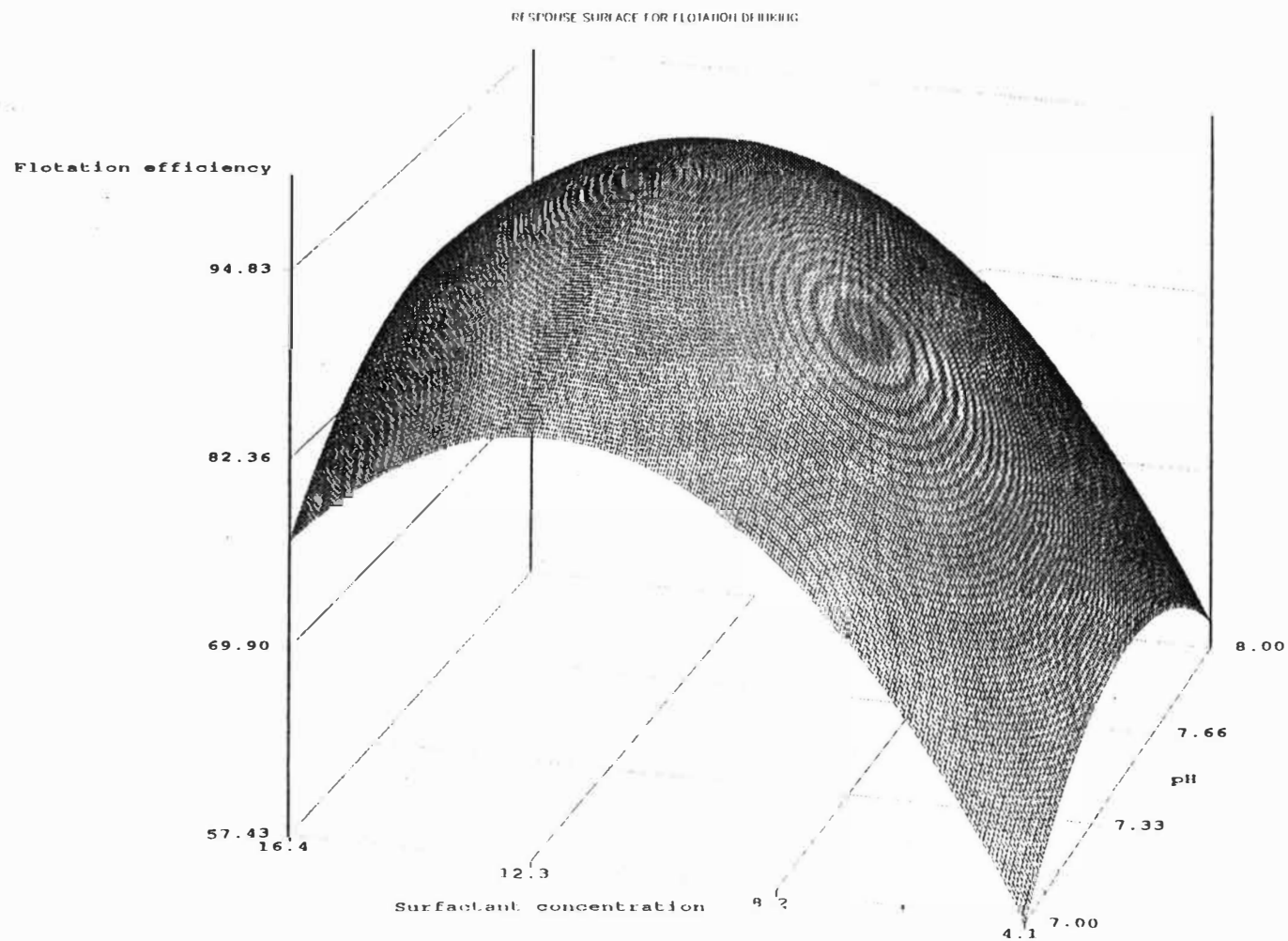
Figure 9. Three Dimensional Representation of
the Flotation Model
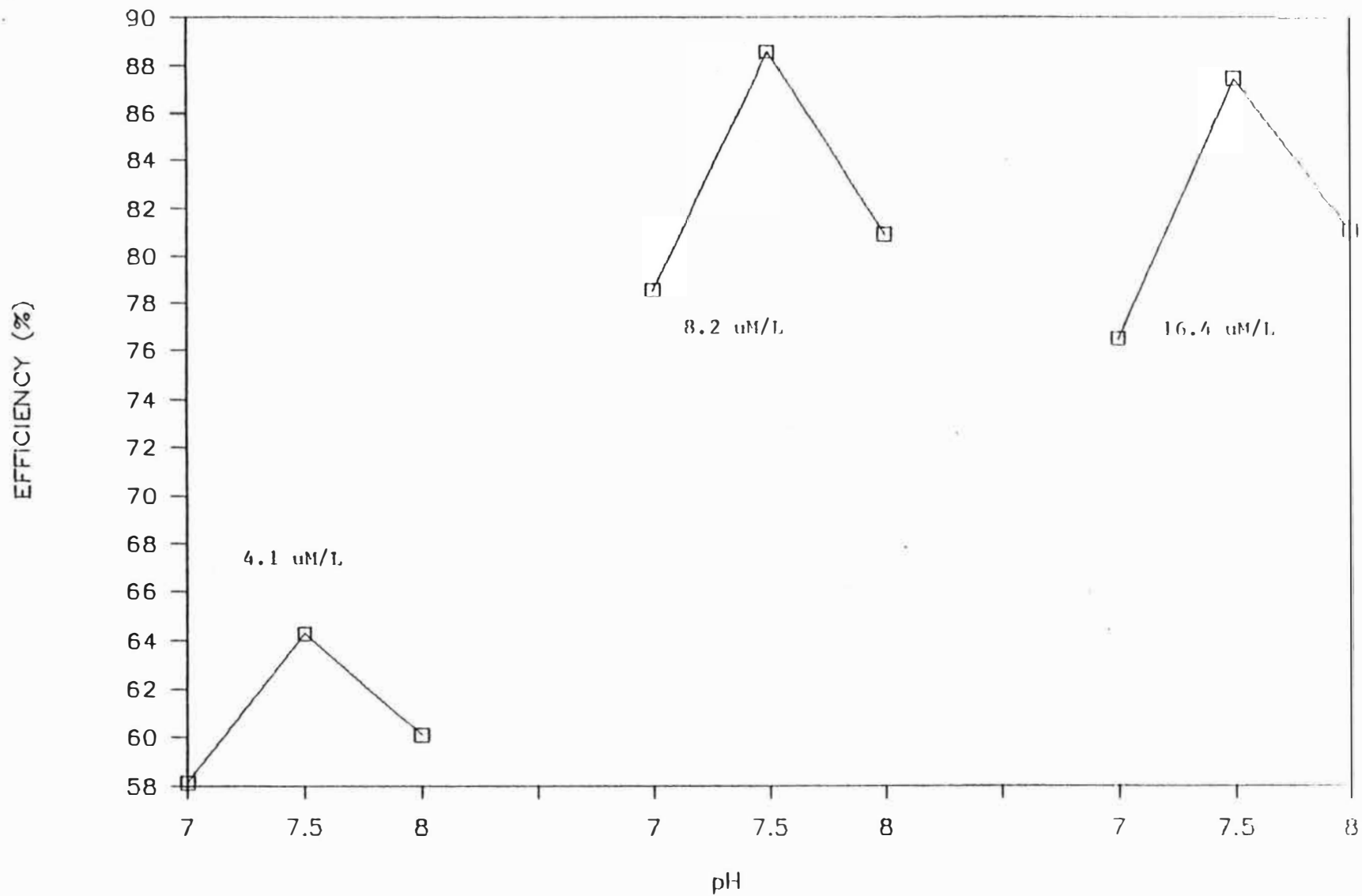
Figure 10 Effect of pH and Surfactant Concentration on Flotation Efficiency

Table 3

Data of the Experimental Results (Y=Flotation Effici-
ency, X1 = pH and X2 = Surfactant Concentration)

SAS

OBS	Y	X1	X2
1	58.10	7.0	4.1
2	64.32	7.5	4.1
3	60.10	8.0	4.1
4	78.60	7.0	8.2
5	88.60	7.5	8.2
6	80.90	8.0	8.2
7	76.50	7.0	16.4
8	87.50	7.5	16.4
9	81.10	8.0	16.4

Table 4

Predicted Value of Flotation Efficiency and the
Solution for Optimum Response

SOLUTION FOR OPTIMUM RESPONSE

FACTOR CRITICAL VALUE

X1	7.55836995	pH
X2	12.17704122	Surfconc(m.mole/K.liter)

PREDICTED VALUE AT OPTIMUM   94.82506

EIGENVALUES	EIGENVECTORS	
	X1	X2
-0.442978	0.00369777	0.9999932
-30.3604	0.9999932	-0.00369778

SOLUTION WAS A MAXIMUM

Table 5

Statistics of the Experimental Data

RESPONSE SURFACE FOR VARIABLE Y   Efficiency

         RESPONSE MEAN   75.08
         ROOT MSE   1.38174
         R-SQUARE   0.9945455
         COEF OF VARIATION   1.840357

REGRESSION	DF	TYPE I SS	R-SQUARE	F-RATIO	PROB
LINEAR	2	498.65549524	0.4749	130.59	0.0012
QUADRATIC	2	543.76157143	0.5178	142.41	0.0011
CROSSPRODUCT	1	1.92011905	0.0018	1.01	0.3898
TOTAL REGRESS	5	1044.33719	0.9945	109.40	0.0014

RESIDUAL	DF	SS	MEAN SQUARE	F-RATIO	PROB
LACK OF FIT	3	5.72761429	1.90920476	9999.990	1.0000
PURE ERROR	0	0	0		
TOTAL ERROR	3	5.72761429	1.90920476		

PARAMETER	DF	ESTIMATE	STD DEV	T-RATIO	PROB
INTERCEPT	1	-1684.99	219.93205595	-7.66	0.0046
X1	1	456.25000000	58.67108475	7.78	0.0044
X2	1	9.12595819	1.77073320	5.15	0.0142
X1*X1	1	-30.36	3.90815021	-7.77	0.0044
X2*X1	1	0.22125436	0.22062464	1.00	0.3898
X2*X2	1	-0.443387	0.02959448	-14.98	0.0006

FACTOR	DF	SS	MEAN SQUARE	F-RATIO	PROB	
X1	3	130.338	43.446	22.76	0.0145	pH
X2	3	915.9193	305.3064	159.91	0.0008	Surf

Forester (15) reported that the widely reported pH range to obtain maximum brightness for the normal emulsified inks was 8-10. He also concluded from his experimental results on UV-cured inks that for maximum brightness a pH near 6.0 was desirable. Larsson et al.(7) reported that above a pH of 9 the flotability of the ink particles decreased with increasing pH for mineral oil based inks.

Dinkfeld (5) obtained brightness and yield of 75.1 and 90.1, respectively at a pH 6.5 for the deinking of UV-cured inks. The optimum pH for maximum flotation was 7.6. The brightness method of determining the flotation efficiency has the limitation that many ink particles affect brightness more than a few large particles compared to that of the image analysis technique. Hence, the recommended values of pH found in this fundamental study using image analysis are different from the others.

Larsson et al. (7) showed that the flotability of the mineral oil based ink decreased as the surfactant concentration, alkyl polyglycol ether (nonionic surfactant), increased above 5 mg/L (0.05%).

Similar results were obtained for this work as the surfactant concentration was increased from 0.05% (8.2 μM/L). The surfactant concentration at which the

flotability of the ink decreased was lower (about 10%) compared to that of Larsson et al. (7). This may probably be due to the type of ink, type of surfactant and other factors involved in the flotation.

The response surface regression calculates the maximum value of the flotation efficiency by completing the response surface of the response variables for the flotation efficiencies obtained in the experiments. The optimum values of pH and surfactant concentration corresponding to the maximum flotation are also calculated by the computer.

The predicted polynomial response surface model for the flotation efficiency was

$$Y = -1684.99 + 456.25\ x1 + 9.12596\ x2 - 30.36\ x1^2 + 0.22125\ x1*x2 - 0.44339\ x2^2$$

where:

Y = flotation efficiency

x1 = pH

x2 = surfactant concentration

From the results of the analysis it is evident that the contribution of the crossproduct term of both the pH and the surfactant concentration was very low or negligible compared to that of the linear and the quadratic terms.

Effect of pH and Surfactant Concentration

The effect of pH on flotation efficiency and zeta potential was studied at three different levels of surfactant concentration.

The experimental results for zeta potential measurements are tabulated in Table 6 and plotted in Figure 11. At a surfactant concentration of 49.3 µM/L for a pH increase from 4.5 to 7.5 the zeta potential changed from -24.4 to -47.7. A change of zeta potential from -29 .8 to -50.3 was observed for an identical pH change at a surfactant concentration of 115.1 µM/L. The increase in zeta potential was probably due to the ionization of the carboxylic or hydroxyl groups at high pH. It was noticed that the increase in zeta potential due to pH was more than that due to surfactant concentration.

In otherwords, there was no significant change in zeta potential due to increase in surfactant concentration and the increase was mainly dependent on pH. At 45°C, the relationship was similar to that of room temperature except that the values were more negative than at room temperature.
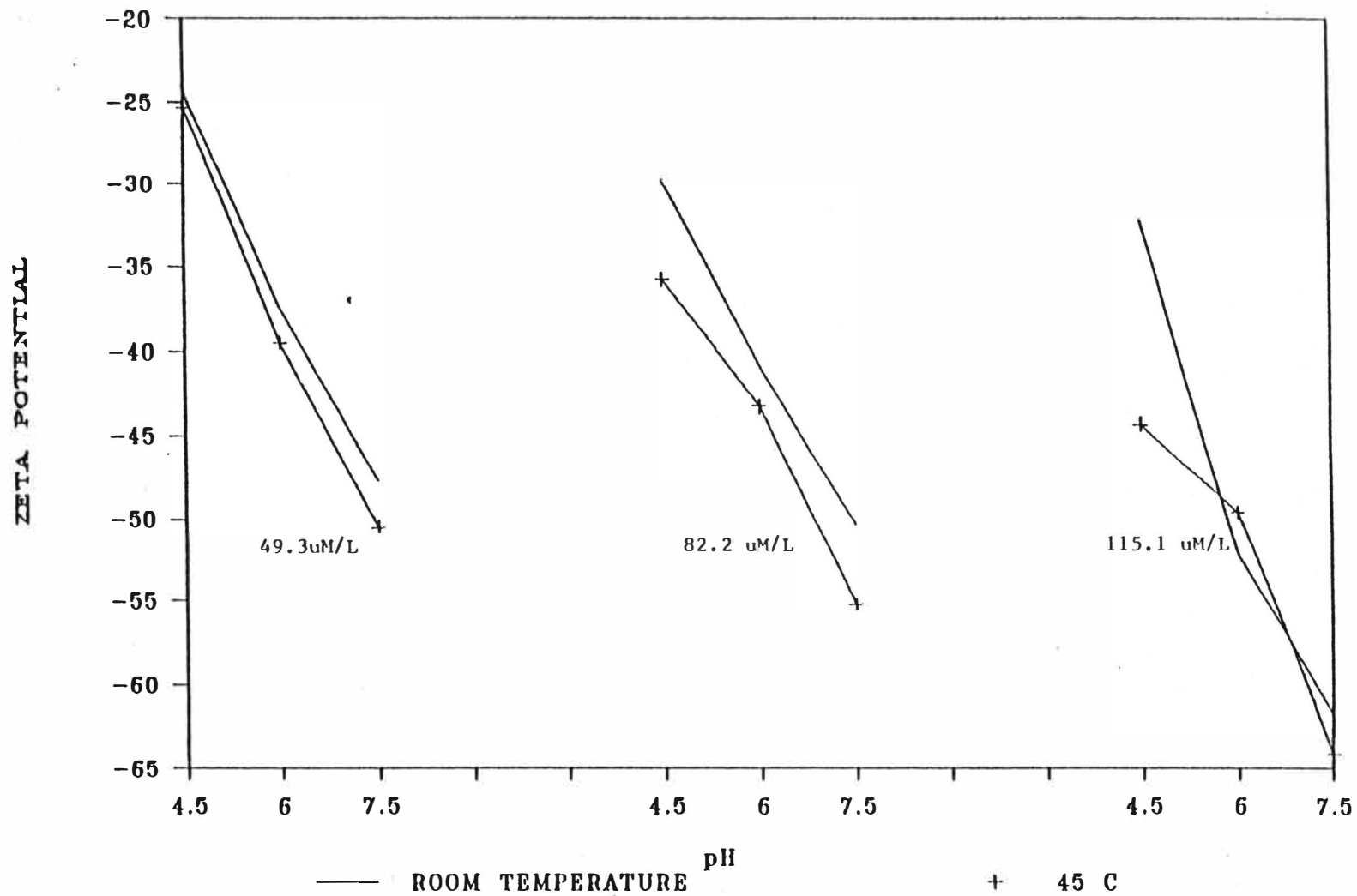
Figure 11. Relationship Between Zetapotential and pH for

Preliminary Experiments

Table 6

The Effect of pH and Surfactant Concentration on
Zeta Potential at Room Temperature

pH	Surfactant Conc µM/L	Zeta potential (mV) 20°C	45°C
4.5	49.3	-24.4	-25.3
6.0	49.3	-37.4	-39.5
7.5	49.3	-47.7	-50.5
4.5	82.2	-29.8	-35.8
6.0	82.2	-40.8	-43.2
7.5	82.2	-50.3	-55.2
4.5	115.1	-32.2	-44.3
6.0	115.1	-52.2	-49.6
7.5	115.1	-61.8	-64.2

These results also confirm the results of Pornpaitoonskul (4), who determined that the zeta potential increased as the pH increased. The increase in the zeta potential is favorable for dispersion in the pulping stage as the ink particles released from the fibers are more stable in water due to electrostatic repulsion.

The data for flotation efficiency are tabulated in Table 7 and plotted in Figure 12. It is evident from Table 7 that at room temperature, (20°C), the flotation efficiency increased as the pH increased from 4.5 to 7.5 at all the three concentration levels. The maximum flotation was obtained at 7.5 pH. This confirms closely the results of Dinkfeld (5) who studied a combination of surfactants for deinking of UV-cured inks at different conditions.

But at 45°C, the flotation efficiency values were high compared to those at room temperature at all levels of and surfactant concentration. This contradicts the results of Larsson et al.(7) who got a decrease in flotability with increase in temperature above 20°C. Although there were small variations in flotation efficiencies there was no significant increase with increase in pH similar to that at room temperature. Although this system employed a block co-polymer surfactant the effect of temperature could be explained by the concept of Kraft temperature (20).

Above the Kraft temperature the solubility of colloidal electrolytes increases very rapidly leading to the precipitation of solid soap rather than micelle formation (20). Block co-polymer surfactants do not form conventional micelles. Hence, a new mechanism of deinking might have occured in this study as the flotation
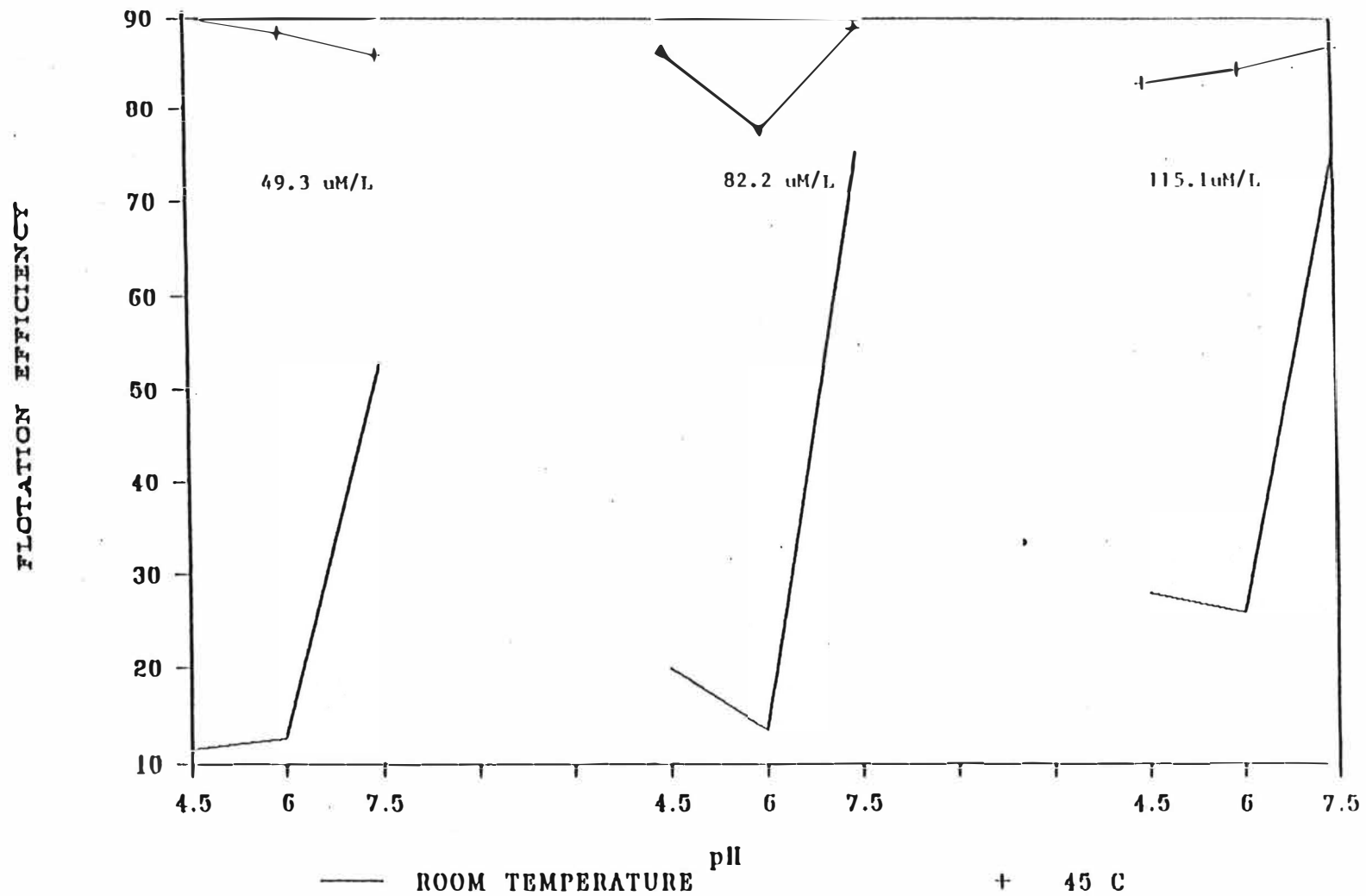
Figure 12· Effect of pH on Flotation Efficiency for Preliminary Experiments

efficiency was higher at higher temperature.

Table 7

The Effect of pH and Surfactant Concentration
on Flotation Efficiency

pH	Surf. conc. μM/L	Flotation efficiency 20°C	45°C
4.5	49.3	11.4%	89.95%
6.0	49.3	12.5%	88.4%
7.5	49.3	52.9%	86.0%
4.5	82.2	20.0%	86.3%
6.0	82.2	13.5%	77.7%
7.5	82.2	75.4%	88.9%
4.5	115.1	28.3%	83.0%
6.0	115.1	26.2%	84.5%
7.5	115.1	75.3%	86.9%

It is evident from this study that the flotation
efficiency increased very rapidly at a pH of 7.5. The pH
range was selected around 7.5 for the determination of the
flotation model. The increase in flotation efficiency due

to surfactant concentration was not very significant compared to that of pH. Hence, the study of the effect of surfactant concentration on flotation efficiency was done.

Effect of Surfactant Concentration

The Pluronic L10 surfactant used belongs to the polyglycol nonionic family. Two series of flotation experiments were done at room temperature for the ink and water as well as the ink, fiber and water system. The experimental results are tabulated in Tables 8, 9 and 10. From Table 8 it is evident that for the ink and water system the flotation efficiency increased as the surfactant concentration increased from 0 to 8.2 μM/L. As the surfactant concentration increased further up to 164.4 μM/L the efficiency dropped and remained practically constant at this lower level. The flotation efficiencies are plotted against the surfactant concentration in Figure 12.

These results can be explained using the following proposed mechanism. The Critical Micelle Concentration (cmc) of the surfactant is the concentration of the surfactant at which the detergent ability rises and remains practically constant thereafter. The Pluronic L10 surfactant produces no micelles. Though the phenomena of

Table 8

Ink and Water System

Effect of Surfactant Concentrations on Flotation
Efficiency at a pH pf 7.5 at Room Temperature

Surfactant Concentration	Efficiency	
μM/L	Run I	Run II
0	0%	0%
4.1	58.9%	61.6%
8.2	82.9%	82.1%
16.4	74.6%	72.2%
49.3	51.2%	49.4%
82.2	45.0%	41.7%
115.1	49.6%	47.3%
148.0	47.1%	46.0%
164.4	56.6%	50.9%

these experiments can not be compared because of a major difference in the detergent charecter between the conventional and the Pluronic L10, the effect of surfactant concentration on flotation efficiency may be due to the above mentioned phenomena.

From Table 9 it can be understood that the ink, fiber and water system behavior was similar to that of the ink and water system.

Table 9

Ink, Water and Fiber System

Effect of Surfactant Concentration on Flotation
Efficiency at 7.5 pH at Room Temperature

Surfactant Concentration µM/L	Efficiency Run I	Run II
0	0%	0%
4.1	64.3%	69.1%
8.2	88.6%	88.4%
16.4	87.5%	86.9%
49.3	41.4%	50.1%
82.2	50.5%	50.4%
115.1	46.2%	45.9%
148.0	52.4%	46.3%
164.4	66.3%	60.2%

The flotation efficiency increased and decreased at
the same range of surfactant concentration as for the ink
and water system (Figure 13). For the ink and fiber system
the floated portion of the dispersion contained fibers. As
the amount of fibers was too low and difficult to measure
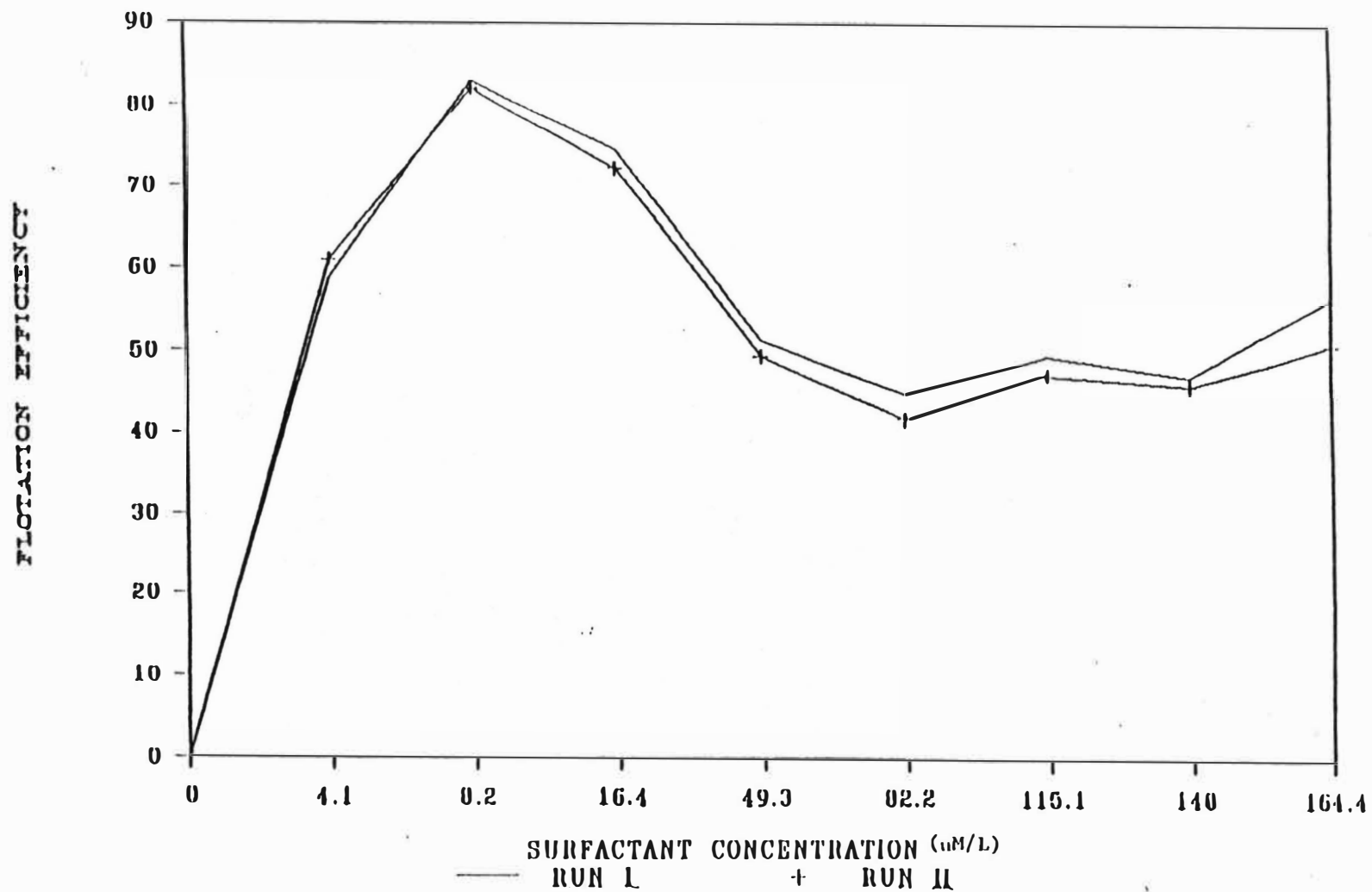
Figure 13. Effect of Surfactant Concentration on Flotation Efficiency for Ink and Water System
(At Room Temperature and at 7.5 pH)

by weight the exact fiber loss was not determined. Also the amount of ink in either portion of the dispersion was not known for the quantification of the fiber loss. Duplicate experiments reproduced the results with approximately 1-5% variations in flotation efficiencies. From these results the range of the surfactant concentration for maximum flotation efficiency was determined to be around 8.2 μM/L. From these results with the combination of the results of the study of the effect of pH and surfactant concentration the experiments for the determination of the flotation model were designed and performed.

The zeta potentials of the dispersions for flotation were measured at different surfactant concentrations and pH's. The results are tabulated in Table 10. It is evident from the data that the zeta potential increased as the surfactant concentration increased. From Table 6 it was observed that the increase in zeta potential with increase in surfactant concentration was relatively low compared to that of increase of the pH. A similar relationship was observed for these experiments at constant pH of 7.5.

Table 10

Effect of Surfactant Concentrations on Zeta Potential at
Room Temperature at 7.5 pH.

Surfactant Concentration µM/L	Zeta potential (mV)	
	w/o fiber	with fiber
4.1	-16.7	-24.5
8.2	-29.4	-33.9
16.4	-39.6	-41.7
49.3	-47.7	-54.0
82.2	-50.3	-63.5
115.1	-61.8	-72.4
148.0	-65.1	-80.0
164.4	-71.3	-88.6

The values of the zeta potential were observed to be higher
for the ink, fiber and water system compared to the ink and
water system. The graphic representation of these results
is shown in Figure 14 and 15.
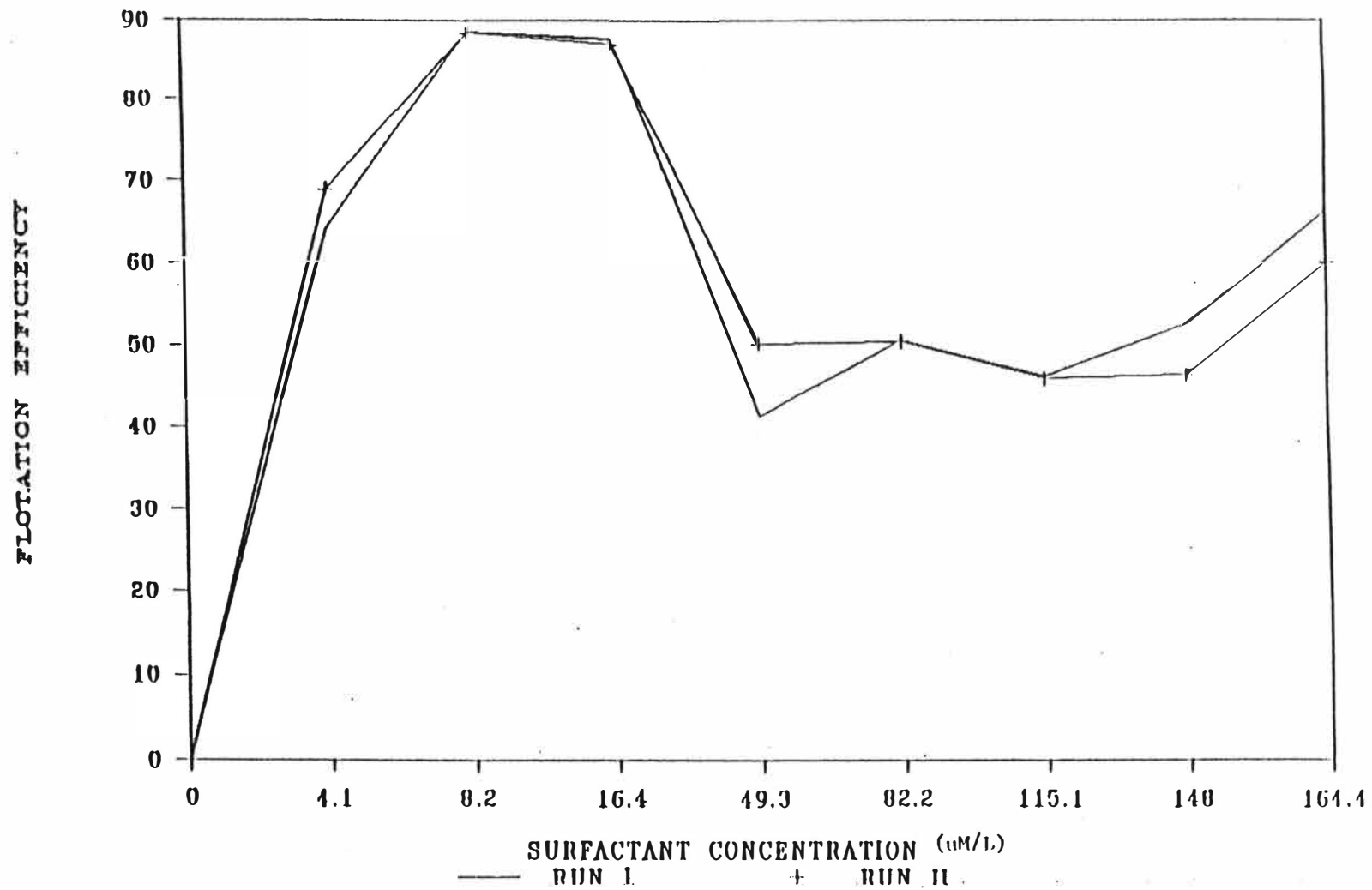
Figure 14. Effect of Surfactant Concentration on Flotation Efficiency for Ink, Fiber and Water System (At Room Temperature and at 7.5 pH)
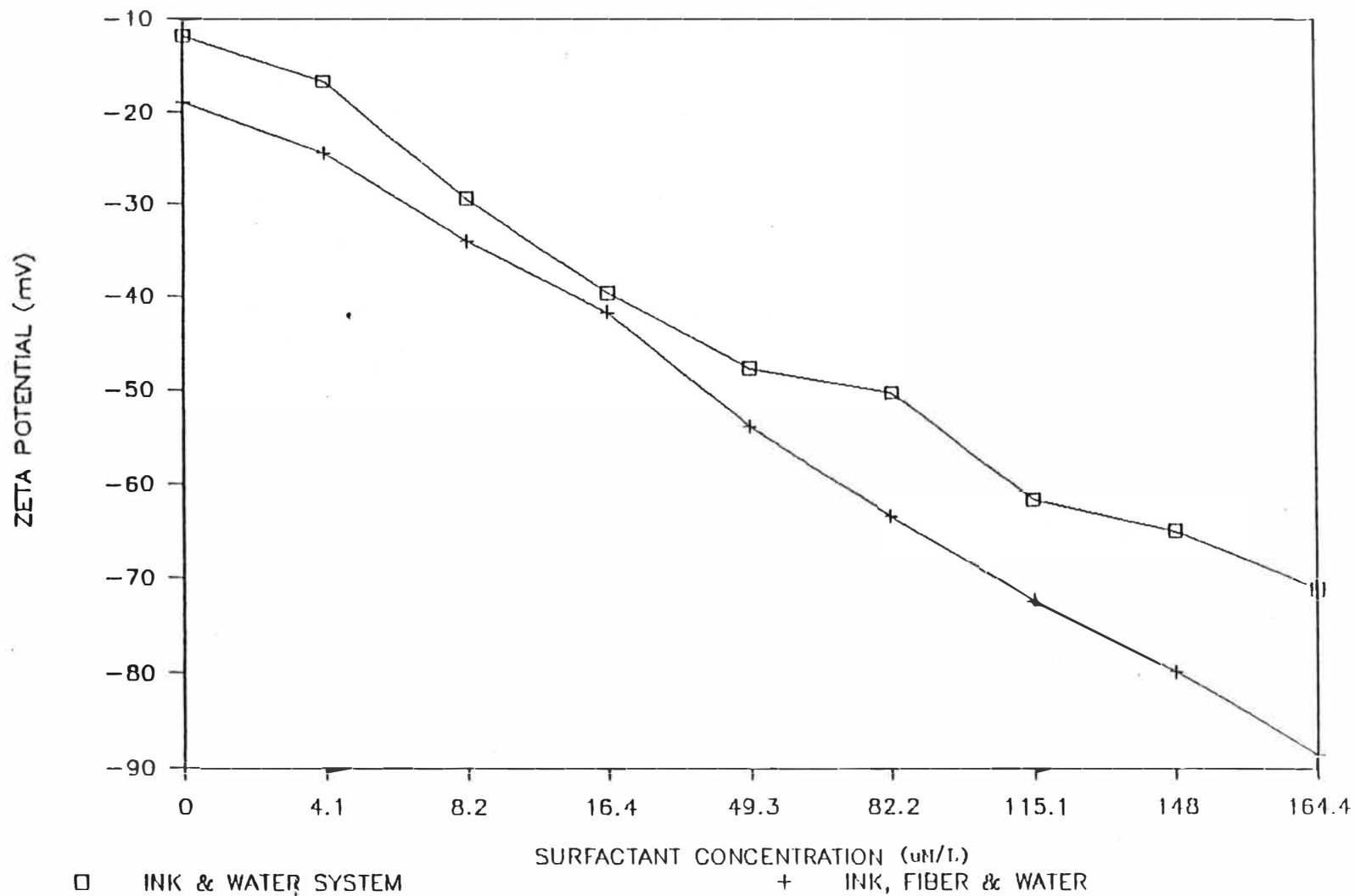
Figure 15, Effect of Surfactant Concentration on Zetapotential for Both the Systems
(At Room Temperature and at 7.5 pH)

Study of the Ink Particles

The Image Analyzer output for the particle size distribution of the samples before and after flotation was used. Though there was no common bin size for all the distribution analyses, an approximate calculation of the percentages of the particles for the corresponding mid-points of the class intervals yielded a relationship between the size ranges of the particles for the before- and after-floation samples. From Tables 11 and 12 it can be seen that the ink particles floated in the range of 17 μm are approximately proportional to the amount of ink particles of the same size range before flotation. Ratio of the ink particles in the range of 25-40 μm are approximately 60% more than the ratio of the corresponding size before flotation.

Larsson et al. (7) reported that the flotation rate increased with particle diameter in the range of 1-20 μm

Table 11

Percentage of Ink Particles of Different Sizes
for Sample Before Flotation

Mean Diameter (μm)	Particle Count	Percentage
17	334	32.75
25	152	14.90
33	99	9.70
41	75	7.35
44	74	7.25
58	51	5.00
66	44	4.30
74	40	3.90
82	28	2.75
90	22	2.16
98	25	2.45
107	15	1.47
115	10	0.98
123	16	1.57
131	18	1.76
139	8	0.78

Table 12

Percentage of Ink Particles of Different Sizes in the
Floated Part of the Dispersion

Mean Diameter (µm)	Particle Count	Percentage
17	224	29.44
26	146	19.19
35	138	18.13
44.5	89	11.69
53.5	47	6.17
62.5	39	5.13
72	37	3.55
81.5	18	2.37
90.5	12	1.58
100	11	1.44
108.5	2	0.26
117.5	3	0.39
127	0	0

for the mineral oil-based ink. Such a low size range of the
particles could be obtained as the ink was based on mineral
oil unlike UV-cured ink, which is highly cross-linked. Most
of the other literature discusses the brightness of the
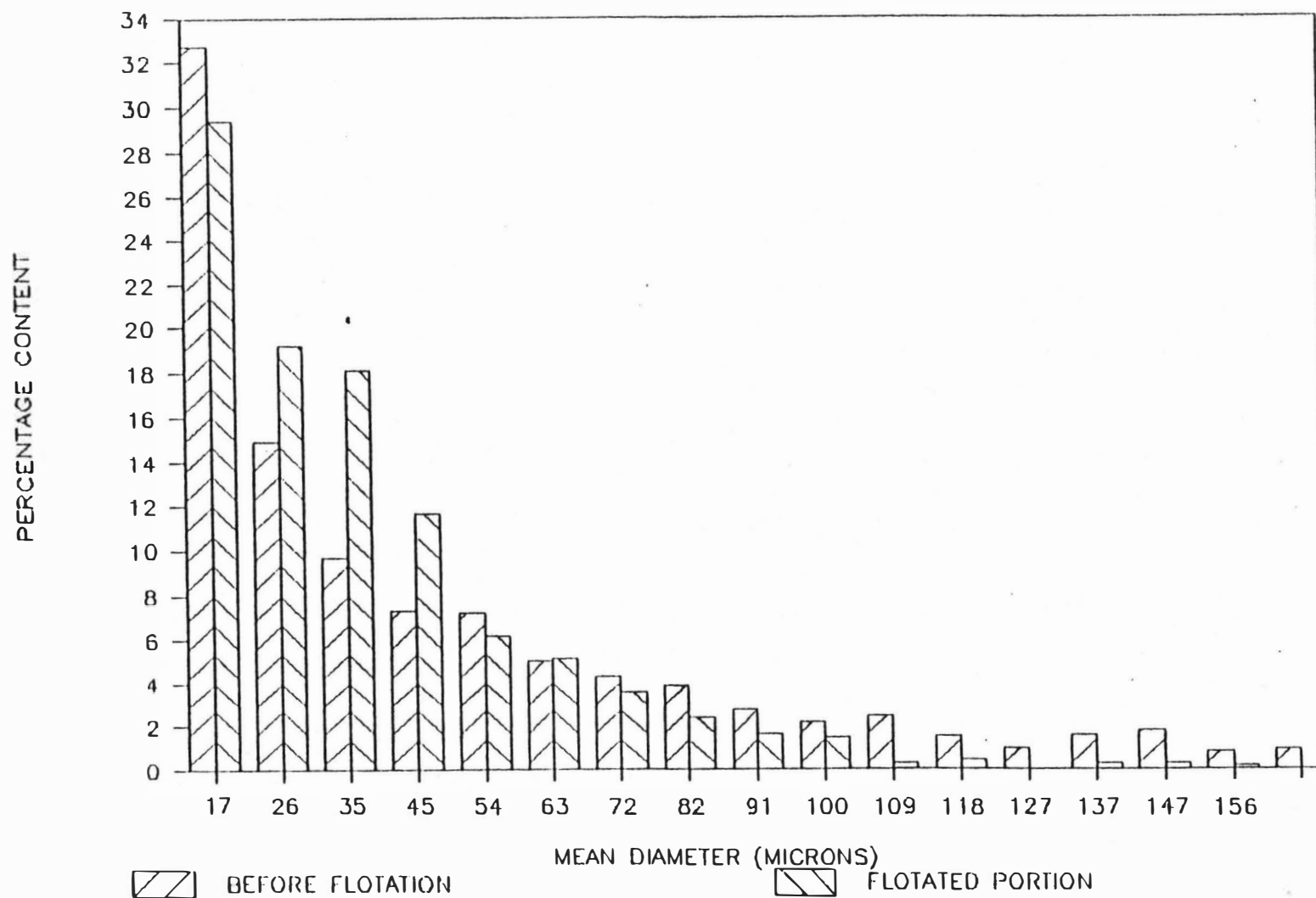
Figure 16. Percentage Size Distribution of the Ink Particles in the Floated and Before-Floated Portions.

deinked pulp rather than the area of the ink particles.

From literature (3), the size of the UV-ink particles produced in the pulpers of the recycling mills are normally in the range of 50-100 μm.

INK PARTICLE DISTRIBUTION

a) Ink Particle Distribution in Pulper

100% News

b) Ink Removal Efficiencies by Various Operations
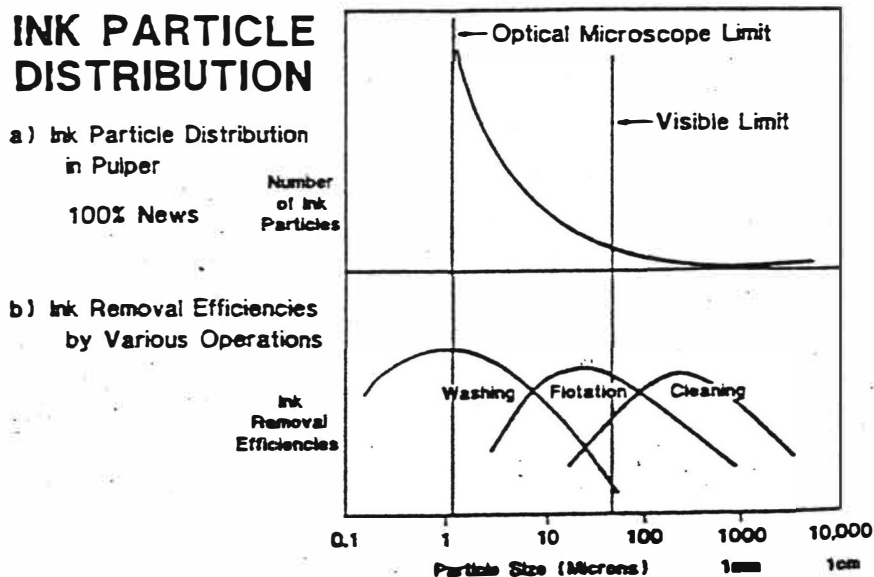
Figure 17. Ink Particle Distribution and Removal Efficiencies (25).

Inspite of studying the deinkability of UV-ink at various deinkinig conditions with several receipes Vanderhoff (3), Dinkfeld (5) and Pornpaitoonsakul (4) concluded that the mechanism of the behavior of the UV-ink particles needed to be studied and the optimum values of the variables were also urged to be determined.

Figure 16 shows that as much as 79% of the floated particles falls within the size range of 12-45 μm

approximately. From Figure 17, flotation process in general seems to be efficient in the ink particles size range of 10-50 μm. So far there is no study done with UV-ink particles, which is believed to be different in behavior than the conventional ink.

Though the UV-ink produced comparatively larger particles than present in conventional inks, it is evident from this study that the UV-ink particles in the range of 26-45 μm floated most effectively. This leads to a better understanding of the mechanism of the UV-ink particles during flotation.

Hence a new technique of dispersion for UV-ink is proposed.

CHAPTER VI

SUMMARY OF RESULTS

From the analysis of the results for the flotation experiments to find the optimum values of pH and surfactant concentration it was found that the maximum response of flotation efficiency was 94.8%. The experiments were done at room temperature. The corresponding values of pH and surfactant concentrations were computed and determined to be 7.56 and 12.18 μM/L, respectively.

Prior to the determination of the actual range of the parameters, the flotation experiments led to different types of results. For the ink and water system at room temperature and at a pH of 7.5 the flotation efficiency increased as the surfactant concentration increased to 8.2 μM/L. As the surfactant concentration increased further the flotation efficiency decreased considerably and remained practically constant up to 164.4 μM/L. Similar results were obtained for the flotation experiments for the ink, fiber and water system under the same conditions.

The zeta potential of the flotation dispersion

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increased negatively as the pH increased. The increase in zeta potential negativity was smaller in the case of increase in surfactant concentration.

For experiments at room temperature the flotation efficiency increased as the pH was increased to 7.5 at three levels of surfactant concentration. For experiments at 45°C the flotation efficiency was higher and practically constant for all the levels of pH and surfactant concentrations. The zeta potential increased negatively as the pH increased. This verifies the results of Pornpaitoonskul (4) who also studied the zeta potential of UV-cured ink.

Ink particles in the size range of 26-45 µm were found most favorable for flotation. This verified the conventional chart for flotation efficiency for various ink particle size distribution.

CHAPTER VII

CONCLUSIONS

There are six major conclusions.

1. The optimum values of pH and surfactant concentration were 7.56 and 12.18 µM/L respectively for the maximum response of 94.83% flotation efficiency, at room temperature.

2. At room temperature the flotation efficiency of the ink and water system increased as the surfactant concentration increased to 8.2 m.mole/K.liter and then decreased to a lower constant value.

Similar results were obtained for the ink, fiber and water system indicating that the presence of fiber did not alter significantly the ink flotation mechanism.

3. The flotation efficiency increased as the pH increased at constant surfactant concentration at room temperature and at 45°C remained practically constant at a higher value with small variations. The zeta potential of the dispersion increased negatively as the pH and the surfactant increases.

Though there was an increase in the zeta potential due to the increase in surfactant concentration the increase was smaller compared to that of the pH.

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4. The Image analysis technique was developed on a reliable means for the determination of flotation efficiency.

5. Polynomial response surface methodology was a suitable analysis method for determining the optimum values of the parameters, pH and surfactant concentration, for the maximum response of the dependent variable, flotation efficiency.

6. Ink particles in the size range of 26-45 μm diameter were most suitable size range for best flotation.

CHAPTER VIII

SUGGESTIONS FOR FURTHER STUDY

Because most of the limitations in pursuing this study were corrected, further research in this area can be done without any significant exploration of the equipment, procedures and analysis techniques.

Though this research gives significant contribution to the understanding of flotation deinking, the following suggestions for further research are offered.

1. Different types of surfactants should be investigated. The optimum range of surfactant concentration should be determined for each surfactant.

2. Instead of studying two variables (pH and surfactant concentration) at a time, more variables (time, temperature, air quantity, hardness, etc.) should be studied simultaneously. The range of optimum values of the parameters should be determined in exploratory study similar to this research. After finding the range of the variables a completely randomized block design should be conducted at three or four levels of the values of the variables in the optimum range. There shall be reasonable

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missing cells.

After getting the results of the flotation efficiency for the five or six independent variables the data can be analyzed using the polynomial response surface methodology. A diagrammatical expression of the result may not be possible but the optimum values may be calculated for the critical conditions similar to this research result.

3. The number of ink particles floated should be related to the time of flotation so the kinetics of the process can be investigated.

4. Waste paper printed with the type of ink of this study should be researched.

5. Experiments should be performed to find the effect of the temperature on flotation efficiency.

6. An extensive study of the generation of smaller UV-ink particles in the range of 12-50 µm during pulping is necessary. Flotation experiments on these particles is needed.

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APPENDICES

Appendix A

Preparation of UV-Cured Ink Dispersion

Preparation of UV-Cured Ink Dispersion

UV-ink was obtained from Norcote Chemical Company in the cured form on plastic sheets. The ink film was peeled off from the plastic sheets and one gram was mixed with 1000 ml of distilled water and dispersed in a Waring blender for five minutes. Though the particle size was reduced it were still too large (50-100 μm was the desired range). Aliquots were taken periodically for another series of dispersions at 120, 150, 180, 210, and 240 minutes. All the samples were diluted to a constant volume (500 ml) and a portion of the contents was used for monolayer Millipore sample pad preparation as explained in Appendix B.

All five samples made from the aliquots were analyzed for size distribution of the ink particles. A sample of the image analysis output for 240 minutes dispersion in the blender is shown in Figure 18. No improvement was observed and a ball mill used for the same purpose was also not applicable. An attempt with a laboratory homogenizer was dropped as Morehouse mill gave good results.

The Morehouse mill, (Figure 4), has a circular stationary grinding stone and a rotary grinding stone attached to the drive shaft of an electric motor. The clearance between the grinding stones can be adjusted with

Area	Log Dimension	Equiv Diam.

Field 1

Count:	275	275	275
Min:	25987.7	210.54	214.06
Max:	4.5784E+05	1473.3	771.80
Total:	2.8780E+07	1.0739E+05	95441.7
Mean:	1.0469E+05	390.52	347.06
StDev:	77007.8	193.61	112.55

DISTRIBUTION ANALYSIS on Equiv Diam.

UnderSize : 0
OverSize : 0

BIN	START	END	COUNT	BIN	START	END	COUNT
1	214.06	232.05	77	17	501.92	519.92	0
2	232.05	250.04	0	18	519.92	537.91	8
3	250.04	268.03	0	19	537.91	555.90	0
4	268.03	286.02	0	20	555.90	573.89	5
5	286.02	304.02	59	21	573.89	591.88	0
6	304.02	322.01	0	22	591.88	609.87	1
7	322.01	340.00	0	23	609.87	627.87	0
8	340.00	357.99	0	24	627.87	645.86	1
9	357.99	375.98	46	25	645.86	663.85	0
10	375.98	393.97	0	26	663.85	681.84	2
11	393.97	411.97	0	27	681.84	699.83	0
12	411.97	429.96	55	28	699.83	717.82	1
13	429.96	447.95	0	29	717.82	735.82	0
14	447.95	465.94	0	30	735.82	753.81	0
15	465.94	483.93	18	31	753.81	771.80	0
16	483.93	501.92	0	32	771.80	789.79	2

Frequency Histogram -

Linear Distribution

Equiv Diam.

Offset : 214.06
Size : 17.992
Range : 789.79
Under : 0
Over : 0

Cal. : μm
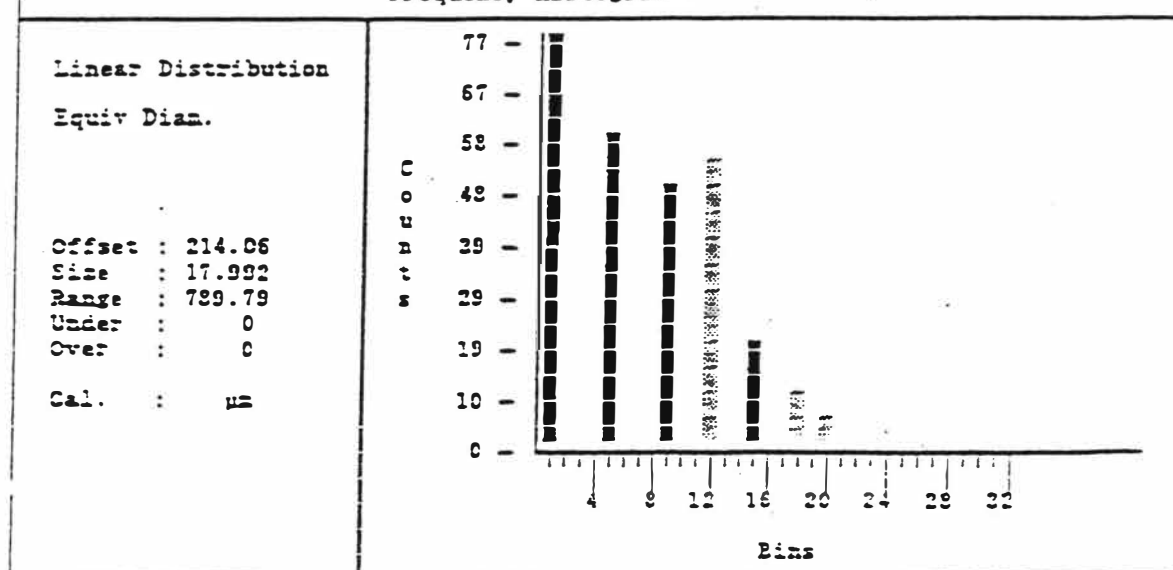
Figure 18. Size Distribution of UV-Ink Particles After 240
Minutes of Dispersion in the Waring Blender

a wheel.

One gram of UV-cured ink was dispersed in a Waring blender with distilled water to reduce the overall size of the particles and then ground in the Morehouse mill. No load was given between the grinding stones and the contents after one pass were passed ten times so that the particles could be broken down further. The accepts had no contaminants and the Morehouse mill was judged convenient for sample preparation.

Aliquots were taken at third, fifth and tenth passes. Millipore samples, prepared as mentioned before, were analyzed in the Image Analyzer. The data output from the Image Analyzer together with the size distribution for the tenth pass is shown in Figure 19.

The minimum size of the ink particle was 35.6 μm and the maximum number of ink particles were in the lower diameter range. The overall size of the ink particles reduced as the number of passes increased. Further reduction in particle size was prefered and a similar trial was done by increasing the load between the grinding stones. The position of the wheel was marked at load and an aliquot was taken after five passes. The sample analysis gave the size distribution shown in figure 20. The minimum size of the particles obtained was 15.28 μm. The maximum

OMNICON 3600 MEASUREMENT REPORT
8:15 A.M. on Thurs., Feb. 16, 1989
Calibration : 1x 9.9777E+02 μm²/pp
SAMPLE NAME : 2/15/89 sample +10

DISTRIBUTION ANALYSIS on Equiv Diam.
UnderSize : 0
OverSize : 0

BIN	START	END	COUNT	BIN	START	END	COUNT
1	35.643	46.386	17	17	207.54	218.28	2
2	46.386	57.130	12	18	218.29	229.03	3
3	57.130	67.873	6	19	229.03	239.77	1
4	67.873	78.617	11	20	239.77	250.51	0
5	78.617	89.360	14	21	250.51	261.26	1
6	89.360	100.10	4	22	261.26	272.00	2
7	100.10	110.85	9	23	272.00	282.74	2
8	110.85	121.59	6	24	282.74	293.49	0
9	121.59	132.33	5	25	293.49	304.23	0
10	132.33	143.08	11	26	304.23	314.97	0
11	143.08	153.82	8	27	314.97	325.72	0
12	153.82	164.56	7	28	325.72	336.46	0
13	164.56	175.31	3	29	336.46	347.20	0
14	175.31	186.05	3	30	347.20	357.95	0
15	186.05	196.79	2	31	357.95	368.69	0
16	196.79	207.54	2	32	368.69	379.43	1

Figure 19. Size Distribution of UV-Ink Particles After Ten Passes Through Morehouse Mill with no Load.

	Equiv Diam.	Field Area	Percent Area	Fld Perimeter	Feature Count

Field 1

Count:	125	1	1	1	1
Min:	15.280	2.8460E+05	1.0537	21431.4	125.00
Max:	151.27	2.8460E+05	1.0537	21431.4	125.00
Total:	5490.8	2.8460E+05	1.0537	21431.4	125.00
Mean:	43.926	2.8460E+05	1.0537	21431.4	125.00
StDev:	31.290	0.0	0.0	0.0	0.0

DISTRIBUTION ANALYSIS on Equiv Diam.
UnderSize : 0
OverSize : 0

BIN	START	END	COUNT	BIN	START	END	COUNT
1	15.280	19.980	24	17	90.483	95.183	0
2	19.980	24.680	14	18	95.183	99.883	2
3	24.680	29.381	12	19	99.883	104.58	0
4	29.381	34.081	5	20	104.58	109.28	1
5	34.081	38.781	15	21	109.28	113.98	0
6	38.781	43.481	15	22	113.98	118.68	2
7	43.481	48.181	4	23	118.68	123.38	0
8	48.181	52.882	1	24	123.38	128.08	2
9	52.882	57.582	6	25	128.08	132.78	0
10	57.582	62.282	4	26	132.78	137.48	1
11	62.282	66.982	5	27	137.48	142.19	0
12	66.982	71.682	2	28	142.19	146.89	2
13	71.682	76.382	2	29	146.89	151.59	1
14	76.382	81.083	3	30	151.59	156.29	0
15	81.083	85.783	0	31	156.29	160.99	0
16	85.783	90.483	2	32	160.99	165.69	0
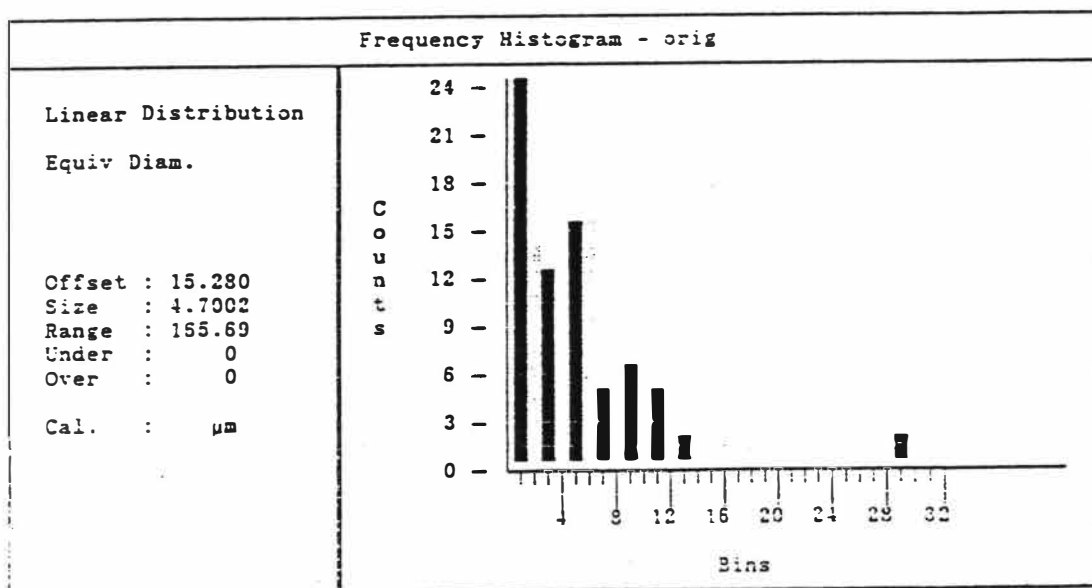
Figure 20. Distribution of UV-Ink Particles After Five Passes Through Morehouse Mill With Load.

number of ink particles was obtained in the smaller size region. The size could be reduced to 12.83 μm by increasing the load further.

For the experiments in this research the minimum size was maintained at 12.83 μm by keeping the condition of the Morehouse Mill constant. A fresh batch of the ink dispersion containing one gram of UV-cured ink in 1000 ml of distilled water was made for each series of experiments. Thus, the amount of ink used for every experiment was approximately equal for a constant volume of the dispersion.

Appendix B

Monolayer Millipore Membrane Sample Preparation

Monolayer Millipore Membrane Sample Preparation

Calculation of ink weight per sample

1 gm of UV-ink was dispersed (see Appendix A) in 1000 ml of distilled water and stirred for 20 min.with a magnetic stirrer. About 100 ml of this solution was diluted to 1000 ml with distilled water and 90 ml of this dispersion was used for flotation. The floated and non-floated part were diluted and equally divided into three parts. Hence the original dispersion of 90 ml for flotation was divided into three parts for sample filter.

Instead of a mass check at the ink particles before and after flotation, parameters like total ink area and number of ink particles can be used to check the balanced operation of flotation. The total area of the ink particles before flotation should be equal to the sum of the total areas of the ink particles in the floated and non-floated portions.

$$\text{Amount of ink on one sample pad} = \frac{1 \times 100 \times 90}{1000 \times 1000 \times 3} \quad \text{gm}$$

$$= 0.003 \text{ gm}$$

The amount of ink in the floated and non-floated part varied depending upon the efficiency of flotation.

Monolayer Sample Preparation

The monolayer Millipore samples were prepared with a procedure similar to that of Peel, J.D., and Nanda, A.K., (24). A representative sample of 90 ml was taken from a dispersion of 1000 ml containing 0.1 gm of UV-ink, after stirring the contents for 20 min, and was divided equally into three parts of 30 ml each. The Millipore membrane was marked and held under vacuum after wetting in the filter holder to avoid the formation of air bubbles in between the membrane and disc. The vacuum was adjusted so that no impression of the disc was formed on the membrane filter and also to avoid the swirling of the contents under filtration. Each part of the sample was poured without any splashing outside the holder and a monolayer filter was made without any significant overlapping of the ink particles. As soon as the filtration was complete the filters were removed and placed in a specially made pulp sheet holder so that the samples were not exposed to air. A fixative (Krylon No:1303 Crystal clear Acrylic spray coating) was sprayed on the ink particles so that the ink particles were not disturbed during the analysis in the image analyzer. For a particular experiment a total of six sample pads were made from both the floated and non-floated part. Only one set of the original sample was made

for each series. As far as the pulp and ink system was concerned, the floated and non-floated parts were diluted and divided into twelve parts and the total area was back-calculated for the floated, non-floated and before-floated portions.

Appendix C

Image Analysis Technique

Image Analysis Technique

The Image analysis system used was an Omnicon 3600, manufactured by Artek Systems Corporation.

Image Analysis Technique Used For the Analysis

The image analysis system was turned on and allowed to warm up for 30 minutes. The camera and the microscope, figure 19, were set up so that the object stage is in the range of the focal length of the lens in the microscope.

The lighting was adjusted to the darkened and lightened setting with the help of the buttons in the computer (figure 21). This was done by closing the aperture of the microscope completely and pressing the "set up if darkened" button for darkening and opening the aperture completely, with the special high intensity light kept close to the stage so that the stage was very bright (figure 22) to have a combination of angled incident and transmitted light. The magnification of the microscope was set at 16.

After setting the lighting system the cursor was moved to image functions and selected. Image utilities of the image functions was selected to choose detection color.

Figure 21.   The Image Analysis System

Green was chosen out of green and white color options. This make the pixels "I" of interest as described above to have green color.

As the interest was to store the results in the disk as well as to print, the results options of the main menu was selected to choose both the printer and disk. A file name was specified to the results being stored.

Calibration was done by the sequence as follows. The calibration scale printed on a glass plate was kept under the microscope and focused to give a sharp image of a suitable circle which could occupy a maximum area of the frame. The diameter of the circle chosen was 2.5 mm or 2500 microns.
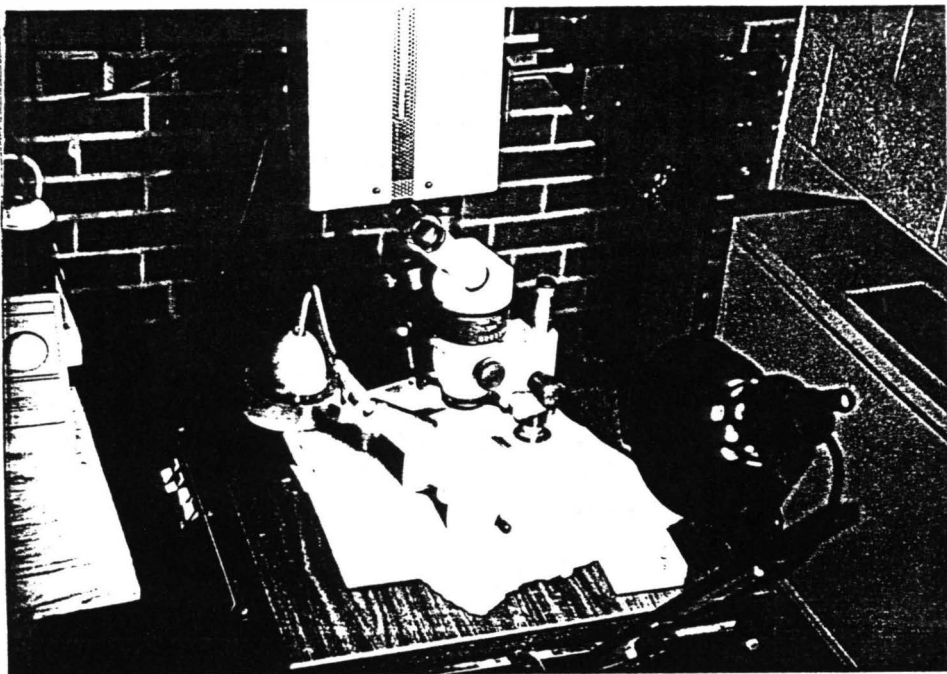
Figure 22. Lighting Arrangement in the Image Analyzer

The upper threshold was kept at 250 where as the lower threshold was adjusted to have a fully-covered circle with green color. This was done as the detection color was green and the circle was the object or field of interest. The lower threshold was at 125. The frame was adjusted so as to have the circle of interest alone would be selected for (calibration).

After setting the circle with green color in a smaller frame the calibration option in the main menu was chosen and the area in square microns was chosen. The unit was selected as micron and 2500 was printed as the calibration

factor as the size of the ink particles was anticipated in microns. The computer was calibrated after this step and a check of the calibration was done by measuring the diameter (known) the chosen circle .

The measurement option of the main menu was selected and equivalent diameter was selected. Then the measure button was pressed to analyze the circle under calibration for the above measurement option (Equivalent diameter). The measured diameter was equal to the actual diameter. Calibration was done every time the computer was used after a fresh start up for analysis.

	Equiv Diam.	Field Area	Percent Area	Fld Perimeter	Fld Features
Field 1					
Count:	1	1	1	1	1
Min:	2499.9	4.9085E+06	59.547	7988.1	1.0000
Max:	2499.9	4.9085E+06	59.547	7988.1	1.0000
Total:	2499.9	4.9085E+06	59.547	7988.1	1.0000
Mean:	2499.9	4.9085E+06	59.547	7988.1	1.0000
StDev:	0.0	0.0	0.0	0.0	0.0

Figure 23. Computer Output for Calibration

After the calibration (Figure 24) the measurements selections were chosen as equivalent diameter, field area, field percent area, field feature count and field

perimeter. The data for these options could be obtained in raw, statistical or distribution (for options like diameter,area and etc.) form.

Hence a distribution for equivalent diameter was chosen, as the area distribution would be the same by a constant multiplication factor. The rest were chosen for statistical data.

The definitions of the above measurement options are as follows.

Equivalent diameter is the diameter of a circle with the same area as the feature. Equivalent diameter is equivalent to SQRT(4*(area)/pi).

Field area is the total detected area of all features in the frame, less the area of any features excluded by touching the frame. If all particles were merged into one particle, this would be its area.

Field feature count (Field particle count) is the number of detected particles in the field of view. All particles are reported. The "Measurement filter" has no affect on field particle count.

Field percent area is the ratio of the detected field area to the area of the frame, expressed as a percent.

Field perimeter is the length of all the feature

boundaries, including all the interior boundaries. Field perimeter is the sum of all individual feature perimeters.

As per the efficiency calculation the total area of the ink particles was necessary for the floated, non-floated and before-floated portions of the ink dispersion. A total of twelve fields was analyzed in the Image analyzer.

Figures 25 and 26 show the view of the floated and non-floated portions for the ink and water dispersion whereas Figures 27 and 28 show the view of the floated and non-floated portions of the ink, fiber and water system.

To do this analysis in series a multiple field option was selected in which the area and other measurement options were added cumulatively as the number of fields increased to ten. Thus, this option reduced the time required for extra calculations.

To have a sharp image of the field, Laplacian edge enhance was done and also the automatic range of the equivalent diameter distribution was selected. There are several other options which are helpful in different purposes for various research projects.
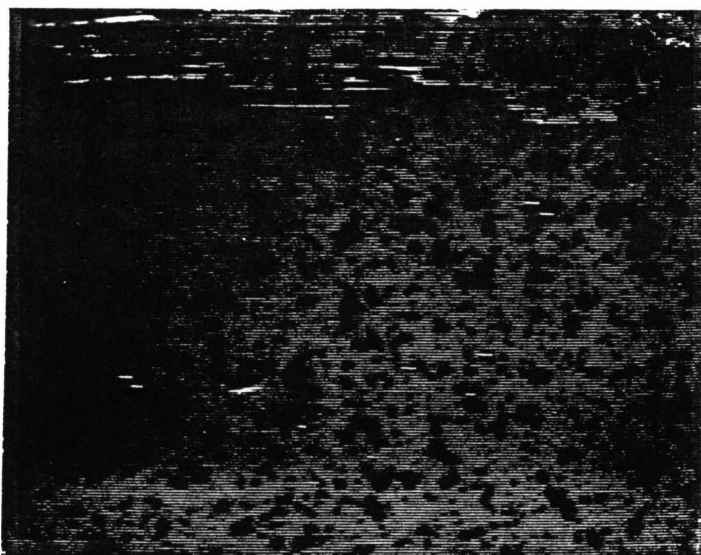
Figure 24. View of the Floated Portion of the Dispersion
for Ink and Water System
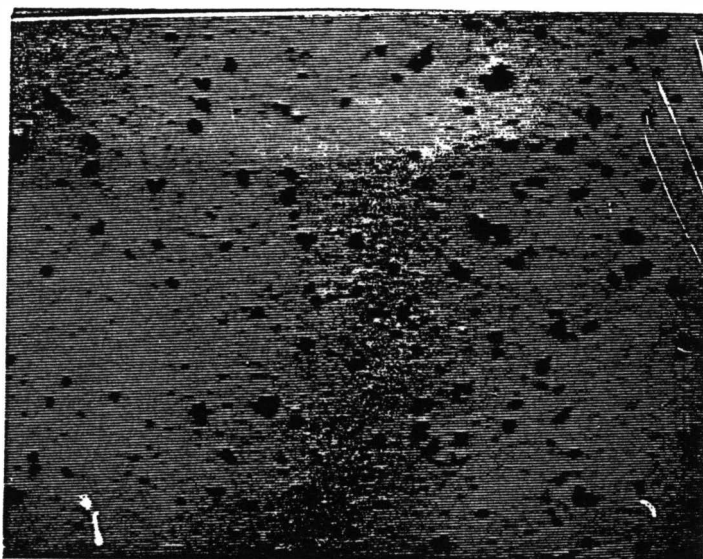
Figure 25. View of Non-Floated Portion of the Dispersion
for Ink and Water System
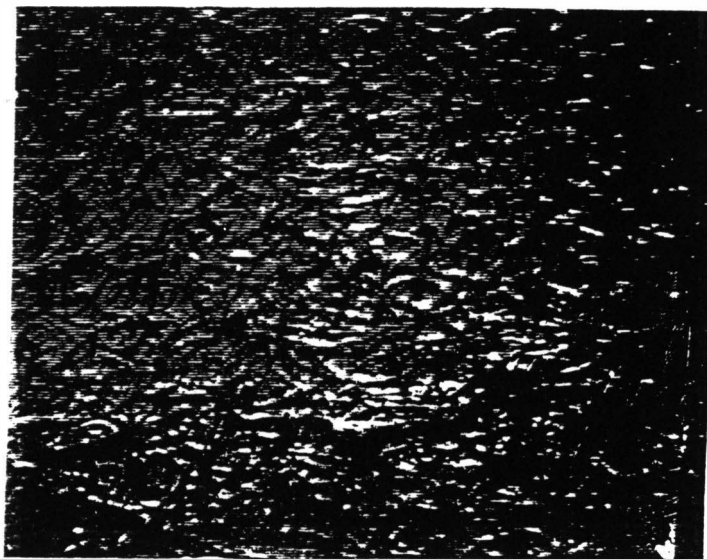
Figure 26. View of the Floated Portion of the Dispersion
for Ink, Fiber and Water System
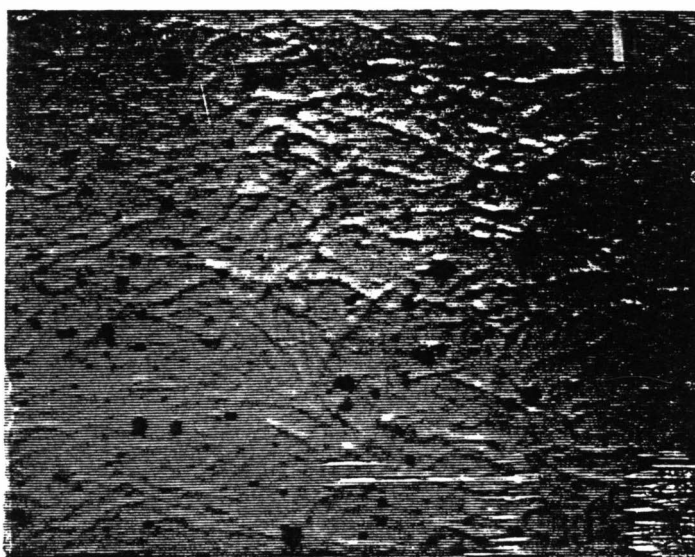
Figure 27. View of the Non-Floated Portion of the Disper-
sion for the Ink, Fiber and Water System

Appendix D

The Hallimond Tube

The Hallimond Tube

A laboratory type of flotation device called a modified "Hallimond Tube" (8) was used in this work. The Hallimond Tube, made of glass, was originally used for the flotation studies of mineral ores of different kinds. The original Hallimond tube was modified periodically by the researchers resulting in a modified "Hallimond tube".

This device consists of an inclined glass tubular section of volume approximately 250 ml. The gas for flotation is passed from the bottom of the tube. In the works of Fuerstenau et al (8) and Larsson et al (7), a capillary tube (80μm) extension from the bottom of the tube, was used to pass the air. They (8) also used a magnetic stirrer inside the tube to increase the collision frequency of the particles and air-bubbles. Due to the unavailability of this device from the commercial equipment manufacturers in United States, a similar one was designed and made with the help of Dr. Robert Nagler. As the bottom of this device was not flat enough to use a magnetic stirrer, a sintered glass bottom was added. Nitrogen gas from a pressurized cylinder was passed through the sintered glass bottom into the suspension under study.

The ink particles were carried to the surface of the

suspension along with the air-bubbles. If the air-bubbles burst at the surface, the floated ink particles will drop into the side arm of the device. Otherwise, the concentrated ink particles will remain in the froth which can be removed at the end of the experiment. In this experiment, the dispersion was filled to the junction of the side tube with the main tube (figure 5). The floated ink together with the foam were collected periodically during the flotation through the side arm for further analysis.

Operation and Modification of Hallimond Tube

Flotation cells or washing devices are usually used for the deinking research. But in this research a "modified Hallimond tube" was used. The Hallimond tube, shown in Fig.5., is generally used for the froth flotation studies of mineral ores of different kinds. The original Hallimond tube was modified several times by many researchers. The Hallimond tube (fig.5), was made with the available information in the literature (8).
The tube was connected to an air-regulator which in turn was connected to a nitrogen cylinder. Nitrogen was used instead of air as air was not available in pressurized form and also it was used in the studies of Larsson et al (7).

A cleaning procedure with nitric acid and distilled water was also developed to keep the sintered glass clean. Thus, the modified Hallimond tube was made suitable for the experiments.

For experiments at elevated temperatures a water-bath was used and some small changes were done in the experimental set up. The side tube was inserted into a one-hole rubber stopper fitted in a conical glass flask to prevent the water from the waterbath entering the floated portion of the dispersion.

Appendix E

Calculation of Surfactant Concentration and Addition Level

Calculation of Surfactant Concentration and Addition

The surfactant chosen was Pluronic L-10, a product of BASF.

Surfactant Specification

Trade name: PLURONIC L10

Chemical name: Polyoxypropylene-polyoxyethylene
                       block copolymer

Chemical Family: Polyglycol

Molecular weight: 3200

Ingredients: 100% PLURONIC L10

Formula: Not available

Boiling/melting point: Not available

Specific gravity: 1.06

Solubility in water: Complete

pH: 5.0-7.5 (1% aqueous solution)

Cloud point: 32°C

Appearance: Liquid

Supplier: BASF

The calculation of surfactant addition on the basis of (O.D) pulp was as follows:

Surfactant addition was decided to be from 0% to 1% on the basis of pulp (0%, 0.025%, 0.05%, 0.1%, 0.3%, 0.5%, 0.7%,

0.9% and 1%) at 5% consistency.

For the experiment a 1000 ml dispersion of ink in water was taken and the surfactant amount was calculated for 1000 ml.

Calculation of Surfactant amount

For 5 gm (O.D) pulp the amount of surfactant (at 0.025%) necessary   = 5 x 0.00025 gm

i.e., the amount of surfactant required for 95 ml(95 gm of water + 5 gm of pulp) of water and ink present in the pulp

= 0.00125 gm

Hence for a dispersion containing 1000 ml of water and ink the amount of surfactant   = 0.00125 x 1000/95 gm

= 0.01316 gm

1 ml of the surfactant was diluted with 99 ml of distilled water in a container so that such a small mass of the surfactant necessary for the experiments could be taken volumetrically as per the calculation below. The error in addition was expected to be less in this way.

For 1.06 gm of the surfactant 100 ml of the solution is necessary. Therefore for 0.01316 gm of surfactant the amount of surfactant solution = 100 x 0.01316/1.06

= 1.24 ml

1.24 ml of the solution was added to 1000 ml dispersion and used for the experiment.

Similarly, the calculation for the rest of the addition levels is as follows:

For 0.05% addition, the volume of the solution for 1000 ml dispersion $= 0.0005 \times 5 \times 1000 \times 100/95 \times 1.06$

$$= 2.48 \text{ ml}$$

A common multiplication factor for simple calculations was determined as follows:

$$\text{Factor} = 5 \times 1000 \times 100/95 \times 1.06$$

$$= 4965.2433$$

For 0.1% addition, the solution volume

$$= \text{Factor} \times 0.001$$

$$= 4.97 \text{ ml}$$

For 0.3% addition $= \text{Factor} \times 0.003$

$$= 14.896 \text{ ml}$$

For 0.5% addition $= \text{Factor} \times 0.005$

$$= 24.83 \text{ ml}$$

For 0.7% addition $= \text{Factor} \times 0.007$

$$= 34.76 \text{ ml}$$

For 0.9% addition $= \text{Factor} \times 0.009$

$$= 44.69$$

For 1.0% addition $= \text{Factor} \times 0.01$

$$= 49.65 \text{ ml}$$

Calculation of Surfactant Concentration

1000 ml of the dispersion was taken for an experiment. The amount of surfactant necessary for 100 ml of the dispersion @ 0.3% addition = 0.003 x 5gm

= 0.015 gm

ie, for 95 ml of the ink dispersion 0.015 gm of surfactant was necessary. Therefore, for 1000 ml of the ink dispersion the amount of surfactant necessary = 0.015 x 1000/95 gm

= 0.1579 gm

Surfactant concentration = .16 gm/L

= 0.016% (weight basis)

Molecular weight of the surfactant = 3200

Surfactant concentration = 0.1579/3200 moles/liter

= 0.000049 moles/liter

= 49 μM/L

Similarly for 0.5% addition, the surfactant concentration is determined as 82.2 μM/L and for 0.7% addition, the surfactant concentration was 115.1 μM/L.

Appendix F

Determination of Flotation Efficiency by Image Analysis

Determination of Flotation Efficiency by Image Analysis

The main aim of the analysis of the samples was to find the area of the ink floated and the area of the ink left behind in the Hallimond tube so that the flotation efficiency could be determined. Efficiency was expressed on the ratio of the area of the floated ink particles to the summation of the area of the floated and non-floated ink particles.

An Image Analyzer, Figure 6., was used for finding the field area and size distribution and the procedure used is explained in Appendix B. A sample of the Image Analysis results obtained for each field for the floated, non-floated and before floated portions of the dispersion are shown in Figures 29, 30 and 31 respectively. A sample calculation of the flotation efficiency (for ten fields) and the check of field area and number of ink particles balance is shown in pages 122, 123 and 124.

	Equiv Diam.	Field Area	Percent Area	Fld Perimeter	Fld Features

Field 1

count:	761	1	1	1	1
in:	12.830	1.1015E+06	4.4636	1.1942E+05	761.00
ax:	155.03	1.1015E+06	4.4636	1.1942E+05	761.00
otal:	27544.2	1.1015E+06	4.4636	1.1942E+05	761.00
ean:	36.195	1.1015E+06	4.4636	1.1942E+05	761.00
tDev:	23.283	0.0	0.0	0.0	0.0

| BIN	START	END	COUNT	BIN	START	END	COUNT
1	12.830	17.417	138	17	86.223	90.810	7
2	17.417	22.004	86	18	90.810	95.397	5
3	22.004	26.591	103	19	95.397	99.984	5
4	26.591	31.179	43	20	99.984	104.57	5
5	31.179	35.766	84	21	104.57	109.16	1
6	35.766	40.353	54	22	109.16	113.75	1
7	40.353	44.940	57	23	113.75	118.33	1
8	44.940	49.527	32	24	118.33	122.92	2
9	49.527	54.114	22	25	122.92	127.51	0
10	54.114	58.701	25	26	127.51	132.09	0
11	58.701	63.288	23	27	132.09	136.68	1
12	63.288	67.875	16	28	136.68	141.27	1
13	67.875	72.462	16	29	141.27	145.86	1
14	72.462	77.049	11	30	145.86	150.44	1
15	77.049	81.636	8	31	150.44	155.03	0
16	81.636	86.223	10	32	155.03	159.62	1
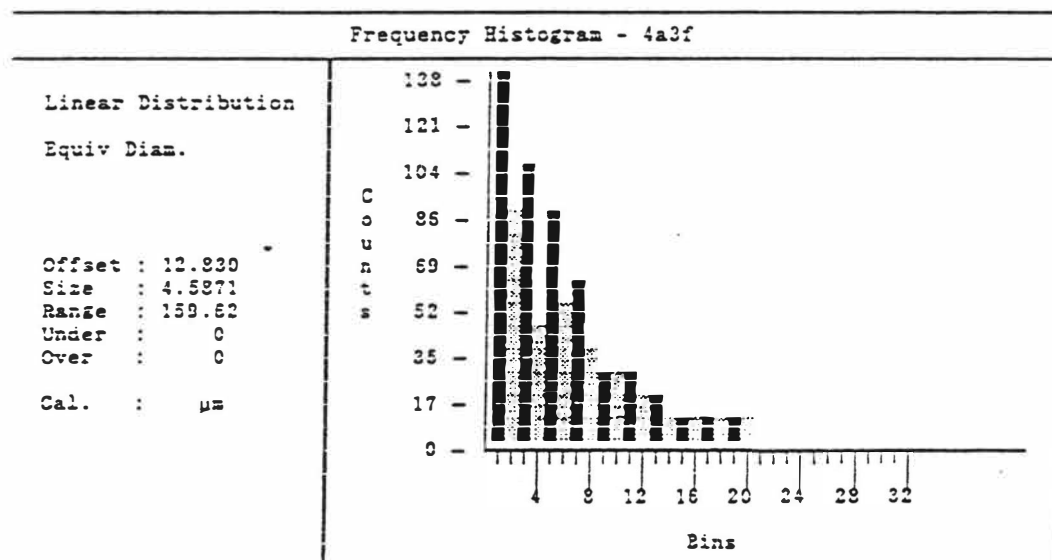
Figure 28. Sample Image Analyzer Output for One Field of a Sample of Floated Portion of the Dispersion.

OMNICON 3500 MEASUREMENT REPORT
5:57 P.M. on   Fri., Apr. 28, 1989
Calibration :   1x   1.2929E+02   μm²/pp
SAMPLE NAME : 4a3nf

	Equiv Diam.	Field Area	Percent Area	Fld Perimeter	Fld Features

Field 1

Count:	161	1	1	1	1
Min:	12.830	2.4022E+05	0.97341	25138.0	161.00
Max:	124.29	2.4022E+05	0.97341	25138.0	161.00
Total:	5942.8	2.4022E+05	0.97341	25138.0	161.00
Mean:	36.912	2.4022E+05	0.97341	25138.0	161.00
StDev:	23.339	0.0	0.0	0.0	0.0

BIN	START	END	COUNT	BIN	START	END	COUNT
1	12.830	16.429	27	17	70.412	74.011	4
2	16.429	20.028	19	18	74.011	77.609	1
3	20.028	23.627	14	19	77.609	81.208	2
4	23.627	27.226	11	20	81.208	84.807	1
5	27.226	30.825	8	21	84.807	88.406	4
6	30.825	34.423	17	22	88.406	92.005	1
7	34.423	38.022	3	23	92.005	95.604	1
8	38.022	41.621	5	24	95.604	99.202	0
9	41.621	45.220	10	25	99.202	102.80	0
10	45.220	48.819	4	26	102.80	106.40	0
11	48.819	52.418	10	27	106.40	110.00	1
12	52.418	56.016	6	28	110.00	113.60	0
13	56.016	59.615	4	29	113.60	117.20	0
14	59.615	63.214	2	30	117.20	120.80	0
15	63.214	66.813	3	31	120.80	124.39	1
16	66.813	70.412	1	32	124.39	127.99	1

Frequency Histogram - 4a3nf

Linear Distribution

Equiv Diam.

Offset : 12.830
Size   : 3.5999
Range : 127.99
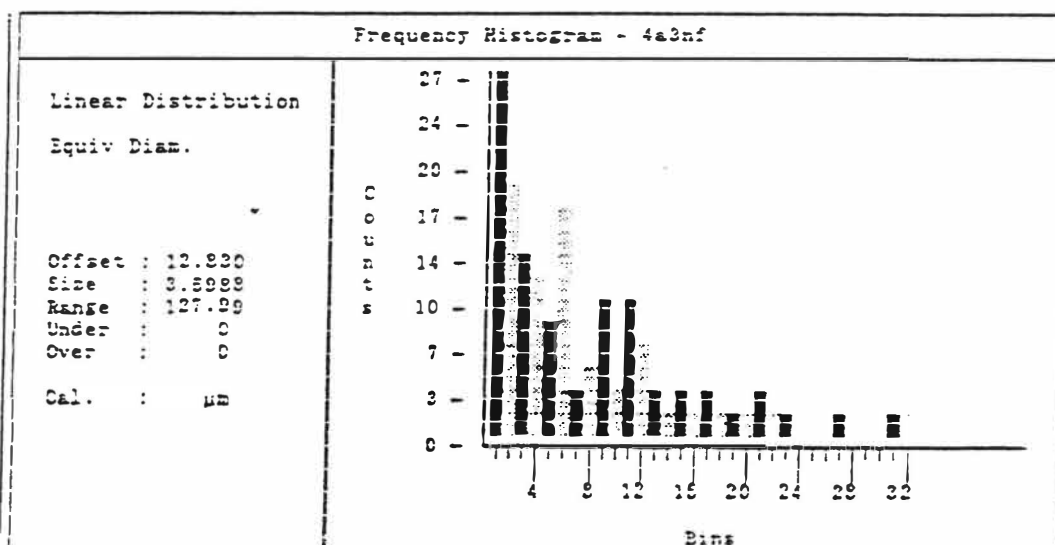Under :   0
Over   :   0

Cal.   :   μm

Figure 29. Sample Image Analyzer Output for One Field of a Sample of
Non-Floated Portion of the Dispersion.

OMNICON 3600 MEASUREMENT REPORT
9:58 P.M. on   Fri., Apr. 28, 1989
Calibration :   1x 1.2929E+02 $\mu=^2$/PP
SAMPLE NAME : bf3&4

	Equiv Diam.	Field Area	Percent Area	Fld Perimeter	Fld Features

Field 1

	Equiv Diam.	Field Area	Percent Area	Fld Perimeter	Fld Features
Count:	1045	1	1	1	1
Min:	12.830	3.0166E+06	12.224	2.2843E+05	1045.0
Max:	266.98	3.0166E+06	12.224	2.2843E+05	1045.0
Total:	48346.6	3.0166E+06	12.224	2.2843E+05	1045.0
Mean:	46.265	3.0166E+06	12.224	2.2843E+05	1045.0
StDev:	39.477	0.0	0.0	0.0	0.0

BIN	START	END	COUNT	BIN	START	END	COUNT
1	12.830	21.029	334	17	144.01	152.20	9
2	21.029	29.227	152	18	152.20	160.40	3
3	29.227	37.426	99	19	160.40	168.60	6
4	37.426	45.624	75	20	168.60	176.80	4
5	45.624	53.822	74	21	176.80	185.00	3
6	53.822	62.021	51	22	185.00	193.20	2
7	62.021	70.219	44	23	193.20	201.39	2
8	70.219	78.418	40	24	201.39	209.59	1
9	78.418	86.616	28	25	209.59	217.79	1
10	86.616	94.814	22	26	217.79	225.99	0
11	94.814	103.01	25	27	225.99	234.19	2
12	103.01	111.21	15	28	234.19	242.39	0
13	111.21	119.41	10	29	242.39	250.58	0
14	119.41	127.61	16	30	250.58	258.78	0
15	127.61	135.81	18	31	258.78	266.98	0
16	135.81	144.01	8	32	266.98	275.18	1

Frequency Histogram - bf3&4

Linear Distribution

Equiv Diam.

Offset : 12.830
Size : 8.1984
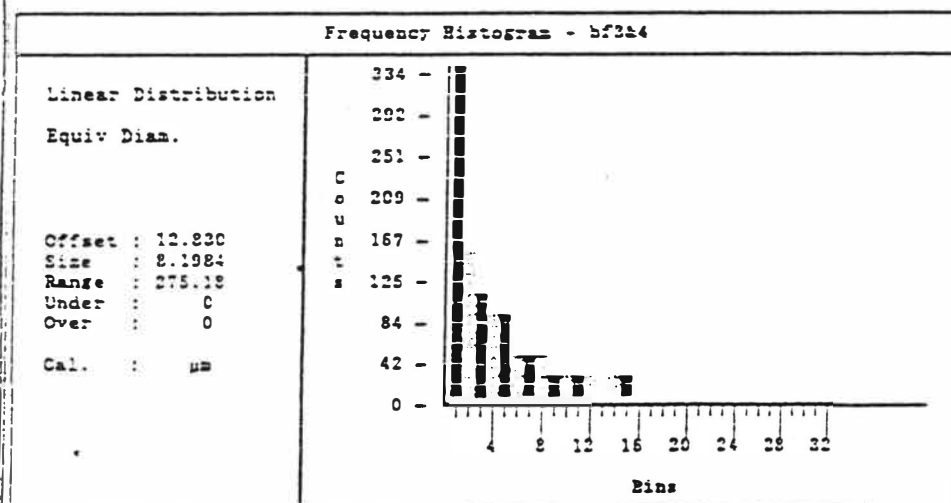Range : 275.18
Under :   0
Over :   0

Cal. : μm

Figure 30. Sample Image Analyzer Output for One Field of a Sample of Original
Dispersion Before Flotation

Calculation of Flotation Efficiency and a Balance Check

Floated portion		Non-floated portion	
Field area μm^2	particle number	Field area μm^2	particle number
2118900	1090	63610	37
1101500	761	73436	43
1165000	675	216820	164
1206400	716	153200	130
1569700	772	127610	117
1436000	856	159930	131
1516700	852	175060	159
1979200	908	240220	161
1964600	974	255480	158
2326700	1108	199360	146

μ = 1639000 μ = 871.2 μ = 166473 μ = 124.6
    = 433000   = 149.5   = 65000   = 47.25

Before flotation

Field area μm^2	Particle number
2637600	1201
2425900	995
2574700	1021
2203400	1019
2334800	951
2468000	985
1596500	649
2367400	973
2110000	933
2036700	980

μ = 2275500
    = 306000
	μ = 970.7
	    = 13

Average area of the floated ink particles per field

$$= 1,639,000 \ \mu m^2$$

Average area of the non-floated ink particles per field

$$= 166,473 \ \mu m^2$$

Efficiency of flotation   = 1639000 x 100/(1639000 + 166473)

$$= 90.78 \ \%$$

Balance Check for the Area

Average area of the ink particles before flotation per field

$$= 2275500 \ \mu m^2$$

Total area of the ink particles in the floated and non-floated portion per field

$$= (1639000 + 16647)3 \ \mu m^2$$

$$= 1805473 \ \mu m^2$$

The difference in area $\quad = 470027 \ \mu m^2$

Error $\quad = 470,027 \ x \ 100/2,275,500 = 20\%$

Balance check for the number of ink particles

Average number of ink particles before flotation per field

$$= 970.7$$

Average number of ink particles in the floated and non-floated portion per field

$$= (871.2 + 124.6)$$

$$= 995.8$$

The difference in the number of ink particles per field

$$= 25.1$$

Error $\quad = 25.1 \ x \ 100/971 = 2.5\%$

The balance check for both the field area and number of ink particles verifies a satisfactory result of the analysis.

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